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42nd International Conference & Exposition on Advanced Ceramics and Composites

ABSTRACT BOOK

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Introduction

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How to Use the Abstract Book

Refer to the Table of Contents to determine page numbers on which specific session abstracts begin. At the beginning of each session are headings that list session title, location and session chair. Starting times for presentations and paper numbers precede each paper title. The Author Index lists each author and the page number on which their abstract can be found.

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Monday, January 22, 2018

Plenary Session

Plenary Session

Room: Coquina Salon D
Session Chairs: Jingyang Wang, Shenyang National Laboratory for Materials Science, Institute of Metal Research, Chinese Academy of Sciences; Manabu Fukushima, National Institute of Advanced Industrial Science and Technology (AIST)

8:50 AM

(ICACC-PLEN-001-2018) Tiny Bubbles: An Innovative Ceramic Opens New Opportunities in Medicine, Security, Energy, and Environmental Remediation

G. Wicks*[1]

1. CTO, Applied Research Center, USA

Tiny Bubbles or Porous Wall Hollow Glass Microspheres (PWHGMs), represent an example of Multi-use Technologies. The technology was originally developed for nuclear applications at the Savannah River National Laboratory and now is being further advanced and tailored for a multitude of new uses in other fields and disciplines. This work is currently being conducted at the Applied Research Center (ARC) in Aiken SC, as well as at a new biotech spin off company, SpheroFill, LLC. Among the interesting initiatives are applications of tailored PWHGMs in medicine (ex. drug delivery platforms, contrast agents, tissue augmentation, laryngeal use), security (ex. non-proliferation, anti-counterfeiting), energy (ex. hydrogen storage, batteries), and environmental remediation (ex. CO2 sequestration). The PWHGMs are tiny hollow glass microspheres or micro-balloons about 1/3 the diameter of a human hair. They range in size from a few to 100 microns in diameter, and have thin outer shells approximately 1-2 microns thick. The most unique feature that distinguishes these microspheres from others, is that a continuous, through-wall porosity is induced via phase separation and subsequently controlled on a scale of 100 to 1,000 Angstroms. This provides pathways from the outside of the microspheres to their interior, and allows the tiny glass cocoons to be filled with cargos of interest, including solids, liquids and gases. The cargos or payloads can then later be released on demand. The development of these unique materials and some of the exciting new applications being studied will be discussed.

9:30 AM

(ICACC-PLEN-002-2018) Strategies for searching for damage tolerant ceramics: from MAX phases to MAB phases

Y. Zhou*[1]

1. Aerospace Research Institute of Materials & Processing Technology, China

Transition metal carbides, nitrides and borides are potential materials for extreme environment applications. However, the brittleness and defect sensitivity are main obstacles to their applications. Formation of nanolaminated structures like MAX phases ($M_{n+1}AX_n$, where M is an early transition metal, X is carbon or nitrogen, A is a III_A-VI_A group element, n=1-6) has been proven to be an effective approach to overcome the brittleness of transition metal carbides and nitrides. These materials are characterized by a transition metal carbide or nitride $M_{n+1}X_n$ layer interleaved by a close packed A-group element layer, which exhibit a unique combination of the merits of both metals and ceramics. These properties have been proven to be underpinned by the diverse chemical bonding. The continuous discovery of new members and properties, and new applications of MAX phases engenders an enormous interest in searching for materials with similar structure and properties. In this presentation, the multi-scale structural features and the applications of MAX phases will be introduced first. Then strategies for searching for new layer structured damage tolerant ceramics will be proposed. Finally, the structure and properties of Cr, Si, Al or Y containing new layer structured materials including MAB phases, $(MC)_nAl_3C_2$, $(MC)_nAl_4C_3$ and$(MC)_n[Al(Si)]_4C_3$ phases will be described.

10:40 AM

(ICACC-PLEN-003-2018) Research. Why? For whom? How?

R. J. Brook*[1]

1. University of Oxford, Dept. of Materials, United Kingdom

The motives for undertaking research are many and diverse, but three major driving forces can be identified; these are presented. The relationships between research sponsor and research performer are similarly of many different types. The one between researcher and government is, however, particularly prevalent; the ambitions of the two sides are not always parallel and risks then arise for the shared enterprise. Rules for the judgement and support of research are reviewed and some thought is given to the approaches which can be taken by the researcher in search of true originality.

11:20 AM

(ICACC-PLEN-004-2018) 3D Microstructure is the "Know it All" – Advanced Classification and Quantitative Analysis including Data Mining and Deep Learning Methods

F. Muecklich*[1]

1. Saarland University and Materials Engineering Center Saarland, Dept. Mat.Science & Engineering, Germany

The term microstructure refers to the complete "internal" structure of a material on the micro, nano and atomic scales. On one hand it records de facto, the entire history of material's processing procedures through its phase composition, defect structure and microstructural morphology. On the other hand, almost all properties are predetermined by the microstructure. Thus, it can be seen as the "multi-scale archive" from which we can "read" at each relevant scale the precise information about the genesis of microstructure formation processes as well as predict the final material properties. Recent advances in 3D tomography methods on the micro, nano and atomic scales allow not only for higher local resolution but also for correlative combination of microscopic techniques in order to investigate microstructures with higher morphological and topological complexity, which is very crucial for modeling and quantitative understanding of high performing materials in the future. In our daily experimental work but also in commercial quality control, the best possible classification of microstructures by individual experts using image analysis tools is the typical procedure, which convoys our investigation.

S1: Mechanical Behavior and Performance of Ceramics & Composites

Wear, Errosion, Oxidation and Shock

Room: Coquina Salon D
Session Chairs: Jonathan Salem, NASA Glenn Research Center; Richard Todd, University of Oxford

1:30 PM

(ICACC-S1-001-2018) Nanoscale Wear of Ceramics by In Situ TEM Scratch Testing

E. D. Hintsala*[1]; D. D. Stauffer[1]; S. Asif[1]

1. Bruker NI, R&D, USA

A 2D MEMS based transducer designed to operate in situ TEM was recently developed for use with the Hysitron PI 95 PicoIndenter. This allows for simultaneous measurement of normal and lateral forces, with sub-nm displacement resolution and sub-μN force resolution. Such an instrument opens possibilities for fundamental

studies into nanoscale tribology, including friction, adhesion and wear, by coupling high resolution mechanical data with TEM imaging to help understand deformation mechanisms. In the case of ceramics, nanoscale wear mechanisms, i.e., formation of the initial damage layer is of interest for multi-scale modelling to better predict material behavior, particularly for geological materials. The individual defect nucleation and propagation of cracks, and potentially, dislocations can be studied in the context of material volume removal. This allows important comparisons against atomistic scale simulations, furthering the goal of understanding wear resistance in terms of fundamental mechanical properties, such as hardness and fracture toughness.

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(ICACC-S1-002-2018) Novel Benchtop Technique to Study the Behavior of High Friction Materials for Brake Applications

G. Ramirez*[1]; S. Shaffer[1]; C. Greening[2]; P. Filip[3]; K. Farokhzadeh[1]

1. Bruker Nano Surfaces, USA
2. Greening Test Laboratories, USA
3. Southern Illinois University Carbondale, USA

Development of new generation of materials for automotive brake applications comes with many challenges including formulation of materials that meet the demanding performance expectations, cost reduction and compliance with environmental regulations. Evaluation techniques, such as the dynamometer are fully developed, but the cost and time of preparing materials for component level tests are high. Here we present a technique that allows one to characterize the tribological performance of small samples made of friction materials in a precise and rapid manner, while monitoring key parameters like friction, temperature and wear. We show the results of the characterization of materials that were evaluated using an industry-standard dynamometer test (SAE J2522), and the comparison of those against a benchtop technique (Bruker UMT-TL). The testing conditions were extracted from the dynamometer tests, and implemented on the benchtop test, using same ranges of contact pressures, temperatures, and linear speed and acceleration. The results of these experiments indicated that this technique can rank materials in similar way as the dynamometer, and providing more information of the material behavior, like easy collection of debris and observation of stick-slip behavior, making this technique a complementary and valuable tool to study fundamental phenomena in brake material friction and wear.

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(ICACC-S1-003-2018) Niobium Carbide NbC as cutting tool material and for wear protection

M. Woydt*[1]; H. Mohrbacher[2]; J. Vleugels[3]; S. Huang[3]

1. BAM Federal Institute for Materials Research and Testing, Germany
2. Niobelcon bvba, Belgium
3. Katholieke Universiteit Leuven, Department of Metallurgy and Materials Engineering (MTM), Belgium

The present paper illuminates the metallurgical progress on niobium carbide based hard metal developments, which are characterized by: a.) the substitution of cobalt binder by nickel, b.) the change from SPS to conventional sintering and c.) by switching from lab to pilot scale. The toughness was increased in the frame of these develop-ments without loosening the hardness level. Stoichiometric and sub-stoichiometric, submicron NbC powders were used. The hardness-toughness profile of NbC grades match those of WC and cermet grades. Apart from the aforementioned parameters, the properties depend from the powder processing and sintering conditions. The functional profile of NbC and WC grades bonded by cobalt and nickel are benchmarked by 4-point bending strength, elastic moduli and hot hardness until 1000C, dry sliding wear (T= 22/400C; v= 0.1-10 m/s), abrasive wear (G65) and cutting performances under emulsion and coolant-free turning and milling against different alloys (C60, 100Cr6, 42CrMo4, X90CrMoV18, 300WA, GG35).

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(ICACC-S1-004-2018) Assessment of time-temperature equivalence for hydrothermal ageing of zirconia using a fast, stepwise procedure

L. Gremillard*[1]; C. Wei[1]; J. Chevalier[1]; K. Hans[2]; T. Oberbach[2]

1. INSA, Materials, Engineering and Science, France
2. Mathys Orthopaedie GmbH, Germany

Hydrothermal ageing of zirconia is an important issue impacting both the lifetime and functionnality of zirconia-based ceramic components. Hydrothermal ageing can be defined as a transformation, triggered by the presence of water, from the metastable tetragonal phase to the stable monoclinic phase. The mechanism of hydrothermal ageing is the same between room temperature and ~150°C. Moreover, hydrothermal ageing is thermally activated, and can thus be evaluated at temperatures higher than the usage temperature and extrapolated to usage (room or body) temperature. This procedure requires the measurement of whole ageing kinetics at different temperature, usually between 100 and 134°C. This can be time consuming especially if one wishes a high-precision extrapolation and uses low temperatures. Besides, this procedure requires at least one sample per test temperature. Ageing being considered as a cumulative damage, we propose here a much faster procedure based on the measurement of partial kinetics at different temperatures on one single sample. This new procedure can be ten times faster than the usual one, and only requires a single sample. We will show itsapplication on several zirconia and zirconia-toughened alumina materials. A discussion around the reliability and the usefulness of this procedure will be presented.

2:50 PM

(ICACC-S1-005-2018) Quantitative estimation of hydration layer during chemical mechanical polishing of glass

S. Suda*[1]; T. Sugimoto[1]; S. Kawasaki[1]

1. Shizuoka University, Engineering, Japan

Cerium oxide (ceria) is widely used as abrasives for polishing glasses. Ceria particles have relatively low mechanical strength, but they show extremely high removal rate and result in scratch-free surface. This high polishing performance of ceria abrasives is derived from chemical reactions during polishing that is generally referred to as chemical mechanical polishing (CMP). Chemical factor of CMP (chemical polishing) would generate hydration layer on the surface of glasses during polishing, and the other factor of mechanical would then sweep the hydration layer off the glasses. The formation rate of hydration layer derived from the chemical polishing would be a dominant parameter to make scratch-free ultra-smooth glass efficiently by polishing. We then prepared the polishing model that it is possible to estimate slurry resistivity and the interfacial area specific resistance (ASR). The hydration layer formed as a result of the shear stress during polishing but was independent of polishing loads. The formation rate of hydration layer was increasing with increasing lanthanum concentration dissolved in ceria lattice. Redox of Ce^{4+}/Ce^{3+} would play a major role for the formation of hydration layer by the chemical factors of CMP. This redox accompanies the process of the charge transfer at glass/slurry interface. Electron charge carrier would be important role in chemical polishing.

3:30 PM

(ICACC-S1-006-2018) Boron Effects on Sodium Sulfate-Induced Hot Corrosion of SiC

L. A. Herweyer*[1]; E. J. Opila[1]

1. University of Virginia, Materials Science and Engineering, USA

Boron effects on the Na_2SO_4 –induced hot corrosion of SiC were explored to fundamentally understand how BN interphases in SiC ceramic matrix composites (CMCs) will influence these materials. To isolate the underlying mechanism, the complex architecture of SiC CMCs was modeled by depositing a planar 600 nm thick BN coating

on commercially sintered SiC (Hexoloy™). Controlled amounts of Na_2SO_4 salt were then applied as films on specimen surfaces. Time dependent hot corrosion experiments were conducted from 0.75h-24h at 800°C and 1000°C, while temperature dependent experiments were conducted at 24h from 700°C-1100°C, all in a flowing 0.1% SO_2/O_2 gas mixture. After exposure, bench top weight change, SEM, EDS and XRD were used to characterize the specimens. Corrosion products were removed using HF and the Si, Na, B and S contents of the corrosion products were chemically analyzed using inductively coupled plasma optical emission spectroscopy (ICP-OES). Optical profilometry was additionally used to quantitatively assess the surface attack features resulting from hot corrosion. It was found that hot corrosion occurred below the melting point of Na_2SO_4 (T_m=884°C), to temperatures as low as 750°C, and that two distinct hot corrosion morphologies occurred below and above the melting point of Na_2SO_4 due to the presence of Boron.

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(ICACC-S1-007-2018) Erosion Behavior in an Oxide/Oxide Ceramic Matrix Composite

M. J. Presby*[1]; N. Kedir[1]; L. Sanchez[1]; C. Gong[1]; D. Faucett[1]; S. R. Choi[1]

1. Naval Air Systems Command, USA

The erosion behavior of ceramic matrix composites (CMCs) is markedly different from traditional monolithic ceramic materials due to their unique architectural configurations. As CMCs are further implemented into aero engines, the need to characterize their erosion behavior arises as these materials can be subjected to erosive environments. The erosion behavior of an N720/alumina oxide/oxide CMC was characterized at ambient temperature as a function of particle type, size, and velocity. The erosion damage was quantified with respect to the erosion rate, and the damage morphology was characterized via SEM and optical microscopy along with 3D image mapping. Post-erosion residual strength testing was performed in flexure to quantify the severity of the erosion damage.

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(ICACC-S1-008-2018) Effects of Ablation Products on Expanding Hypersonic Flows

B. Donegan*[1]; R. Greendyke[1]; R. Ravichandran[2]; S. Lewis[2]; R. Morgan[2]; T. McIntyre[3]

1. Air Force Institute of Technology, Department of Aeronautics and Astronautics, USA
2. The University of Queensland, Centre for Hypersonics, Australia
3. The University of Queensland, School of Mathematics and Physics, Australia

Thermal protection is required for vehicles entering planetary atmospheres to protect against severe thermal and mechanical loads. Despite decades of research, our understanding of the gas-surface interaction of high-temperature materials remains limited. Uncertainties in engineering models used to predict heating and aerodynamics can lead to large factors of safety when determining the thickness of the TPS. Much of this uncertainty is due to the inability to accurately predict heating and ablation rates, particularly of the afterbody. Though afterbody heating rates are much lower than forebody values, uncertainties in the prediction of these values is significantly higher with radiative heating identified as a major contributor. The uncertainty in predicting afterbody heating is due to the complexity of the flowfield in the wake region. In this study, numerical simulations were conducted using the LAURA flowfield solver coupled to ablation and radiation models. Data from experiments performed at the X2 expansion tunnel at the University of Queensland were compared to the simulations. Preheated strips of carbon-carbon and silicon carbide-coated carbon-carbon were mounted in a two-dimensional compression wedge and tested in 8.5 km/s Earth entry flow. Calibrated spectral measurements were obtained in the expansion region targeting atomic silicon and CN violet bands for surface temperatures of 1500 - 2700 K.

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(ICACC-S1-009-2018) Fracture evaluation of ceramic insulating substrate subjected to cyclic heating

S. Honda*[1]; T. Ohno[1]; Y. Daiko[1]; T. Ideno[2]; F. Momose[3]; Y. Iwamoto[1]

1. Nagoya Institute of Technology, Japan
2. DOWA Power Device Co., Ltd., Japan
3. Fuji Electric Co., Ltd., Japan

The resistance of thermal fracture of aluminum nitride insulated direct copper bonding (DCB) substrate was evaluated by the newly developed heat cycle testing system. Crack initiation and propagation was detected by monitoring acoustic emission, and the deformation of DCB substrate was measured by LVDT during heat cycle testing. The number of heat cycle on fracture was evaluated using DCB substrates which have various thicknesses and patterns of copper plate. The crack initiation was not the timing of rapid temperature change, but the cooling state in about room temperature when the temperature distribution in the substrate was almost uniform. The number of heat cycle on fracture in DCB substrates was dependent on the thickness of copper plate. The thermal fracture of DCB substrates was caused by the change during heat cycles of residual stress that was generated in the bonding of copper and ceramics. This work was supported in part by NEDO, Japan.

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(ICACC-S1-010-2018) Mechanical properties and thermal shock resistance of Si_3N_4-BN-MAS ceramics

D. Cai*[1]; D. Jia[1]; Z. Yang[1]; Y. Zhou[1]

1. Harbin Institute of Technology, School of Materials Science and Engineering, China

A novel composite ceramics with excellent mechanical properties were fabricated by means of hot-pressing using Si_3N_4, h-BN and magnesium aluminum silicate (MAS) as raw materials. The influences of starting h-BN content on the microstructural evolution, mechanical properties and thermal shock resistance of the ceramics were investigated. h-BN flakes with layered structure can play a role in toughening the Si_3N_4 matrix. Composite ceramics incorporated with 20 wt.% h-BN exhibits the highest fracture toughness of 7.37 ± 0.20 MPa $m^{1/2}$. The effect of h-BN on the thermal shock resistance of composites were investigated at temperature differences from 600 °C up to 1400°C. The strengthening effect of h-BN and the surface microstructural evolution of composites are responsible for the improved thermal shock resistance of the ceramics.

S2: Advanced Ceramic Coatings for Structural, Environmental, and Functional Applications

Thermal Barrier Coatings - Processing & Properties

Room: St. John

Session Chair: Douglas Wolfe, Pennsylvania State University

1:40 PM

(ICACC-S2-001-2018) Three-dimensional Characterization of Rumpling and Cracking in cyclic furnace tests of TBC

E. H. Jordan*[1]; P. Shahbeigi[2]; N. Asadi Aznjani[3]; S. Shahabazmohamadi[2]

1. University of Connecticut, Mechanical Engineering, USA
2. University of Connecticut, Institute of Materials Science, USA
3. University of Florida, Department of Electrical and Computer Engineering, USA

McrAlY bond coats are often assumed not to rumple. Detailed SEM 3D surface characterization will be presented that has sufficient repeatability to demonstrate the occurrence of rumpling of MCrAlY bond coats on bare bond coated samples during a cyclic furnace test. The cracking of TBCs has often been predicted to be related to bond

coat surface geometry. X-ray tomography was used to characterize the behavior of individual cracks non-destructively. The cracking was not characterized by progressive growth of specific cracks but was dominated by the occurrence of multiple new cracks and then crack linkage. Individual cracks did grow but the appearance of new cracks dominated the area increase. In addition no relationship between pre-existing bond coat geometry and the location of initiated cracks was apparent. The implications of these results to possible damage modeling will be discussed.

2:00 PM

(ICACC-S2-002-2018) Characteristics of Double-ceramic-layer TBCs Fabricated by Suspension Plasma Spray

S. Kim*[1]; S. Lee[1]; Y. Oh[1]; S. Lee[1]; H. Kim[1]; K. Lee[2]; B. Jang[3]

1. Korea Institute of Ceramic Engineering and Technology (KICET), Engineering Ceramics Center, Republic of Korea
2. Kookmin University, School of Mechanical Engineering, Republic of Korea
3. National Institute of Materials Science, Research Center for Structural Materials, Japan

Rare-earth zirconates, such as $La_2Zr_2O_7$ and $Gd_2Zr_2O_7$, have been investigated as one of the candidates for replacing conventional yttria-stabilized zirconia (YSZ) for thermal barrier coating (TBC) applications at higher turbine inlet temperatures. Rare-earth zirconate oxides exhibit little phase transformation upon heating up to melting temperature as well as low thermal conductivity, where as their mechanical properties are inferior to those of YSZ TBCs. Double-ceramic-layer (DCL) TBCs have been introduced in order to take advantage of beneficial characteristics of both YSZ and rare-earth zirconate. In this study, the fabrication of DCL-TBCs with YSZ layer and rare-earth-zirconate top layer by using suspension plasma spray are reported. Microstructure, compositional profile, thermal conductivity, and thermal durability of DCL-TBCs are characterized. The usefulness of these DCL-TBCs is also discussed.

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(ICACC-S2-003-2018) Lifetime of plasma sprayed Gadolinium-Zirconate/ Yttria Stabilized Zirconia Thermal Barrier Coatings

C. Vorkötter*[1]; R. Singh[1]; M. Tandler[1]; R. Vassen[1]; O. Guillon[1]

1. Forschungszentrum Juelich, IEK-1, Germany

Advanced Thermal Barrier Coatings are essential to increase the efficiency of next-generation gas turbine engines. Different materials and process technologies give the possibility to extend the lifetime of TBCs. Ceramic Gadolinium-Zirconate/YSZ Top Coat layers have excellent performance and can make use of the full potential of the underlying metallic Bond Coat. Oxide dispersion strengthened Materials used as an additional Flash Coating show a superior Bond Coat lifetime in comparison to single layered Bond Coats. This study investigates the thermal cycling behavior and defect mechanism of multilayered TBCs with GZO/YSZ Top Coats on advanced ODS Bond Coats in relation to the mechanical properties and porosity of the layers. Furthermore diffusion and phase transformations are analysed. The TBC is applied by atmospheric plasma spray for the Top Coat and vacuum plasma spray for the Bond Coat.

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(ICACC-S2-004-2018) The Influence of Microstructural Defects on Wear Mechanisms in Zirconia-Based Abradable Coatings

K. Bridges*[1]; D. R. Mumm[1]

1. University of California, Irvine, USA

Abradable coatings applied to turbine shrouds play an integral role in maintaining blade tip clearances, thereby preventing unnecessary gas leakage and significantly increasing turbine efficiency. As turbine material technologies advance, there is a push for the development of abradable coatings that can withstand more severe operating conditions and retain the optimum balance of abradability and durability. This study aims to investigate the influence of microstructural defects present in current technology ceramic abradable coatings on the preferred wear behavior of these systems to guide future design strategies. The coatings to be compared are air plasma sprayed dysprosia- or yttria- stabilized zirconia with varying fractions of pore former and secondary phases. The wear of both as-received and aged coatings will be tested, and deformation mechanisms will be reported. Links between different defects, their evolution with aging, and observed wear behavior will be compared with two competing definitions of desired abradable damage accommodation mechanisms, with one being energy dissipation through plastic deformation and the other depending on crack propagation and frictional sliding of the removed material to dissipate energy.

Environmental Barrier Coatings - Processing & Properties I

Room: St. John

Session Chair: Peter Mechnich, DLR - German Aerospace Center

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(ICACC-S2-005-2018) Processing effects on characteristics of rare-earth silicates for considerations as environmental barrier coatings for ceramic composites

E. Garcia Granados[1]; H. Lee[1]; S. Sampath*[1]

1. Stony Brook University, Center for Thermal Spray Research, USA

The severe working conditions that are foreseen for forthcoming power generation and aircraft/spacecraft turbine engines in order to increase their thrust and efficiency make ceramic-matrix composites (CMCs) the cornerstone to meet these stringent requirements. The performance of CMCs based on SiC is hindered by Si volatilization caused by the combustion atmosphere. The envisioned solution is protecting the CMCs based components with environmental barrier coatings (EBCs). Ceramic compositions with low thermal expansion coefficient and stable in water vapor atmospheres have been explored; barium–strontium aluminosilicate (BSAS), rare earth silicates. Atmospheric Plasma Spray (APS) stands out among other deposition techniques due to its versatility and cost/deposition efficiency. The main bottleneck for their application is the processing of these materials as dense, crystalline crack-free coatings. The present work seeks to understand the process – microstructure – property relationships in plasma sprayed rare earth silicate coatings through subjecting the materials to a deliberate choice of processing conditions to engender different thermal and kinetic energies as well as deposition temperatures in sprayed particles, evaluating the crystalline nature (XRD, DTA), microstructure (SEM) and chemical composition (EDS) of the obtained coatings.

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(ICACC-S2-006-2018) Current EBC Development and Testing at NASA

K. N. Lee*[1]; D. Waters[1]; B. Puleo[1]

1. NASA Glenn Research Center, Materials, USA

SiC/SiC Ceramic Matrix Composites (CMCs) are a game changer for advanced gas turbines because of their high temperature capability, oxidation resistance, and light weight that translate to significant reduction in fuel consumption and emission. Water vapor is highly detrimental to CMC durability by causing rapid surface recession and oxidation. Environmental Barrier Coating (EBC) is an enabling technology for SiC/SiC CMCs by protecting SiC from water vapor-induced degradation. The first CMC component entered into service in 2016 in a commercial engine and more CMC components are scheduled to follow. The introduction of CMCs represents significant challenges as failure of the EBC means rapid reduction in component life. A robust and long life EBC, a reliable lifing method, and engine-relevant testing methods to validate life need to be developed. This paper will discuss current activities in EBC development and testing at NASA.

4:00 PM

(ICACC-S2-007-2018) Environmental Durability of Environmental Barrier Coatings Deposited Via Plasma Spray–Physical Vapor Deposition

B. J. Harder*[1]; K. Lee[1]; S. Kalluri[2]

1. NASA Glenn Research Center, USA
2. Ohio Aerospace Institute, USA

Gas turbine engine efficiencies are heavily influenced by combustion temperature and component cooling requirements which has resulted in the use of silicon-based ceramics such as SiC over traditional superalloys. However, environmental barrier coatings (EBCs) are needed to protect these materials in combustion conditions and operating temperatures of 1482°C may require advanced processing methods. Plasma Spray- Physical Vapor Deposition (PS-PVD) is a hybrid technique that can tailor microstructures and compositions to optimize performance. PS-PVD EBCs of $Yb_2Si_2O_7$ were thermally cycled in a steam environment (90% H_2O/O_2) at 1426°C and heated with a high heat flux laser to a surface temperature of 1482°C. Samples were alternated between isothermal steam for oxidation and laser gradient heating in air for thermomechanical effects to determine combinatorial degradation mechanisms. Coating morphology and oxide layers were analyzed with electron microscopy, and composition and crystal structure were tracked with X-ray diffraction.

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(ICACC-S2-008-2018) Development of yttrium and ytterbium silicates from their oxides and an oligosilazane for coating application to protect Si_3N_4 in hot gas environments

M. Lenz Leite*[1]; G. Barroso[1]; W. Krenkel[1]; G. Motz[1]

1. University of Bayreuth, Ceramic Materials Engineering, Germany

Environmental barrier coatings (EBC's) are required to protect Si_3N_4 against hot gas corrosion and enable its application in gas turbines. Among which, rare-earth silicate-based EBC's stand out due to the very low corrosion rates in moist environments at high temperatures and the compatibility of thermal expansion coefficient to Si_3N_4 ceramics. Thus, the polymer-derived ceramic route was used to synthesize yttrium and ytterbium silicates for basic investigations regarding their intrinsic properties from a mixture of Y_2O_3 or Yb_2O_3 powders and the oligosilazane Durazane 1800. After pyrolysis above 1200 °C in air, the corresponding silicates are already the predominant phases. The corrosion behavior of the resulting composites was tested under flowing moist air at 1400 °C for 80 h. The material containing Yb_2SiO_5 and $Yb_2Si_2O_7$ as main crystalline phases undergoes the lowest corrosion rate (-1.8 μg cm^{-2} h^{-1}), while the corrosion rate of yttrium-based composites remained at least ten times higher. Lastly, the processing of Y_2O_3/Durazane 1800 as well-adherent, crack-free and thick (40 μm) coatings on Si_3N_4 was achieved after pyrolysis at 1400 °C in air. The resulting coating consisted of an Y_2O_3/Y_2SiO_5 top-layer and an $Y_2O_3/Y_2Si_2O_7$ interlayer due to diffusion of silicon from the substrate and its interaction with the coating system.

4:40 PM

(ICACC-S2-009-2018) Modeling of Rare Earth Disilicate Environmental Barrier Coating Degradation Through Reaction with Water Vapor

C. G. Parker*[1]; R. A. Golden[1]; V. Tikare[2]; E. J. Opila[1]

1. University of Virginia, Materials Science and Engineering, USA
2. Sandia National Laboratories, Multiscale Science, USA

Rare earth disilicate environmental barrier coatings (EBCs) for SiC/SiC ceramic matrix composites react with water vapor formed in the combustion stream of gas turbine engines. This reaction proceeds by transport of water vapor to the rare earth disilicate surface which then reacts to form rare earth monosilicate and $Si(OH)_4$ gas. The formation of rare earth monosilicate is accompanied by a reduction in volume of approximately 25%, creating a pore network for outward diffusion of the $Si(OH)_4$ gas. This reaction has been studied in the laboratory by exposing rare earth disilicate EBCs in a steam-jet furnace at 1200°C for test times of 6 – 250 hours with steam velocities of 150 – 180 m/s. SEM characterization of the evolution of the rare earth monosilicate reaction layer with time enabled the determination of the kinetics of the SiO_2 volatilization and coarsening of both the rare earth monosilicate and the pore structure. These data were used to inform a computational hybrid Potts/diffusion model of the SiO_2 volatilization reaction and microstructural evolution of the system. Implications for life prediction of rare earth disilicate EBCs at times in excess of the laboratory exposures as well as determination of the rate limiting step of the reaction between water vapor and rare earth disilicate will be discussed. SAND2017-8747 A

5:00 PM

(ICACC-S2-010-2018) Degradation of Ytterbium disilicate EBCs in a high temperature steam environment

N. Rohbeck*[1]; P. Xiao[1]; P. Morrell[2]; R. McIntyre[2]

1. University of Manchester, Material Science, United Kingdom
2. Rolls-Royce, United Kingdom

The development of a suitable Environmental Barrier Coating (EBC) system is necessary for the protection of SiC/SiC Ceramic Matrix Composites (CMCs) from water vapour recession in turbine engines. To-date, a number of different systems have been developed to protect the SiC/SiC composite material which have met with varying success. The most recent EBC systems to be considered are based on rare earth silicates as they possess many of the properties desired: well matched coefficient of thermal expansion to the SiC/SiC material; good high-temperature stability; low water vapour recession rates; are compatible with production ready application technologies. This work focusses on air-plasma sprayed environmental barrier coatings consisting of ytterbium disilicate deposited onto an air plasma sprayed silicon bond coat and studies the development of the micro- and phase- structure of the coatings from the as deposited state and their behaviour under dry air and water vapour thermal ageing. This study also reviews the degradation mechanisms and the changes in stresses in the coatings measured by X-ray methods following ageing isothermally at 1350°C in a 95% water vapour 5% argon atmosphere for varying times.

5:20 PM

(ICACC-S2-011-2018) Formation of $Yb_2Si_2O_7$ layer by surface treatment of SiC

T. Hinoki*[1]; S. Yanagawa[1]; K. Kawasaki[1]; F. Shinoda[1]

1. Kyoto University, Japan

Silicon carbide composites are utilized for a jet engine due to excellent high temperature mechanical properties. However environmental barrier coatings are required in particular for high temperature steam. Ytterbium silicate is one of the candidate material for the coating. The $Yb_2Si_2O_7$ layer was formed by surface treatment of SiC in high temperature air or steam. The SiC was fabricated by liquid phase sintering or reaction sintering containing Yb_2O_3.

S3: 15th International Symposium on Solid Oxide Fuel Cells (SOFC): Materials, Science and Technology

System Design and Demonstration

Room: Crystal
Session Chair: Narottam Bansal, NASA Glenn Research Center

1:30 PM

(ICACC-S3-001-2018) Overview of DOE Office of Fossil Energy's Solid Oxide Fuel Cell Program (Invited)

P. Burke*[1]; S. D. Vora[1]

1. National Energy Technology Laboratory, Department of Energy, USA

The mission of the U.S. Department of Energy is to ensure America's security and prosperity by addressing its energy, environmental and nuclear challenges through transformative science and technology solutions. Supporting this mission is the Office of Fossil Energy's Solid Oxide Fuel Cell (SOFC) Program. Administered by the National Energy Technology Laboratory, the SOFC Program is committed to developing efficient, low-cost electricity from natural gas or coal with intrinsic carbon capture capabilities for distributed generation (DG) and utility-scale applications, maintaining cell and core technology research to increase the reliability, robustness, and durability of cell, stack, and system technology, and providing the technology base to permit cost-competitive DG applications. The SOFC Program maintains a portfolio of research, development, and demonstration projects that address the technical issues facing the commercialization of SOFC technology and a series of increasingly larger demonstration projects intended to validate the solutions to those issues. The status of these research programs, the status of the program's integrated systems tests, and the roadmap to deploy a MWe-class natural gas fueled DG system, will be presented.

2:00 PM

(ICACC-S3-002-2018) All-Ceramic SOFC Technology by Saint-Gobain: Progress in Architecture and Performance (Invited)

A. Sarikaya*[1]; B. Barry[1]; B. Feldman[1]; Y. Takagi[1]; J. Pietras[1]; S. Poizeau[1]

1. Saint-Gobain, USA

Saint-Gobain, the global leader in material processing and manufacturing innovation, creates and delivers innovative solutions to the growing energy and environment markets. With annual sales over $43BN, Saint-Gobain ranks among the world's top 100 companies. Energy generation using solid oxide fuel cells (SOFCs) is a strategic program for Saint-Gobain. The novel all-ceramic SOFC technology is developed and manufactured to exceed the reliability, durability and cost targets for residential and commercial distributed power generation applications. Ultra-thin ceramic interconnects, simplified stack-supported design and multi-cell co-firing processes allowed achieving substantial improvements in the operational reliability and reduction in manufacturing cost. Unique design features of the hot-box such as integrated current collection and gas delivery systems are presented along with other aspects for future performance improvements. Updates on Saint-Gobain's SOFC technology, including the improved performance, stable operation for extended hours and the hot-box development are reported. Saint-Gobain's stack is shown to withstand thermal cycles over long-term operation, hundreds of power cycles and realistic redox cycling. Response of the stack to fuels simulating internal and external reforming as well as high fuel utilizations is also presented.

2:30 PM

(ICACC-S3-003-2018) Solid Oxide Fuel Cell Development at FuelCell Energy (Invited)

J. M. Barton*[1]; H. Ghezel-Ayagh[1]; E. Tang[2]; A. Torabi[1]

1. FuelCell Energy, USA
2. FuelCell Energy, Canada

Significant progress has been made towards the development of Solid Oxide Fuel Cell (SOFC) at FuelCell Energy Inc. (FCE). The genesis of the advances in the technology is rooted in scale-up of the Company's anode-support technology made by the TSC (Tape Casting-Screen Printing-Sintering) Process. Cell materials development advancements have been made in performance, endurance, cell size (area) scale-up and cost reduction. These advancements led to an enhanced cell performance by reducing cell ASR (Area Specific Resistance) while reducing cell cost through cell design changes and materials reduction. Scaled-up cell size of up to 1000 cm2 have been made and tested with the current baseline cell size of 550 cm2 as the standard unit size for building SOFC stacks. The baseline stack design has been successfully scaled-up from 120-cell 550 cm2 cell area (16 kW nominal) stack. Using standardized manifolds and gas-electrical interconnects, these stacks have been successfully integrated into stack towers and arrays. FCE recently designed, built, and tested a 50 kW fully automated SOFC system under a Co-operative Agreement with Department of Energy. In a follow-on DOE supported project, currently, FCE is engaged in design and construction of larger 200kW units that utilize baseline factory-built stack blocks representative of the future MW-scale power plants.

3:00 PM

(ICACC-S3-004-2018) Development of Portable Solid Oxide Fuel Cell System Driven by Hydrocarbon and Alcohol Fuels (Invited)

H. Sumi*[1]; T. Yamaguchi[1]; H. Shimada[1]; Y. Fujishiro[1]; M. Awano[1]

1. National Institute of Advanced Industrial Science and Technology (AIST), Japan

Solid oxide fuel cells (SOFCs) can, in principle, directly use not only hydrogen but also hydrocarbon and alcohol fuels such as methane, propane, butane, and ethanol. National Institute of Advanced Industrial Science and Technology (AIST), Japan has developed anode-supported microtubular SOFCs with high power density via the New Energy and Industrial Technology Development Organization, Japan (NEDO) project entitled "Advanced Ceramic Reactor Research Project" in FY2005-2009. Then, we developed microtubular SOFC stacks, and demonstrated a proto-type portable SOFC system via NEDO project entitled "Technology Development for Promoting SOFC Commercialization" in FY2013-2014. The microtubular SOFCs using nickel-gadolinia doped ceria (Ni-GDC) anode can be operated continuously for more than 24 h in butane at S/C = 0.044 and 610 °C. However, The Ni-GDC anode deteriorated for 15 h and 2 h at S/C = 0.044 in butane at 660 °C and 710 °C, respectively, due to carbon deposition. In the present work, internal partial oxidation reforming of butane and steam reforming of ethanol were investigated for microtubular SOFCs supported on Ni-GDC anode. And, the second 100 W-class proto-type portable SOFC system driven by liquefied petroleum gas (LPG) and ethanol fuels was demonstrated.

System Design and Demonstration / High Temperature Electrolysis and rSOC

Room: Crystal
Session Chair: Jeffry Stevenson, Pacific Northwest National Lab

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(ICACC-S3-005-2018) DEMOSOFC Project: Results From an Industrial-Size Biogas-Fed SOFC (Invited)

M. Santarelli*[1]; M. Acri[2]; U. Fausone[3]; E. Fontell[4]; M. Gandiglio[1]; S. Giarola[5]; T. Hakala[4]; A. Hawkes[5]; J. Kiviaho[6]; A. Lanzini[1]; E. Lorenzi[2]; M. Rautanen[6]

1. Politecnico di Torino, Italy
2. SMAT, Italy
3. Risorse Idriche, Italy
4. CONVION, Finland
5. Imperial College, United Kingdom
6. VTT Technical Research Centre of Finland, Finland

The EU-funded DEMOSOFC project (www.demosofc.eu) aims to demonstrate the technical and economic feasibility of operating a 174 kWe SOFC in a wastewater treatment plant (WWTP). The fuel for the three SOFC modules (3x58 kWe) is biogas, which is available on-site from the anaerobic digestion of sludge collected from the treated wastewater. A heat-recovery loop allows to recover useful thermal energy from the hot SOFC exhaust gases (90-100 kWth), transferred through a water loop to the sludge. The integrated biogas-SOFC plant includes three main units: 1) the biogas clean-up and compression section; 2) the three SOFC power modules, and 3) the heat recovery loop. Main advantages of the proposed layout is the net electric efficiency of the SOFC, which is in the range 53-55%, and the near-zero emissions in terms of NOx, SOx, VOC, PM. The presented works aims to present results from the first months of operation of the DEMOSOFC system, by showing results in terms of plant electrical production and thermal recovery, integration with the existing plant and loads coverage, performance of the biogas purification unit. The data analysis will lead to first evaluations in terms of criticalities and advantages of biogas-fed SOFC in industrial plants. Furthermore, economic and business perspective will be presented for the proposed plant concept.

4:20 PM

(ICACC-S3-006-2018) Development of Solid Oxide Metal-air Redox Battery for Stationary Energy Storage – A Progress Report (Invited)

K. Huang*[1]

1. University of South Carolina, Mechanical Engineering, USA

Cost-effective and large-scale energy storage technologies are a key enabler of global grid modernization. Viable energy storage solutions can greatly enhance the resilience of the electric grid while preserving its reliability. Energy storage technologies currently being researched, developed and deployed for grid storage applications include pumped hydro and compressed-air energy storage, high-speed flywheels, superconducting magnetic inductors, electrochemical capacitors, traditional and advanced batteries (e.g., lead-acid, metal hydrides, lithium-ion, high-temperature sodium-sulfur, redox flow, and metal-air). Significant advances in materials and designs are still needed for batteries to realize their full potential in advanced energy storage. This presentation reviews recent development of a newly discovered rechargeable solid oxide metal-air battery that combines a regenerative solid oxide fuel cell (RSOFC) and iron/iron oxide redox couple energy store. The RSOFC serves as the electrical discharger/charger, operating between the fuel cell and electrolysis modes, respectively, while the iron/iron oxide redox couple stores the oxygen shuttling between the anode and redox couple mediated by H_2/H_2O oxygen shuttling gas. A brief history and key energy storage characteristics of the new battery under a variety of conditions are particularly presented.

4:50 PM

(ICACC-S3-007-2018) High Performance Solid Oxide Electrolysis Cells (SOECs) for Hydrogen Production

N. Osada*[1]; M. Yoshino[1]; T. Kameda[1]

1. Toshiba Corporation, Power and Industrial Systems R&D Center, Japan

Solid oxide electrolysis cells (SOECs) are expected to provide the highest efficiency of electrolytic production of pure hydrogen, owing to the lower applied voltage due to favorable thermodynamic and kinetic conditions. Furthermore, the Solid Oxide Cells can be operated reversely as solid oxide fuel cells (SOFCs) to generate electricity with high efficiency. We have developed high performance SOECs and evaluated the durability with single cells. In this study, the degradation factors and mechanisms were investigated and it became clear that the SOEC degradation was caused by undesirable solid state reaction between oxygen electrode and electrolyte. The results in detail will be shown in our presentation. A part of this study is carried out in a project commissioned by the New Energy and Industrial Technology Development Organization (NEDO).

5:10 PM

(ICACC-S3-008-2018) SOC co-electrolysis operation

M. Kusnezoff*[1]; S. Megel[1]; N. Trofimenko[1]; M. Jahn[1]; E. Reichelt[1]; A. Michaelis[1]

1. Fraunhofer IKTS, Germany

The efficient high temperature electrolysis is of high interest for potential application in a renewable energy system. The possibility to use high temperature heat for the electrolysis reaction makes the SOEC technology an attractive option for the integration in process concepts for the production of valuable chemicals from renewable energies. Moreover, the SOEC allows for the electrolysis of water and carbon dioxide in a so-called co-electrolysis. The produced syngas from co-electrolysis can be used directly in a subsequent synthesis step, e.g. a Fischer-Tropsch synthesis. The development of such power-to-product processes as well as of necessary components are in focus of current research activities in Europe. The main component is the high temperature electrolyzer (SOEC). CFY stack technology allows for the proof of concept for different operation options utilizing adjusted electrodes on electrolyte supported cells. This stack technology can be applied for SOFC, SOEC and reversible operation (rSOC) and establishes an universal platform for different applications. Performance maps of a MK352 CFY stack in steam (H_2O) and H_2O/CO_2 electrolysis operation show a difference in electric power demand of less than 5 %. This low excess power demand of co-electrolysis together with the advantages of direct syngas production makes it promising for highly efficient integration within a process concept for the production of valuable hydrocarbons.

5:30 PM

(ICACC-S3-009-2018) Performance Test for Anode-supported and Metal-supported Solid Oxide Electrolysis Cell under Different Current Densities

S. Wu[1]; J. Lin[1]; W. Shiu[1]; C. Liu*[1]; T. Lin[1]; R. Lee[1]; H. Ting[1]; H. Lin[1]; Y. Cheng[1]

1. Institute of Nuclear Energy Research, Nuclear Fuels and Materials Division, Taiwan

In this study, a proprietary apparatus developed by INER was used to assess the hydrogen production for anode-supported and metal-supported solid oxide electrolysis cells (SOECs) under different current densities at elevated temperatures. Two types of home-made solid oxide fuel cell (SOFC): anode-supported cell (ASC) with the configuration of NiO-YSZ||YSZ||YSZ-LSM, metal-supported cell (MSC) with the configuration of Ni-Mo||NiO-YSZ||NiO-GDC||LDC||LSGM||LDC||SDC-SSC, were tested under SOEC mode. Additionally, a commercial available ASC type SOFC was measured as a benchmark for comparing the electrolytic performance. All the cells were in the same size of 10×10 cm^2, and the performance tests

were conducted with single cell at 700, 750, and 800 °C, respectively, under the gas flow of 2000 sccm air at the air electrode and 805.3 sccm 90%H_2O/10%H_2 mixture at the hydrogen electrode. Results indicated that the electrical efficiency for both ASC and MSC operated under SOEC mode increase with increasing the current density applied at elevated temperatures. However, the H_2O/H_2 ratio has a lot of influence on the stability of cell voltage. The calculated electrical efficiencies, which ignored the energy consumption of system components, are 108%, 134%, and 143% for the home-made ASC, MSC, and commercial ASC, respectively, under a current density of 350 mA/cm^2 at 800 °C.

S4: Armor Ceramics - Challenges and New Developments

Quasi-static and Dynamic Behavior I

Room: Coquina Salon F
Session Chairs: Jerry LaSalvia, Army Research Laboratory; Sikhanda Satapathy, Army Research Laboratory

1:40 PM

(ICACC-S4-001-2018) An Extended Mohr-Coulomb Model for Ultrahigh Pressure Response of Structural Ceramics

G. Subhash*[1]

1. University of Florida, Mechanical and Aerospace Engineering, USA

The traditional Mohr-Coulomb model for brittle materials assumes a linear increase in strength with confinement pressure. While this model is valid for low-pressure deformation, experimental observations have revealed that the strength of a ceramic saturates beyond a critical level. Constitutive models, such as the JH-2 (developed by Johnson and Holmquist), for pressures up to HEL often force the curve to take an unusual shape regardless of the experimental data. It also fails at high pressures past the HEL, as it predicts a continuous increase in strength. Further, the constants used in the model have to be derived for each ceramic through, often expensive, experiments. To overcome these limitations, an extended Mohr-Coulomb model has been developed to capture the nonlinear pressure-dependent shear strength of ceramics at pressures beyond HEL. In this model, a single exponential curve represents the failure envelope of all intact ceramics across a wide range of pressures with a single set of universal empirical constants. Such a model reduces the requirement on experimental data. A sensitivity analysis is performed to validate the universal constants for numerous ceramics. It is shown that the single set of constants can capture the pressure-shear response of a wide range of brittle materials, and that the extended Mohr-Coulomb model out-performs the JH-2 model in most cases.

2:00 PM

(ICACC-S4-002-2018) Generation of polycrystalline microstructures for the discretization on-the-fly of FE models for multi-scale simulations

S. Falco*[1]; N. Bombace[1]; N. Petrinic[1]

1. University of Oxford, United Kingdom

The behaviour of ceramics, and more generally of brittle materials, depends strongly upon the microstructural features that determine the initiation and propagation of crack. The difficulties in the direct observation of the crack initiation and propagation at the microscopic level, especially under dynamic loading conditions, as well as the complexity in the manufacturing materials with defined features, hinders the research of the microstructural configuration with increased performance under specific loading conditions. The in-house developed adaptive concurrent multi-scale method offers the possibility to efficiently simulate the failure of relatively large structures with sub-grain spatial resolution, by adaptively combining two models of the same structure at two different scales. The larger (macro) scale is used to simulate the elastic behaviour of the grain assembly. The inter-granular crack initiation and propagation, however, is simulated at the lower (micro) scale which is only activated if a certain failure condition is reached within the single macro element. The method presented in this paper allows to discretise on-the-fly the macro-elements with representative polycrystalline microstructures to simulatie the deformation and failure of large structures under dynamic loading conditions due to crack initiation and propagation at the grain scale.

2:20 PM

(ICACC-S4-004-2018) Comparative Analysis of Response to High Velocity Impact of New Ceramic Materials Based on High-boron Compounds Developed at IPMS NASU

E. Kartuzov*[1]; V. Kartuzov[1]; S. Ivanov[1]; B. Galanov[1]

1. Frantsevich Institute for Problems in Materials Science NAS of Ukraine, Ukraine

A number of ceramic materials developed and produced by Frantsevich Institute for Problems in Materials Science, National Academy of Sciences of Ukraine are analyzed and discussed. Boron carbide based ceramic materials and other high-boron compounds (AlC_4B_{24}, $AlMgB_{14}$ etc.) enhanced the mechanical properties (hardness, strength, crack resistance) compared to conventional ones, and can be promising materials for use as protective components during high-velocity impact. Applicability of such materials is determined by both technological and economical factors as well as by their increased resistance to penetration. On the basis of comparative analysis of penetration models using the formalism of Alekseevskii-Tate equation and its modifications proposed by the authors, an indicative factor for shock resistance estimation is introduced. For a number of materials of practical interest this factor changes within the range of 30-70 and for the above said materials it arrives values of ~100, which makes these materials an attractive for armor applications. The results of investigation of mechanical properties of a number of new ceramic materials developed in IPMS are presented. A prognosis of their ballistic properties with the introduced indicative factor is made.

2:40 PM

(ICACC-S4-003-2018) Mesoscale Simulations of Boron Carbide Subjected to Shockwave Propagation

B. Aydelotte*[1]; J. Sietins[1]; C. Hofmeister[1]; T. Holmquist[2]

1. US Army Research Laboratory, USA
2. Southwest Research Institute, USA

Boron carbide is a ceramic of great interest due to its high strength, low density, high hardness, and of course, its tendency to undergo amorphization during failure. Commercial boron carbide commonly contains a variety of defects including pores, carbon inclusions, and aluminum nitride inclusions, but what role do the defects play? Certainly they can be failure initiation sites, but do they influence the formation of amorphous material? In order to begin to address this question, we have performed high resolution XCT (X-ray computed tomography) scans to quantify the internal defect structure of samples of commercial boron carbide. Select portions of this data were converted into a form where mesoscale simulations of shock wave propagation could be conducted to explore the role that defects play in the failure process. These simulations provide insight into the effect of these defects on the local stress state during shock wave propagation and their role in contributing to or diminishing the formation of amorphous material.

Quasi-static and Dynamic Behavior II

Room: Coquina Salon F
Session Chair: Jeffrey Swab, Army Research Laboratory

3:20 PM

(ICACC-S4-005-2018) Compression Strength of Ceramics

J. Swab*[1]; C. Meredith[1]; W. R. Gamble[1]

1. Army Research Laboratory, USA

The intrinsic compression strength of ceramics and glasses can be very difficult to determine. The specimen geometry and fixture used to apply the load, if not properly designed, can result in the generation of tensile stresses that lead to premature fracture and misleadingly low strength values. Often the compression strength is inferred from hardness values but this is not appropriate for ceramic materials. The compression strength is an input parameter in numerous modeling and simulation packages used to predict performance in some applications. As a result it is imperative that the compressive strength of the candidate ceramics be properly and accurately measured. Several ceramics and glasses were machined into dumbbell-shaped specimens that were designed to induce fracture from within the gage section while minimizing the stress concentrations that can lead to the undesirable tensile stresses. Quasi-static experiments were performed using a screw-driven load frame and the fracture process was recorded with a high speed camera. Dynamic experiments were performed using a split-Hopkinson pressure bar setup with bars having the same diameter as the specimen and an ultra-high speed camera to record the fracture process. This presentation will summarize the results to date and identifies if a change in strain rate influences the compression strength value.

3:40 PM

(ICACC-S4-006-2018) Novel Mechanical Response of Hard Nanocrystalline Ceramics with Grain Sizes Below 30nm

J. Wollmershauser*[1]; H. Ryou[2]; J. Drazin[2]; K. Wahl[1]; E. Gorzkowski[1]; B. Feigelson[1]

1. U.S. Naval Research Laboratory, USA
2. American Society for Engineering Education Postdoctoral Research Fellow sited at U.S. Naval Research Laboratory, USA

Recent leaps in ceramics processing technology has allowed fabrication of dense ceramics with grain sizes well below 30nm and the works generally reveal that hardness increases with a reduction in grain size. However, some work suggests that hardness finds its maximum at 30nm, while other work determines that hardness continues to increases with decreasing grain size (at least down to ~7nm). Since ballistic performance is tied to hardness and strength, it would be prudent to determine the intrinsic grain size-hardness relationship in nanocrystalline ceramics from a DoD perspective. This work uses a novel environmentally-controlled pressure-assisted sintering approach to synthesize magnesium aluminate ceramics with grain sizes ranging from ~4nm to ~40nm. Careful indentation studies with an instrumented indentor reveal multiple distinct size effect regions in the mechanical response. Furthermore, density measurements and work-energy calculations suggest a transition to unique strain accommodation in nanocrystalline ceramics with very small grain sizes. Importantly, the new strain accommodation regime appears to have different dissipative energy characteristics than larger grain sized ceramics.

4:00 PM

(ICACC-S4-007-2018) Influence of stress states during amorphization of single crystal boron carbide

J. Ligda*[1]; K. D. Behler[1]; J. Lloyd[1]; V. Domnich[2]; J. LaSalvia[1]; B. Schuster[1]

1. US Army Research Laboratory, USA
2. Rutgers University, USA

Spherical nanoindentation was performed on single crystal boron carbide to delay the on-set of in-elastic deformation. A spherical tip geometry creates a well-defined transition from elastic to in-elastic deformation in the ceramic, and by performing tests to different maximum loads the initiation of in-elastic deformation is controlled. Identifying the in-elastic damage as a result of cracking or amorphization is done using Raman spectroscopy and scanning electron microscopy. The degree and location of any amorphous bands under the indents are characterized by Raman spectroscopy and transmission electron microscopy. The stress states underneath the indent are modeled to identify if the orientation of the damage bands follows a crystallographic direction or stress trajectories.

4:20 PM

(ICACC-S4-008-2018) In-situ Investigation of Shear Induced Amorphization in Boron Carbide with Varying B/C Ratios

V. Domnich*[1]; M. C. Schaefer[1]; R. A. Haber[1]

1. Rutgers University, USA

High hardness and low density make boron carbide (B_4C) an ideal candidate material for lightweight armor applications. However, while B_4C clearly outperforms other armor ceramics in ballistic testing involving small arms ammunition, it experiences a drop in ballistic efficiency when the high-density, high-velocity threats are used. This observation has been linked in the literature to the loss of shear strength in B_4C, possibly related to its shear-induced amorphization. Amorphized B_4C has been demonstrated to form in the ballistically impacted material, as well as in the material subjected to quasiuniaxial loading in the Diamond Anvil Cell (DAC). Further, theoretical studies imply that shear-induced amorphization in B_4C may be mitigated if materials with higher B/C ratio, e.g., $B_{6.5}C$, are synthesized. Here, we report on the results of the investigation of the variation in amorphization pressure threshold in boron carbide samples with B/C ratios varying from 4 to 9, produced by hot pressing of the B_4C/B powder mixes. To achieve high deviatoric stresses required for the activation of the amorphization mechanism, the samples were crushed into powders and compressed in the DAC without pressure transmitting medium. In situ Raman spectroscopy was used for the identification of the amorphization threshold. The recovered samples were further analyzed by means of electron microscopy.

4:40 PM

(ICACC-S4-009-2018) Influence of Grain Size and CNT addition on Static and Dynamic Properties and Amorphization of Boron Carbide

M. DeVries*[1]; G. Subhash[1]

1. University of Florida, Mechanical and Aerospace Engineering, USA

To mitigate the deleterious effects of amorphization in boron carbide, numerous strategies have been adopted: grain size reduction to nano scale, doping with second phases (e.g., Si), addition of carbon nanotubes (CNT) etc. Here, we investigate the influence of grain size and CNT addition on the amorphization response of boron carbide. Indentation hardness and uniaxial compressive strengths were determined under quasi-static and dynamic strain rates. Micro-Raman spectroscopy was used to investigate and spatially map the relative intensity of amorphization in each material. It was found that both strategies, i.e., reduction in grain size and addition of CNTs, improve the resistance to amorphization. Results of on-going analysis using SEM and TEM will also be presented.

5:00 PM

(ICACC-S4-010-2018) High Strain Rate Multi-axial Loading Behavior of Granular Phase Boron Carbide

X. Sun*[1]; A. Tonge[3]; K. Ramesh[2]; J. LaSalvia[3]

1. Johns Hopkins University, Mechanical Engineering, USA
2. Hopkins Extreme Materials Institute, USA
3. US Army Research Laboratory, USA

Boron carbide as an advanced structural ceramic has been widely used in applications such as armor protection due to its low density and high strength. Plasticity in boron carbide is negligible, making it intrinsically very brittle in which crack nucleation, propagation and subsequent flow of granular phase are the dominant deformation mechanisms, especially at high strain rate and complex stress states. Granular flow within communized zone during impact has critical effects on ballistic performance. Many experimental and modeling efforts have been devoted to understanding of the crack mechanism, but there has not been much of focus on the granular flow of comminuted boron carbide at high strain rate. In current work, we design and perform ultra high-strain-rate pressure-shear plate impact experiments on a commercial boron carbide powder with an average grain size of 0.7 micron. The granular boron carbide powder is deformed at a shearing rate as high as 5×10^5 s^{-1} with a superimposed hydrostatic pressure at 1.4GPa. The shear stress is 240MPa. Another pressure shear data shows a higher shear stress at 270MPa under lower shear strain rate but higher pressure at 1.75GPa. The normal pressure and shear strain rate effects on behavior of granular boron carbide are examined experimentally. Post-mortem characterization provides useful insights of mechanisms during the deformation.

5:20 PM

(ICACC-S4-011-2018) Influence of Microstructure on the Ballistic Behavior of Alumina

B. Koch*[1]; C. Lo[1]; T. Sano[2]; J. D. Hogan[1]

1. University of Alberta, Edmonton, Mechanical Engineering, Canada
2. US Army Research Laboratory, Weapons and Materials Research Directorate, USA

Understanding how the microstructure of an advanced ceramic affects the mechanisms of failure under high strain rate loading is a key piece of modelling and design. In this presentation, we make efforts towards better understanding dynamic failure mechanisms in advanced ceramics. Two grades of alumina are characterized through use of techniques such as scanning electron microscopy, electron backscatter spectroscopy, and energy dispersive X-ray spectroscopy to determine their microstructure. Samples of the alumina were then compressed to failure using quasi-static and compression bar loading to determine compressive strength, elastic modulus, and bulking behavior. Characterization and experimentation efforts are discussed in terms of design of improved ceramics for ballistic applications.

5:40 PM

(ICACC-S4-012-2018) Quantitative Visualization of Fracture and Failure of Soda-lime Glass

H. V. Tippur*[1]; B. M. Sundaram[1]

1. Auburn University, Department of Mechanical Engineering, USA

Transparent ceramics such as soda-lime glass pose unique challenges for full-field optical techniques to evaluate deformations, stresses and failure behavior. Low toughness and high stiffness are among the factors responsible for these challenges as deformations tend to be too small and confined to a region near a 'mathematically' sharp crack growing at speeds in excess of 1500 m/s. Need for good birefringence, sophisticated optics, or lack of measurement sensitivity are some of the factors against legacy techniques to study brittle ceramics'. Motivated by these, the feasibility of a full-field optical technique called the Digital Gradient Sensing (DGS) is demonstrated for evaluating deformations in glass under stress wave loading conditions. DGS is capable of quantifying angular deflections of light rays, as small as a few micro-radians, caused by stresses. High-speed photography (>1 Mfps) combined with DGS and a Hopkinson bar are used to load soda-lime glass specimens. The measurements are processed to obtain crack-tip parameters near both stationary and dynamically growing cracks. The instantaneous histories of crack speed (V), stress intensity factors and energy release rate (G) are extracted and G-V plots are obtained. Several interesting observations on fracture parameters and fractography leading up to crack branching are revealed.

S6: Advanced Materials and Technologies for Direct Thermal Energy Conversion and Rechargeable Energy Storage

Solid Electrolytes and All-solid-state-batteries I

Room: Tomoka A

Session Chair: Palani Balaya, National University of Singapore

1:30 PM

(ICACC-S6-001-2018) Garnet-type ionic conductors for all-solid-state lithium ion batteries (Invited)

X. Guo*[1]

1. Huazhong University of Science and Technology, Materials Science and Engineering, China

Owing to its high ionic conductivity, $Li_7La_3Zr_2O_{12}$ (LLZO) garnets are promising electrolytes for all-solid-state lithium ion batteries. Herein, Ga is doped at the Li site and Rb is doped at the La site of LLZO to enhance the lithium-ion conductivity. The $Li_{6.20}Ga_{0.30}La_{2.95}Rb_{0.05}Zr_2O_{12}$ electrolyte exhibits a lithium-ion conductivity of 1.62 mS/cm at room temperature, which is the highest conductivity reported until now. All-solid-state lithium ion batteries are constructed from the $Li_{6.20}Ga_{0.30}La_{2.95}Rb_{0.05}Zr_2O_{12}$ electrolyte, metallic Li anode and $LiFePO_4$ active cathode. To reduce the interfacial resistance, $Li(CF_3SO_2)_2N$ electrolytic salt is added in the cathode. The battery shows a high initial discharge capacity of 152 mAh/g and good cycling stability with 110 mAh/g retained after 20 cycles at a charge/discharge rate of 0.05 C at 60 °C. Therefore, the $Li_{6.20}Ga_{0.30}La_{2.95}Rb_{0.05}Zr_2O_{12}$ garnet is very promising for all-solid-state lithium ion batteries.

2:00 PM

(ICACC-S6-002-2018) On the Manufacturing and Operation of Inorganic All-solid-state Batteries (Invited)

S. Uhlenbruck*[1]; C. Tsai[1]; C. Dellen[1]; S. Möller[1]; S. Lobe[1]; A. Windmüller[1]; M. Finsterbusch[1]; O. Guillon[1]

1. Forschungszentrum Juelich, Institute of Energy and Climate Research, Germany

Current lithium ion battery technology for energy storage has reached excellent performance levels, nevertheless further development with regard to stable operation and high energy density is still required. Some of these challenges are associated with the properties of the liquid organic electrolyte. Its substitution by solid inorganic electrolytes leads to so-called all-solid-state batteries. In general, solid-state electrolytes take advantage of high chemical and electrochemical stability. Thus they may lead to high energy density battery cells: For example, by using lithium metal as anode, and cathode active materials with potentials of higher than 5 V vs. Li/Li^+, the energy density can be significantly increased. Nevertheless, new challenges with regard to manufacturing and operation have emerged. This presentation focuses on three issues of current solid-state battery technology when using garnet type solid electrolyte: (i) the chemical stability between solid electrolytes and active materials, (ii) the growth of Li metal filaments in solid-state electrolytes, and (iii) how ambient conditions can affect battery components

during processing. Results of various analysis methods, in particular depth-resolved methods like secondary ion mass spectrometry, nuclear reaction analysis and Rutherford backscattering spectrometry will be presented and discussed in this talk.

2:30 PM

(ICACC-S6-003-2018) Sulfide materials for Li-S and all-solid-state batteries (Invited)

A. Sakuda*[1]; A. Hayashi[1]; M. Tatsumisago[1]

1. Osaka Prefecture University, Graduate School of Engeneering, Department of Applied Chemistry, Japan

All-solid-state lithium secondary batteries using sulfide-based solid electrolytes are anticipated as next-generation batteries on the road to attaining improved safety and long cycle life. It is important for improving the performance of all-solid-state batteries to fulfill (a) low-resistance electrode–solid electrolyte interface, (b) homogeneous lithium-ion and electron conduction pathway, (c) suppression of damage to the electrode active materials, and (d) higher electrode active material content. Obviously, (e) the use of a highly conducting solid electrolyte is the most important point. In this presentation, we will present the unique features of sulfide materials suitable for battery materials. For example, sulfide-based solid electrolytes and electrode materials are deformable by pressing the powder at room temperature. This unique mechanical property yields the formation of a desirable solid-solid intimate contact between the electrode and electrolyte by room-temperature pressing. The polysulfide-based electrode materials show a large reversible capacity based on the formation and deformation of S-S disulfide bonds and changes in the coordination number.

Solid Electrolytes and All-solid-state-batteries II

Room: Tomoka A

Session Chair: Valerie Pralong, CNRS ENSICAEN

3:20 PM

(ICACC-S6-004-2018) Rational design of advanced materials for solid-state Li-metal batteries (Invited)

Y. Guo*[1]

1. Institute of Chemistry, Chinese Academy of Sciences, China

Rechargeable solid-state Li metal batteries have become attractive candidates for the next-generation rechargeable electrochemical energy storage systems with high energy densities driven by an increasing demand on storage devices with higher energy outputs and better safety. Here, we will show our recent progress on improving this emerging battery system by designing advanced materials, including nanostructured hybrid lithium metal anodes, solid composite electrolytes, and nanostructured composite cathodes. Regarding to the uneven Li deposition, unstable solid electrolyte interphase formation, and infinite change of relative dimensions of the Li metal anodes, 3D current collectors (e.g., 3D Cu), nanostructured carbon materials, artificial solid electrolyte interphase (SEI) layers (e.g., Li_3PO_4, Al-containing SEI), functional electrolyte additives (e.g., Al-based colloidal particles, ionic liquids), as well as bifunctional solid polymer electrolytes and LLZO-coated thin film electrolytes with both high Li ion conductivity and high mechanical strength have been demonstrated feasible as functional materials for stable plating of Li to address the above issues. We hope that our perspectives can shed lights on the reasonable design of key-enabling materials for solid-state lithium-metal batteries, and contribute to a helpful discussion for the technology beyond classical Li-ion batteries.

3:50 PM

(ICACC-S6-005-2018) Microstructure and phase control of β”-alumina ceramic electrolytes for sodium salt batteries

M. V. Heinz*[1]; M. Bay[1]; K. Fiore[2]; N. Zanon[2]; U. F. Vogt[1]; C. Battaglia[1]

1. Swiss Federal Laboratories for Materials Science and Technology, Materials for Energy Conversion, Switzerland
2. FZSonick, Switzerland

Known as a fast ion conductor since the 1970s, state-of-the-art β”-alumina electrolytes with conductivities of ~0.2 S/cm at 300°C are key components in sodium-metal halide and sodium-sulfur batteries. Furthermore, the material is re-gaining attention also for application at ambient temperatures, e.g. for all-solid state batteries. However, β”-alumina ceramics are generally not phase pure materials but consist of a complex arrangement of grain and grain boundary phases. As a result, it is rather difficult to prepare them with defined properties: in fact, ion conductivities reported in literature for Na-β”-alumina based ceramics vary by more than a factor of 100, mainly within the conductivity window from 0.005 to 0.2 S/cm at 300°C. In most studies, these variations are vaguely linked to composition, sample density, phase purity, and/or grain size. In this study we re-investigate how different processing conditions affect phase content and microstructure of Na-β”-alumina ceramics. We discuss the competition between densification, exaggerated grain growth, and Na loss and propose measures to improve the preparation of β”-alumina ceramics to obtain both high ionic conductivity (up to 0.4 S/cm at 300°C, >1 mS/cm at room temperature) and high mechanical stability (~200 MPa flexural strength).

4:10 PM

(ICACC-S6-006-2018) Anisotropic properties of Na-β”-Alumina+ YSZ composite synthesized by vapor phase method

L. Ghadbeigi*[1]; T. D. Sparks[1]

1. University of Utah, Material Science and Engineering, USA

Composites of Na-β”-alumina + YSZ was prepared by vapor phase conversion. Prior to vapor phase conversion samples were sintered for 3 hours at 1500°C for sample A and at 1700°C for sample B. Both samples A and B was converted by heat treatment of as sintered samples in the packing powder of Na-β”-alumina at 1450°C for 10 hours. Phase identification, morphological analysis and electrical conductivity was conducted by performing X-ray diffraction, scanning electron microscopy and electrochemical impedance spectroscopy. All analysis confirmed the presence of texturing in samples A and B. Results showed the alignment of the plate-like crystals perpendicular to disc faces with the conduction plane parallel to the synthesis progression that provides the fastest Na ion transport thus facilitating fast conversion. An anisotropy factor of 5.7 for samples sintered at 1500°C was observed compared to an anisotropy factor of 1.8 for samples sintered at 1700°C. Quantitative phase analysis of XRD also showed the previously unobserved evolution of a small amount of monoclinic phase (7 wt%) in sample B after conversion which was likely due to the results of internal stresses and contribution of surface energy change. It was concluded that the transformation of a portion of the YSZ to monoclinic zirconia phase might be responsible for the slow conversion kinetics by increasing grain size.

4:30 PM

(ICACC-S6-007-2018) Micro-mechanical characterization of $Li_7La_3Zr_2O_{12}$ for solid state batteries

J. F. Nonemacher*[1]; M. Finsterbusch[1]; J. Malzbender[1]

1. Forschungszentrum Jülich, Institute of Energy and Climate Research (IEK), Germany

Conventional lithium ion batteries are based on organic liquid electrolytes due to their very high ionic conductivity; however, they possess drawbacks in terms of leakage, toxicity and flammability. Solid state Li ion conductors, such as the garnet type ceramic

material $Li_7La_3Zr_2O_{12}$, possess a higher thermal stability, larger chemical stability towards metallic Li anodes and high-voltage cathodes and are expected to supress dendrite formation. During operation thermo-chemical cycling generates internal stresses which could induce micro-cracking and mechanical failure that would shorten the cyclability. Therefore, the mechanical reliability of solid electrolytes is important to achieve long-term operation of solid state batteries. Thus, the presented work focuses on a mechanical assessment of various doped $Li_7La_3Zr_2O_{12}$ electrolytes based on depth-sensitive indentation. Results on elastic modulus, hardness and fracture toughness with their actual dependency on the penetration depth are given. Hence, results are discussed with emphasis on the effect of local deformation behaviour. The tests were based on different procedures using either constant or loading cycles, revealing different sensitivities for local/global behaviour related to grains, grain boundaries and pores. The interpretation of the results is supported by complementary optical and electron microscopy as well as X-ray diffraction assessment.

4:50 PM

(ICACC-S6-008-2018) Ionic and Thermal Conductivity in Ceramic Solid Electrolytes for Li-Ion Cells

M. Rohde*[1]; Y. Cui[1]; C. Ziebert[1]; H. J. Seifert[1]

1. Karlsruhe Institute of Technology, Institute for Applied Materials, Germany

Within the current development state of Li-ion batteries liquid electrolytes are applied which require a sophisticated thermal management. In contrast to the organic liquids solid electrolytes made of a glass-ceramic are not flammable. Even at higher temperatures they are very stable and therefore reduce the efforts of a sophisticated thermal management. However, one of the main obstacles for the application of solid electrolytes in Li-ion cells is the relatively low ionic conductivity. Within this work we have studied two glass-ceramic systems, which are candidate materials for solid state electrolytes. LAGP (Li_{1+x} Al_xGe_{2-x} $(PO_4)_3$, x=0.5) and LATP (Li_{1+x} Al_xTi_{2-x} $(PO_4)_3$, x=0.5) substrates were prepared using a melt quenching route and by applying different compaction methods. The glass chemical composition and the developed microstructure in LAGP glass-ceramics were very critical to the thermal and ionic conductivity. The measured values of the ionic conductivities were in the range of 10^{-4} to 10^{-3} W^{-1} cm^{-1} at room temperature, but increased at higher temperatures. The thermal conductivity values are very sensitive to the details of the heat treatment. It was shown that a higher ionic conductivity is coupled with a higher thermal conductivity. Furthermore, the temperature dependence of the thermal conductivity cannot be explained by a single mechanism or by an "ionic" Wiedemann-Franz law.

5:10 PM

(ICACC-S6-009-2018) Lithium Diffusion in Lithium Garnet Oxide $Li_5La_3Ta_2O_{12}$: A combined quasi-elastic neutron scattering and molecular dynamics study

W. Lai*[1]; M. Klenk[1]; S. Boeberitz[1]

1. Michigan State University, Chemical Engineering and Materials Science, USA

Solid lithium ionic electrolytes are attractive alternatives to liquid electrolytes to achieve the uttermost safety against leakage and flammability in lithium-ion batteries. Lithium garnet oxides are a class of materials that are very promising as solid electrolytes for lithium-ion batteries. A wide variety of experimental (diffraction, total-scattering, impedance spectroscopy, NMR, muon-spin relaxation, etc) and computational techniques have been applied to the study of structure and dynamics of lithium garnet oxides. In this work, we combine two complementary probes, quasi-elastic neutron scattering (QENS) and molecular dynamics (MD) simulation, to investigate the diffusion mechanism of a model garnet $Li_5La_3Ta_2O_{12}$. The half-width-half-maximum values (HWHM) of QENS signals from both experiments and MD are comparable to each other. Q-dependence of HWHM values suggests the diffusion is of jump type and can be fitted into a Singwi-Sjolander model where lithium oscillates inside a cage followed by a jump. The residence time obeys an Arrhenius relation on the temperature while the variation of jump distance suggests that diffusion occurs between tetrahedral and octahedral sites.

S7: 12th International Symposium on Functional Nanomaterials and Thin Films for Sustainable Energy Harvesting, Environmental, and Health Applications

Nanomaterials for Photocatalysis, Solar Hydrogen and Thermoelectrics I

Room: Coquina Salon C

Session Chairs: Teresa Andreu, Catalonia Institute for Energy Research (IREC); Francesco Enrichi, Centro Studi e Ricerche E. Fermi (Italy) and Luleå University of Technology (Sweden)

1:30 PM

(ICACC-S7-001-2018) Effects of Composition, Local Structure and in-situ Structural Evolution on the Catalytic Properties of Cobalt and Modified Cobalt (Oxy)hydroxides for the Oxygen Evolution Reaction (Invited)

Z. Chen[1]; C. X. Kronawitter[2]; I. Waluyo[3]; L. Cai[4]; B. E. Koel*[1]

1. Princeton University, Chemical and Biological Engineering, USA
2. University of California, Davis, Chemical Engineering, USA
3. Brookhaven National Laboratory, National Synchrotron Light Source II, USA
4. Xi'an Jiaotong University, State Key Laboratory of Multiphase Flow in Power Engineering, China

Development of improved catalysts for the oxygen evolution reaction (OER) is important for emergent technologies in energy conversion and storage. Understanding reaction mechanisms and correlations with catalyst properties provides rational design principles to accelerate catalyst development. The effects of doping and processing on the catalytic properties of cobalt (oxy)hydroxides (CoOOH) illustrated key structure-activity correlations. Catalyst performance of CoOOH and M-modified CoOOH (M=Ni, Mn) increased with increasing concentration of dispersed Ni ions, but not Mn. Ni incorporation reduced charge transfer resistances and improved stabilities of surface intermediates, but Mn incorporation impeded such stabilization. Using ambient pressure photoelectron spectroscopy, we directly observed better stability of surface hydroxyl groups with Ni incorporation. Extensive surface hydroxylation under mild conditions (27 °C, 1 torr H_2O) indicated a low barrier to phase transformation. Investigation of catalyst structural evolution during OER using operando Raman spectroscopy identified the active structure of related $NiCoO_xH_y$ catalysts as NiOOH-CoO_x, which can be formed by an irreversible transformation of spinel Co_3O_4 to fcc-CoO followed by a reversible conversion of the catalyst to NiOOH-CoO_x.

2:00 PM

(ICACC-S7-002-2018) Composite nanostructures for high-efficiency Sunlight conversion (Invited)

A. Vomiero*[1]

1. Lulea University of Technology, Engineering Sciences & Mathematics, Sweden

Composite ceramic nanostructures can be efficiently applied for Sunlight conversion. In most of the applied systems, like excitonic solar cells and photoelectrochemical cells to produce solar fuels, nanomaterials can play a critical role in boosting photoconversion efficiency by ameliorating the processes of charge photogeneration,

exciton dissociation and charge transport. Several strategies can be pursued, including broadening of light absorbance to reduce solar light losses, fastening exciton dissociation and charge injection from the photoactive medium to the charge transporting materials, reducing charge recombination during charge transport and collection at the electrodes. In this lecture, a few examples of application of nanocomposites will be thoroughly discussed, highlighting the role of interface engineering to improve the efficiency of energy conversion from Sunlight to electric power and/or chemical fuels.

2:20 PM

(ICACC-S7-003-2018) Modification and deposition of metaloxide layers via Plasma Enhanced Chemical Vapor Deposition

Y. Gönüllü*[1]; S. Mathur[1]; T. Fischer[1]

1. University of Cologne, Institute of Inorganic Chemistry, Germany

The PE-CVD is a unique technique for thin film deposition, since it provides a good control over several parameters (time, plasma power and reactive gas composition) and therefore over the properties of the resulting films. In addition PE-CVD processes show a high reproducibility and they are scalable for large area productions. It finds applications in growth and processing of nano-materials, such as semiconductor thin films or carbon structures like graphene, carbon nanotubes (CNT), or DLC. Since the PE-CVD is a low temperature process it is possible to deposit under mild conditions onto sensitive materials like polymers. In this work we present the deposition and modification of semiconducting metal oxides (TiO_2, Fe_2O_3, SnO_2) for photoelectrochemical (PEC) hydrogen production. Furthermore the as deposited films were modified using plasma techniques or supporting with graphen layer. Namely those modifications were the partial reduction of the metal oxides by hydrogen plasma, the doping with different metal cations. All this techniques allowed the selective tuning of the optical and electrical properties of the films and therefore lead to a substantially enhanced PEC performance.

Nanomaterials for Photocatalysis, Solar Hydrogen and Thermoelectrics II

Room: Coquina Salon C
Session Chairs: Alberto Vomiero, Lulea University of Technology; Yakup Gönüllü, University of Cologne

3:20 PM

(ICACC-S7-004-2018) Downconversion enhancement by Ag nanoaggregates in Tb^{3+}/Yb^{3+} codoped silica-zirconia sol-gel glasses and glass-ceramics for solar cells (Invited)

F. Enrichi*[1]

1. Luleå University of Technology (Sweden) and Centro Studi e Ricerche E. Fermi (Italy), Sweden

The frequency conversion properties of rare earth ions may be enhanced by the presence of metal nanoaggregates/nanoparticles. In this presentation, we report the study of down-converting silica-zirconia glass and glass-ceramic films doped by Tb^{3+}/Yb^{3+} ions and Ag nanoaggregates, which combine the spectral properties of rare-earth-doped materials with the optical sensitizing effects of the metal nanostructures. The preparation of sodium-containing silica-zirconia layers was carried out by sol-gel synthesis, followed by thermal treatments at 700°C (glass) or 1000°C (glass-ceramics). Ag introduction was then obtained by ion-exchange in a molten salt bath and the samples were subsequently annealed in air to induce the migration and aggregation of the metal ions. Results of structural, compositional and optical characterization are given, providing evidence for UV-VIS to NIR down-conversion, which could find potential applications to increase the efficiency of Si-based PV solar cells.

3:50 PM

(ICACC-S7-005-2018) Nanoscale Morphology Control in Halide Perovskite/Polymer Composites for Printed LEDs and Beyond (Invited)

Z. Yu*[1]

1. Florida State University, Industrial and Manufacturing Engineering, USA

In recent years, remarkable optoelectronic properties have been discovered in a group of materials called halide perovskites. Their potential to invigorate the current solar cell and light-emitting diode (LED) industries has been demonstrated by achieving very high device efficiencies in relatively short periods. In this talk, our recent work of developing perovskite/polymer composites towards the realization of fully printable LEDs will be presented. The perovskite/polymer composites possesses all the remarkable optoelectronic characteristics of pure perovskites. For instance, we have demonstrated their use for blue, green and red LEDs. In addition the device efficiencies exceeded those of pure perovskite LEDs. The perovskite/polymer composites have shown advantages in improving the processability and quality of the perovskite thin films; and enhancing the structural stability of the perovskites especially at high humid fabrication and service environments. By embedding the perovskite crystals inside a polymer matrix, the perovskites can be less toxic and more environmentally benign compared to pure perovskite. At the end of the talk, other potential applications of perovskite/polymer composites will be briefly discussed.

4:20 PM

(ICACC-S7-006-2018) Plasma-catalytic CO_2 conversion to methane using mesoporous Ni-Ce-Al_2O_3

T. Andreu*[1]; M. Biset[1]; J. Guilera[1]; J. R. Morante[1]

1. Catalonia Institute for Energy Research (IREC), Advanced Materials for Energy Area, Spain

The intermittence and fluctuations of the renewable energy sources constitute an important problem that needs solutions in terms of storage the exedent electrical power. A promising solution is to store this energy by converting to chemical energy. In this regard, the production of CH_4 from CO_2 and H_2 stands out over other fuels, as the produced methane can uses the current infrastructure of natural gas. Herein, we have focused on hybrid plasma-catalytic methanation, using a Dielectric Barrier Discharge (DBD) plasma reactor and Ni based catalyst. A modular coaxial DBD reactor is used for the plasma-catalysis methanation where the catalyst is placed between the two coaxial tubes, in the discharge region of the plasma (plasma-catalysis system). Different Ni-Ce based catalysts have been prepared by impregnation method on alumina support. As support, mesoporous alumina has been synthetized by evaporation-induced self-assembly (EISA) method. The results show an increase on the conversion when the reaction is done at low temperature (⟨250 °C). In addition, experiments under adiabatic conditions have been performed, with high selectivity and conversion and allowing the production of CH_4 without a pre-heating stage. By the use of plasma, methane is produced at lower temperature than conventional thermal-catalytic process, avoiding problems such as catalyst deactivation, secondary reactions and water absorption.

4:40 PM

(ICACC-S7-007-2018) Ultra-broadband perovskite-PbS-quantum-dot sensitized carbon nanotube photodetector

I. Ka*[1]

1. Ecole de technologie superieure, Genie electrique, Canada

Organic-inorganic perovskite has been proven to be a potential candidate for optoelectronics and photovoltaics, but their applications are limited in the visible region[1-7]. Here, combining single wall carbon nanotube, PbS quantum dots and perovskite, we synthesize hybrid that operates both in the visible and the near infrared. The photodetectors fabricated using the hybrid as the photoactive material show responsivities as high as 0.8 $A.W^{-1}$and 0.35 $A.W^{-1}$ at 480 nm and at 1400 nm, respectively, at a voltage as low as 1 V. Additionally because of their fast response time of 250 μs, the photodetectors exhibited gain-bandwidth product of 10^5 Hz. Our novel approach of synthesis of this hybrid material, which benefits from the high mobility of the SWCNT, opens new pathways for the development of low-cost and ultra-broad spectral optoelectronic devices.

5:00 PM

(ICACC-S7-008-2018) Au@Sulfide Yolk@Shell Nanocrystals with Diverse Shell Compositions Prepared by Anion Exchange Reaction

M. Fang*[1]

1. National Chiao Tung University, Material Science and Engineering, Taiwan

With the interior hollow space and homogenous reaction environment, yolk@shell nanocrystals in which an individual yolk core is surrounded by a porous hollow shell may find promising potentials in applications requiring facile charge transport and high surface area, for example, solar energy conversion, gas sensing, and electrochemical energy conversion and storage. Here we reported a delicate anion exchange approach for preparation of Au@sulfide yolk@shell nanocrystals with diverse sulfide compositions. By using Au@Cu7S4 nanocrystals as the sacrificial template, the anion exchange reaction led to the replacement of Cu+ with other metal ions including Cd2+, Zn2+, Co2+, and Ni2+, resulting in the formation of Au@CdS, Au@ZnS, Au@CoS and Au@NiS nanocrystals. The photocatalytic hydrogen production activities of the samples were further compared.

5:20 PM

(ICACC-S7-009-2018) Semiconductor Nanoheterostructures for Photoconversion Applications (Invited)

Y. Hsu*[1]

1. National Chiao Tung University, MSE Department, Taiwan

With the inherently high degree of complexity, nanoheterostructures composed of two or more materials joined in unique architectures may exhibit superior synergetic properties that are difficult or impossible to acquire from their individual constituents. For semiconductor nanoheterostructures, the relative band alignment of the constituents promotes effective charge separation to bring them desirable properties for photoconversion applications. Several representative works from our lab including Au-CdS, Au-ZnS core-shell nanocrystals, Au-decorated TiO2 nanowires, ZnO-An-SnO2 Z-scheme nanorods, and Au@Cu_7S_4-decorated TiO_2 nanowires will be introduced to demonstrate the promising potentials of semiconductor nanoheterostructures.

S8: 12th International Symposium on Advanced Processing and Manufacturing Technologies for Structural and Multifunctional Materials and Systems (APMT12)

Advanced Sintering Technologies -FLASH, SPS, etc.

Room: Coquina Salon A

Session Chairs: Zhengyi Fu, Wuhan University of Technology; Hidehiro Yoshida, National Institute for Materials Science (NIMS)

1:30 PM

(ICACC-S8-001-2018) Cold, Flash, Sparks, Nano - Where are Sintering Processes Headed? (Invited)

G. L. Messing*[1]; T. Frueh[1]; W. Rheinheimer[2]; E. R. Kupp[1]; M. J. Hoffmann[2]

1. The Pennsylvania State University, Materials Science and Engineering, USA
2. Karlsruhe Institute of Technology, Institute for Applied Materials -- Ceramic Materials and Technologies (IAM-KWT), Germany

Recent times have seen an explosion of innovative means to densify ceramics at significantly lower temperatures and much shorter times. These innovations have capitalized on either massive increases in driving force by using >100 MPa pressures, or exceptional changes in kinetic processes that enable faster diffusion over short-range distances. This presentation analyzes the spectrum of conditions reported using the sintering approaches listed in the title and summarizes these novel processes in terms of sintering driving force differences and mechanistic alterations in transport required for densification of ceramics powders. In the end, we project how these novel sintering processes will impact commercial sintering technology.

2:00 PM

(ICACC-S8-002-2018) Flash-sintering and related phenomena in oxide ceramics (Invited)

H. Yoshida*[1]; K. Morita[1]; B. Kim[1]; T. Yamamoto[2]

1. National Institute for Materials Science (NIMS), Japan
2. Nagoya University, Japan

Flash-sintering, where densification occurs almost immediately (typically <5 seconds) under high DC electric field, has attracted extensive attention as an innovative sintering technique since the first report by Professor Raj's group in 2010. The flash-sintering is characterized not only by the immediate densification but also by a nonlinear increase in electric conductivity under a threshold condition of temperature and applied field. The flash-sintering has been demonstrated in various ceramics, and nearly full density has been achieved at relatively low furnace temperature for very short time. We have recently demonstrated that dielectric oxide ceramics such as Y_2O_3, $MgAl_2O_4$ and $BaTiO_3$ can be consolidated by flash-sintering technique. For example, high-purity, undoped Y_2O_3 can be fully densified by flash-sintering; full densification is achieved at 1133°C under a field of 500 V/cm. Microstructural observations and analyses by high-resolution transmission electron microscopy indicated that the DC electric current flows produced strongly-reducing conditions in the oxide compacts. In the present paper, our results on the flash-sintering behavior of oxide ceramics are briefly summarized, and recent attempts for elucidation of the underlying mechanisms responsible for the flash event are introduced.

2:30 PM

(ICACC-S8-003-2018) Ultra-fast densification of boron carbide by flash spark plasma sintering

Z. Fan*[1]; W. Wang[1]; Z. Fu[1]

1. Wuhan University of Technology, China

We present a simple method of sintering ceramic powders in 1 min at low temperature and low pressure. Nearly fully dense(99.2%) B_4C with limited grain growth was prepared at 1931 °C with an applied pressure of 15.3MPa. Comparison of our method to a simple SHS/QP process reproducing the same aggregate thermodynamic conditions shows that our process yields significantly higher and faster densification. We attribute this outcome to the particular spatial and temporal variations of Joule heating within the material. A short sintering time and plastic deformation lead to limited grain coarsening. The novel method (FSPS) combines flash sintering and electric field assisted sintering. Over previous reports of flash sintering, our method requires both less energy and less processing time for an improved outcome. Neither preheating of the sample, which is energetically costly, nor the fabrication of a pre-sintered sample, which is temporally costly, is required to initiate the flash sintering. In addition, the Joule heating inspired by the pulsed current is highly localized to the particle interfaces, yielding efficient coalescence with limited energy input. In future work, we will apply this process to prepare other conductive and semiconducting ceramics.

3:10 PM

(ICACC-S8-004-2018) Quantifying Densification Variability in Ionic Conducting Ceramics via Spark Plasma Sintering

C. S. Smith*[1]; N. J. Madden[1]; J. A. Krogstad[1]

1. University of Illinois at Urbana-Champaign, Materials Science and Engineering, USA

Spark plasma sintering (SPS) has gained notoriety for the ability to rapidly and thoroughly densify metal and ceramic structures. However, the precise mechanisms by which densification occurs is not well understood across a wide range of material classes. In particular, ionic conductors such as yttria stabilized zirconia (YSZ) have exhibited highly variable behavior when sintered in the reducing atmosphere of an SPS chamber, often requiring a post-sintering anneal that can negate some of the beneficial effects of using SPS. For this study, conductive capping layers have been applied to YSZ samples over a range of sintering conditions in order to illuminate some of the factors contributing to the variation in densification. These layered samples have been prepared using the same conditions as bare YSZ samples, with sufficient numbers of samples being prepared to collect statistics on the variability within each group. Using Vickers microhardness and microscopy, the mechanical properties and associated microstructures have been compared for both bare and layered YSZ samples. Findings show that the layered samples have a decreased likelihood of reduction but also reduced hardness relative to the bare YSZ samples. These results shed light on the mechanisms involved in densification of ionic conductors and have implications that may lead to more informed design of SPS processing schemes.

3:30 PM

(ICACC-S8-005-2018) Influence of grain size on the entropic transformation in high entropy oxides

A. D. Dupuy*[1]; J. M. Schoenung[1]

1. University of California, Irvine, Chemical Engineering and Materials Science, USA

High entropy stability has emerged as a promising (and controversial) strategy for designing new materials. The recent introduction of high entropy stability to oxides has provided this class of materials with the opportunity to reap the potential benefits from an increase in composition space. High entropy oxide (HEO) materials contain at least five oxide components, which, after appropriate processing, form a single phase that is stabilized through configurational entropy. A unique characteristic of HEO materials is their reversible entropy-driven phase transformation between the single phase and multiphase states. This feature presents an opportunity to produce oxide materials with highly controlled phase states. Since research into HEO materials is still in its infancy, the role of processing and microstructure on the entropic phase transition is largely unknown. In this work, we explore the impact of grain size on the entropic phase transition in (CoCuMgNiZn)O ceramics. Sourced nanopowders are mixed using planetary ball milling. These powders are simultaneously reacted and densified using free sintering and spark plasma sintering (SPS). By leveraging these processing routes, we show that it is possible to produce fully dense HEO ceramics with grain sizes spanning several orders of magnitude. Characterization of these ceramics reveals the length scale dependence of the entropic phase transition.

3:50 PM

(ICACC-S8-006-2018) Flash sintering entropy-stabilized oxide

J. Liu*[1]; D. Liu[2]; G. Liu[1]; Y. Wang[3]; L. An[4]

1. Southwest Jiaotong University, School of Mechanics and Engineering, China
2. Southwest Jiaotong University, School of Materials Science and Engineering, China
3. Northwestern Polytechical University, School of Materials Science and Engineering, China
4. University of Central Florida, Department of Materials Science and Engineering, USA

In this paper, the entropy-stabilized oxide with rocksalt structure was synthesized by flash sintering the equimolar mixture of MgO, NiO, Co_3O_4, CuO and ZnO at 500 °C with power supply for 1 min. The measured temperature on the surface of the specimen is lower than 875 °C. The XRD patterns indicate the transition from multiple-phase to single phase was finished in several tens of second. Compared with previous study, the electric field accelerated the homogeneous cation mixing at lower temperature. In other words, the diffusion of cation during flash sintering is obvious besides the movement of oxygen vacancies.

4:10 PM

(ICACC-S8-007-2018) Effect of oxygen partial pressure on temperature for onset of flash sintering 3YSZ

D. Liu*[1]

1. Southwest Jiaotong University, Key Laboratory of Advanced Technologies of Materials, China

We report that the oxygen partial pressure affects the onset temperature of flash sintering of 3 mol% tetragonal-stabilized zirconia (3YSZ) in an oxygen-enriched environment. Flash sintering experiments were performed with oxygen partial pressures between 0.4 atm and 1 atm. The results indicate that the onset temperature increases with an increase in oxygen partial pressure. Based on the plots of power dissipation as a function of temperature, the oxygen partial pressure might affect the onset temperature by changing the conductivity in the pre-flash stage. Combined with the analysis of the law of mass action, we found that electron conduction may play an important role in the pre-flash stage of flash sintering.

S11: Advanced Materials and Innovative Processing Ideas for the Production Root Technologies

Industrial Root Technology Based on KITECH and GIGAKU Concept

Room: Ponce de Leon
Session Chairs: Tadachika Nakayama, Nagaoka Univ of Tech; Sungwook Mhin, Korea Institute of Industrial Technology

1:30 PM

(ICACC-S11-001-2018) In-situ and on-demand synthesis of carbon-based boundary films from lubricating oils by catalytically active composite coatings (Invited)

A. Erdemir*[1]; G. Ramirez[1]; O. Eryilmaz[1]

1. Argonne National Lab, USA

Increasingly demanding operating conditions of many moving parts in engines are rendering most traditional materials and coatings ineffective and hence there is an urgent need for the development of more robust materials and coatings for the next generation transportation vehicles. At present, DLC coatings are used extensively to meet the specific application needs of current engines and other moving mechanical systems. We have been focusing on the next generation novel tribological coatings that can provide ultra-low wear and –friction even with the use of base lubricating oils. Specifically, these coatings are primarily made of catalytically active hard and soft phases which enable ultra-high hardness, toughness in addition to excellent catalytic responsiveness to the hydrocarbon molecules of lubricating oils. Specifically, when tested in neat or marginally additized base oils, these catalytic coatings fragment long-chain hydrocarbon molecules of lubricating oils to produce a carbon-rich boundary film whose structural chemistry is similar to those of DLC. Overall, comprehensive friction, wear, and scuffing studies have confirmed that these nanocomposite coatings could serve as the next generation materials for a wide range of demanding automotive applications. They can reduce parasitic friction losses as well as minimize wear and scuffing failures.

1:50 PM

(ICACC-S11-002-2018) Motion control of micromachine by nano 3D printer and electric field control (Invited)

T. Nakayama*[1]; K. Moriya[1]; M. G. Herrera Salazar[2]; H. Suematsu[1]; T. Suzuki[1]; K. Niihara[1]

1. Nagaoka Univ of Tech, Japan
2. University of Aarhus, Finland

In recent years, researches have been made to fabricate the micro-structures using a 3D printer and to add functions to them. Among these researches, development of micro-pump to control micro-fluid has attracted attention. In addition, light, magnetic field, and electric field are used to drive the micro-pump. However, any micro-pumps can only control to turn on and turn off for micro-fluid. Therefore, in this study, we aimed to fabricate a mechanism to mix micro-fluid by applied a DC electric field to a micro-pump fabricated by a 3D printer. As the experimental method, a micro-pump is fabricated by using a 3D printer, and a ZrO_2 balls are placed therein. Al electrodes are installed at both ends of a micro-pump, and a DC electric field is applied. Observation and movie shooting are done by using the digital microscope. As the experimental results, it was confirmed that the ZrO_2 balls were moving through the micro-pump by applied a DC electric field. However, the ZrO_2 balls did not move smoothly through the micro-pump and confirmed temporary the stagnation behavior. As a future task, we need to consider about the structure of a micro-pump in which the ZrO_2 balls move more smoothly. Also consider the structure of an Al electrode. If this study succeeds, it is possible to easily mix the chemical solution of micro level.

2:10 PM

(ICACC-S11-003-2018) The properties of ternary Mo-Cu-X-N, (X= Ni, Si, V) coating synthesized by magnetron sputtering process with single alloying targets

H. Lee*[1]; H. Yoon[1]; G. Bang[1]; K. Moon[1]

1. Korea Institute of Industrial Technology, Republic of Korea

In this study, we tried to deposit Mo-Cu-X-N(X= Ni, Si, V) thin coatings showing high hardness and low friction at low and high temperatures to reduce energy consumption and wear problems in engine parts and tools. In general, this kind of nano-composite coating is made by various processes using multiple targets such as Mo, Cu and X(X= Ni, Si, V). However, it is difficult to control the exact composition, homogeneous deposition of large scale specimens during the deposition with multiple targets. We wanted to create the single Mo-Cu-X(X= Ni, Si, V) alloying targets with the composition showing the best friction coefficient and surface hardness. Single alloying targets with the composition showing the best properties were prepared by powder metallurgy methods, such as mechanical alloying and spark plasma sintering. Mo-Cu-X(X= Ni, Si, V) targets were prepared subsequently. The nano-composite Mo-Cu-X(X= Ni, Si, V) coatings prepared using the alloying targets will be eventually compared with the films from the multiple targets.

2:30 PM

(ICACC-S11-004-2018) Modulating the Piezoresistivity and Thickness of Silicone Rubber Coating Films for Pressing-Sensor Applications (Invited)

S. T. Nguyen*[1]; V. Tran-Khac[1]; H. Suematsu[1]; T. Suzuki[1]; K. Niihara[1]; T. Nakayama[1]

1. Nagaoka University of Technology, Japan

In this research, a homogeneous mixture was prepared by stirring the room temperature vulcanization silicone rubber with silica balloon and highly conductive carbon black nanoparticles. A polyimide printed flexible circuit board was then dipped into and withdrew from the mixture to obtain a 60 μm-thick hybrid film deposited on the board. The electromechanical tests revealed that the thickness and piezoresistivity of the hybrid film can be controlled by modulating the mixture composition and dipping process. The optimized hybrid film exhibits a sensitivity comparable to that of commercial pressing-sensor films, but possess a much smaller thickness. Therefore, the fabricating process is considered very promising for micrometer-thick pressing-sensors.

2:50 PM

(ICACC-S11-005-2018) Electrical characteristics of PIN-PMN-PT ceramics by impurities effect for high temperature application (Invited)

T. Moro*[1]; T. Nakayama[1]; J. Kim[2]; Y. Kim[2]; J. Kim[2]; S. Yamanaka[2]; T. Katou[2]; I. Murayama[2]; M. Takeda[1]; N. Yamada[1]; H. Suematsu[1]; K. Niihara[1]

1. Nagaoka Univ of Tech, Japan
2. Daihatsu Motor Co., Ltd., Japan

PMN-PT ceramics has high piezoelectric constant, dielectric constant and spontaneous polarization amount and is widely applied by taking advantage of excellent electric characteristics. However, it is difficult to use in high temperature region almost 200° C. In this research, for the purpose of utilization at high temperature, by dissolving PIN in solid solution in PMN - PT, the Curie temperature was improved and the influence on the electrical characteristics by adding impurities was investigated. The composition ratio was near 26 PIN - 40 PMN - 34 PT, which is reported to have high ferroelectricity in the previous study. Furthermore, we add trace amounts of MnO_2 and La_2O_3, and perform electrical measurements such as dielectric constant, pyroelectric current, hysteresis, etc., and discuss the influence.

3:30 PM

(ICACC-S11-006-2018) Highly Insulation Property of Aerosol Deposited Al_2O_3 Thick Films (Invited)

R. Aoyagi*[1]; H. Tsuda[1]; J. Akedo[1]

1. National Institute of Advanced Industrial Science and Technology, Japan

New semiconductors such as SiC and GaN will realize high power switching devices and high brightness LEDs. In these devices. High-density mounted these devices will release higher heat compared to conventional semiconductor devices therefore heat dissipation in the substrate becomes a serious problem. The ceramic substrate with high thermal conductivity such as AlN and metal substrate covered with insulating resin have been studied as high heat dissipation circuit boards for these devices. However, AlN ceramic substrates have problems in their poor workability and high cost. And the metal-based substrates have problems in peeling between metal and insulating layer due to a difference in thermal expansion of the joint portion. In previous study, we have confirmed that the Al_2O_3 thick film deposited by aerosol deposition method (ADM) exhibits high adhesion strength to the substrate and high insulation properties. In this presentation, we will discuss detailed insulating properties of Al_2O_3 thick films by ADM and potentials as heat dissipation properties. Al_2O_3 thick films (>1μm) was successfully deposited on metal substrates by ADM. The samples were composed with nano-sized particles which is much smaller than the submicron sized starting particles. It was found that the samples had high breakdown voltage more than 1 kVdc at thickness of 10 μm and breakdown field depended on Vickers hardness of the films.

3:50 PM

(ICACC-S11-007-2018) Novel Glass-ceramics from Glass Powders and Reactive Silicone Binders (Invited)

E. Bernardo*[1]; H. Elsayed[1]

1. University of Padova, Dipartimento di Ingegneria Industriale, Italy

The processing of sintered glass-ceramics, i.e. material from the sintering of fine glass particles, with concurrent crystallization, is often conditioned by the debinding step. Typical carbonaceous polymeric binders, namely subjected to complete decomposition upon firing, may determine some defects in the final glass-ceramic directly, by causing some gas evolution continuing even at an advanced state of densification, or indirectly, by offering poor adhesion between particles (so that 'green' compact may be easily damaged). The present investigation is aimed at exploring a novel concept, based on the adoption of silicone polymers, providing an abundant ceramic residue after firing. Some glasses (belonging to the $CaO-MgO-Al_2O_3-SiO_2$ and $CaO-B_2O_3-SiO_2$ systems), normally yielding useful glass-ceramics by heat treatment, were reproduced in form of 'silica-defective' variants, featuring a SiO_2 content, in the overall formulation, reduced up to 15 wt%. The overall silica content was recovered by mixing powders of the new glasses with silicone binders: upon firing in air, the interaction between glass powders and polymer-derived silica led to glass-ceramics with the same assemblage than those formed by the reference glasses. The new approach has been successfully applied to the manufacturing of glass-ceramic joints for SOFCs as well as of glass-ceramic scaffolds for tissue engineering.

4:10 PM

(ICACC-S11-020-2018) Solubility limit of B1-GaN to B1-(Cr,Ga)N thin films (Invited)

Y. Mizuno*[1]; T. Nakayama[1]; H. Suematsu[1]; T. Suzuki[1]

1. Nagaoka University of Technology, Extreme Energy-Density Research Institute, Japan

In order to develop a new hard coating material for cutting tools, we designed and synthesized (Cr,Ga)N thin films by adding B1-GaN into B1-CrN. In the case of (Cr,Al)N which is one of the major hard coating materials, its hardness increases with increasing AlN content up to the solubility limit. However, when AlN content exceeds the solubility limit, (Cr,Al)N thin films show phase transition from B1 to B4 (würtzite) type and its hardness decrease drastically. Thus it is important to reveal the solubility limit of B1-GaN in B1-(Cr,Ga)N. We had already reported the synthesis of B1-(Cr,Ga)N thin films with GaN content of 10 mol%. In this work, in order to explore the solubility limit, Cr-Ga-N thin films with various GaN contents were prepared on MgO(100) substrates by pulsed laser deposition (PLD). Compositional analysis by Rutherford backscattering spectroscopy revealed that the ratio of the amount of GaN to the amount of CrN (C_{GaN} = Ga / (Cr + Ga)) in all Cr-Ga-N thin films was in the range of 0 to 0.88. X-ray diffraction and microstructural observation by a field emission transmission electron microscope (FE-TEM) indicated that Cr-Ga-N thin films with $C_{GaN} \leq 0.28$ were composed of only B1 phase, meaning B1-GaN dissolve in B1-CrN. It seems that solubility limit of B1-GaN into B1-(Cr,Ga)N is more than 28 mol%.

4:30 PM

(ICACC-S11-009-2018) Cobalt Nickel Sulfide Nanoparticles Anchored on Porous Graphene Aerogel as Efficient Electrocatalysts for Oxygen Evolution Reaction

H. Han*[1]; Y. Hong[1]; S. Mhin[1]

1. Korea Institute of Industrial Technology, Republic of Korea

Co-Ni-S (CNS) system integrated in the 3D porous nitrogen doped graphene aerogel (NGA) template (CNS-NGA) is demonstrated as a new efficient OER and overall waters splitting catalyst. CNS-NGA can be prepared via facile one-step hydrothermal process. CNS-NGA can function efficiently as both anode and cathode of a single alkaline electrolyzer. Overall water splitting performance of the CNS-NGA outperforms an electrolyzer composed of Pt/C(cathode)-IrO_2(anode). Results of the experimental and computational calculation demonstrate that the synergetic effects between Co and Ni metal atoms as well as between Co-Ni-S and 3D porous NGA supports mainly contribute to state of the art catalytic performance of the CNS-NGA nanocomposites for electrochemical water splitting.

4:50 PM

(ICACC-S11-010-2018) Developing low temperature Process Temperature Control Rings for accurate measurement of temperature in range of 560-900°C (Invited)

A. Saberi*[1]; H. Jarnicki[1]; J. Amyot[2]; J. Risse[1]

1. Ferro GmbH, R&D, Germany
2. Ferro S.A.R.L, France

Temperature and time have important roles in adjusting final microstructure of most of materials in order to achieve desired properties. Besides, re-producibility of properties and quality assurance in production line need an accurate control of total energy absorbed by product. 7 different PTCR references can measure measuring temperature over temperature range of 560-1750 °C. The PTCRs can be easily placed everywhere in the furnace especially next to product to measure the entire heat energy transmitted to product. A well-defined microstructure together with selecting proper composition are key parameters for producing PTCRs. A linear shrinkage at temperatures below 900°C is only achievable by means of glassy phase(s). Viscosity and crystallization behavior of such phase(s) have decisive roles in sintering behavior of composite. Two different low-Tg glasses were designed as main ingredients for developing the low temperature PTCR references. Thermal behavior of these glasses were characterized. Then interaction between different oxides used as filler together with these glasses on sintering behavior of glass/ceramic composites were studied. Results showed besides thermal characteristics of glasses, congruency of low-Tg glasses and filler type as well as particle size distribution of filler have important roles in achieving PTCRs with accuracy of ±2°C.

5:10 PM

(ICACC-S11-011-2018) Themophysical Properties of Selected Nitrates and Fluorides Measured by Means of Thermal Analysis and Hyphenated Techniques (Invited)

E. Post*[1]

1. NETZSCH Geraetebau GmbH, Germany

Nitrate or fluoride mixtures of inorganic salts are interesting candidates for heat storage, heat- exchanging materials, solar salts or even use in molten salt nuclear reactors. The determination of the important physical data, such as melting enthalpy, specific heat, thermal expansion and thermal conductivity by thermal analysis methods can be a challenging task. Even these well-established methods are fast, reliable and accurate for most cases. The problems are often due to choosing the right container material for these substances. First, the sample must not react with the crucible material. To fulfill this requirement seems not too hard at this relatively low temperature range. More critical is the creeping ability of the molten salts into capillaries, etc., due to wettability. These effects can produce bad thermal contacts, e.g. for c_p measurements or even the sample creeps out of the container arrangement (dilatometer, LFA measurement). Low purity of the salts can also affect application problems. In this contribution, some container/sample material combinations will be discussed. Finally, specific heat, thermal expansion and thermal conductivity data of selected nitrates will be presented. Fluorides were especially investigated by means of TGA coupled to mass spectrometry.

S12: Advanced MAX/mxene Phases and UHTC Materials for Extreme and High Temperature Environment

Novel Applications and Processing Methods for Synthesizing MAX Phases I

Room: Tomoka B

Session Chairs: Michel Barsoum, Drexel University; Thierry Ouisse, Grenoble INP

1:30 PM

(ICACC-S12-008-2018) Towards single phase $Zr_{n+1}AlC_n$ MAX phase ceramics for future fission environments (Invited)

E. Zapata-Solvas*[1]; W. E. Lee[1]

1. Imperial College London, Centre for Nuclear Engineering. Dpt. of Materials, United Kingdom

After Fukushima's nuclear disaster there has been a growing interest in introducing new safety concepts for future fission reactors. One approach is to develop Accident Tolerant Fuels (ATF) that can withstand the harsh environment within a fission reactor for at least 10 hours in a Loss-of-Coolant-Accident (LOCA). MAX phases are potential candidates for use in ATF as cladding. The system that has been targeted is $Zr_{n+1}AlC_n$. Zr offers compatibility with the zircaloy cladding, Al offers resistance to corrosion and oxidation, while C limits nuclear transmutation. This work examines stabilization of $Zr_{n+1}AlC_n$ MAX phases by partial substitutions in the quaternary systems $(Zr,M_2)_{n+1}AlC_n$ or $Zr_{n+1}(Al, A_2)C_n$ as well as the quinary system $(Zr,M_2)_{n+1}(Al,A_2)C_n$ with the goal of obtaining sigle phase Zr-based MAX phases. Effects of impurities and O on Zr-based MAX phases synthesis will be discussed.

1:50 PM

(ICACC-S12-002-2018) TEM study of Ti_3SiC_2 thin films synthesized on 4H-SiC

C. Bail[1]; C. Furgeaud[1]; G. Amiard[1]; T. Cabioch[1]; M. Beaufort[1]; J. Nicolaï[1]; B. Levraut*[1]

1. Institut PPRIME, France

In this work, the formation mechanisms of MAX phases Ti_3SiC_2 onto 4H-SiC during thermal annealing at high temperature (700-1000°C) of $TiAl_2$ layers were investigated. To optimize the growth process, annealing parameters (temperature, dwelling time) were varied systematically and the microstructural evolution of the film and its orientation relationship with the substrate were investigated by XRD and TEM/STEM while the evolution of the chemical composition was studied by EDXS. The thin film structure and its composition are both, time and temperature dependent. At low temperatures (700-800°C) the Ti_2AlC MAX phase is obtained, the crystallinity quality of the film increasing with the annealing time. At higher temperature (above 900°C) and for short annealing times, the formation of both $Ti_3(Si,Al)C_2$ and Ti_2AlC MAX phases was observed. Nevertheless, when increasing the annealing time the 312 MAX phase becomes predominant. Moreover, a large quantity of the initial $TiAl_2$ remains present even for long annealing times. This observation seems to indicate that the MAX phase acts as a diffusion barrier.

2:10 PM

(ICACC-S12-003-2018) Lightweight MAX phases - based materials heat-resistant in oxidizing and hydrogen atmosphere

T. Prikhna*[1]; O. Ostash[2]; V. Sverdun[1]; T. Cabioch[3]; M. Karpets[1]; L. Jaworska[4]; A. Ivasyshin[2]; S. Dub[1]; A. Kuprin[5]

1. Institute for Superhard Materials of the National Academy of Sciences of Ukraine, Ukraine
2. Karpenko Physical-Mechanical Institute of the National Academy of Sciences of Ukraine, Ukraine
3. Universite de Poitiers, CNRS/Laboratoire PHYMAT, UMR 6630 CNRS Universite de Poitiers, France
4. The Institute of Advanced Manufacturing Technology, Poland
5. National Science Center Kharkov Institute of Physics and Technology, Ukraine

The dense MAX-phases-based materials of Ti,Nb–Al–C systems of 211 and 312 structural types are stable in hydrogen and oxidizing environments at 600 °C, are about twice lighter and more stable in air than Cr-containing Crofer steels widely used as interconnects for hydrogen fuel cells. The most resistant in air at 600 °C (for 1000 h) among the studied materials turned out to be Ti_2AlC-based and somewhat less stable $(Ti, Nb)_3AlC_2$ – based. The Auger study confirmed the presence of some oxygen in the structures of Ti_2AlC and $(Ti,Nb)_3AlC_2$ what can be the explanation of their high stability against oxidation. The oxide films on the surfaces of the materials with Nb were thinner. Ti_3AlC_2-based materials demonstrated 4.6-5.8 GPa microhardness at 5 N load, 500-570 MPa bending and 700-1300 MPa compression strengths, their fracture toughness reached 10.2 ± 0.4 MPa$\bullet\sqrt{}$m, and electrical conductivity $2.7\bullet10^6$ Sm/m at 20 °C. The bending strength of Ti_3AlC_2-based material in air at 20 °C was 535 MPa, after been keeping at 600 °C in air and hydrogen it decreased to 490 and 500 MPa, respectively. For $(Ti,Nb)_3AlC_2$ – based materials the bending strength at 20 °C in air was 480 MPa, but increased for 10% after heating at 600 °C in air and in hydrogen.

2:30 PM

(ICACC-S12-004-2018) Synthesis of Phase-Pure $(Zr,Ti)_2(Al,Sn)C$ MAX Phase Solid Solutions

K. Lambrinou*[1]; B. Tunca Altintas[1]; T. Lapauw[1]; R. Delville[1]; J. Hadermann[2]; J. Vleugels[3]

1. SCK-CEN, NMS, Belgium
2. University of Antwerp, EMAT, Belgium
3. KU Leuven, Dept. Materials Engineering, Belgium

Good resistance to high-temperature steam oxidation is a prerequisite for candidate coating materials on accident-tolerant fuel clads designed for Gen-II/III light water reactors. In order to improve the oxidation resistance of Zr-Al-C MAX phases, (Zr,Ti)-Al-C solid solutions were made, but the production of phase-pure ceramics in this system proved challenging due to the concurrent production of large amounts of 'parasitic' phases, such as (Ti,Zr)C carbides and intermetallics. Since assessing the intrinsic oxidation behaviour of novel materials requires testing of phase-pure material grades, quasi-phase-pure (>99%) (Zr,Ti)-(Al,Sn)-C MAX phase solid solutions were produced in this work. No (Ti,Zr)C carbides or intermetallics were detected by X-ray diffraction (XRD) of $(Zr_{1-x},Ti_x)_2(Al_{0.5},Sn_{0.5})C$ ceramics with $0.33 \leq x \leq 0.90$, while other compositions (x=0, 0.1, 1) contained limited amounts (5-15%) of 'parasitic' phases. This showed that adding Sn enables the production of phase-pure 211-type MAX phases in the Zr-Ti-Al-C system. The effect of processing parameters (e.g., applied pressure and sintering atmosphere) on the phase purity and microstructure of ceramics produced from identical Zr-Ti-Al-Sn-C powder mixtures was also studied. Thermal expansion coefficient determination of $(Zr_{1-x},Ti_x)_2(Al_{0.5},Sn_{0.5})C$ MAX phase ceramics with x=0-1 was performed by Rietveld refinement of high-temperature synchrotron XRD data.

2:50 PM

(ICACC-S12-005-2018) Preparation of TiC/Ti_2AlC constituent gradient coating on carbon fiber and investigation on the oxidation resistance properties

M. Li*[1]; J. Wang[1]; L. He[1]; F. Huang[1]; S. Du[1]; Q. Huang[1]

1. Ningbo Institute of Industrial Technology, Chinese Academy of Sciences, Engineering Laboratory of Specialty Fibers and Nuclear Energy Materials, China

A thickness controllable TiC/Ti_2AlC coating was fabricated on carbon fiber by an in-situ reaction in molten salt bath. The constituent of the coating varies along with the radial direction, thus the coating shows a multi-layer structure, in which the inner layer is TiC and the outer layer is Ti_2AlC. The oxidation resistance properties of the as-prepared TiC/Ti_2AlC coated carbon fiber in static air and water vapor at 600 °C were investigated. The results show that the as-prepared TiC/Ti_2AlC coating could provide good protection for carbon fiber in both static air and water vapor. Since both TiC and Ti_2AlC has good irradiation resistance, the present work provides a potential way to development irradiation resistant interphase of C_f/SiC and SiC_f/SiC composites for nuclear applications.

Theoretical Perspective for Designing MAX Phases and Novel Applications and Processing Methods for Synthesizing MAX Phases - II

Room: Tomoka B

Session Chairs: Per Eklund, Linkoping University; Miladin Radovic, Texas A&M University

3:30 PM

(ICACC-S12-007-2018) Computational approach to structural, mechanical, and thermodynamic properties of $Ti_3(Si_xAl_{1-x})C_2$

W. Son*[1]; A. Talapatra[1]; T. Duong[1]; H. Gao[2]; M. Radovic[1]; R. Arroyave[1]

1. Texas A&M University, Materials Science and Engineering, USA
2. Texas A&M University, Mechanical Engineering, USA

In this work, we used the first-principle calculations to study structural, mechanical, and thermodynamic properties of $Ti_3(Si_xAl_{1-x})C_2$. The first-principle calculations were based on the Density Functional Theory (DFT), which is implemented in the Vienna Ab initio Simulation Package (VASP). We are looking for two pure MAX phases, Ti_3AlC_2 and Ti_3SiC_2, and their solid solution, $Ti_3(Si_{0.5}Al_{0.5})C_2$, and the solid solution is modeled using special quasirandom structures (SQS), which is generated by the Alloy Theoretic Automated Toolkit (ATAT). We have calculated lattice parameters, elastic modulus, coefficient of thermal expansion (CTE), and heat capacity, and they agree well with experimental results. In addition, we studied the deformation behavior in terms of cleavage energy and stacking fault energy. Cleavage energy is calculated by cleaving M and A layers, since M-A bond strength is known as weaker than M-X bond strength. Stacking fault energy is calculated by shearing the structure, then relax the structure to vanish all stress components, which do not correspond to the shear deformation. For the stacking fault energy calculation, we used external optimizer GADGET to carry out full relaxation while fixing the shear angle, since VASP does not allow relaxation under arbitrary constraints.

3:50 PM

(ICACC-S12-001-2018) Relationship between microstructure and oxidation resistance of the Ti_2AlC MAX phase produced by powder metallurgy route

B. Levraut*[1]; S. Dubois[1]; V. Gauthier[1]; P. Chartier[1]

1. Institut PPRIME, DPMM, France

During the last decades, new materials have been designed in order to cope with new challenges in high temperature structural materials' domain. In this context, MAX phases deserve to be considered. These ternary compounds are characterized by a hexagonal structure composed of ceramic octahedron (M_6X) interleaved with A metallic layers; M stands for an early transition metal, A is a IIIA to IVA element of the periodic table and X stands for carbon and/or nitrogen. Oxidation studies of MAX phases that contain aluminum as A element have shown relatively good results. Indeed, oxidation of these materials leads to a passivating, dense and resistant a-alumina layer at high temperature. This study focuses on the oxidation behavior of Ti_2AlC MAX phase due to its high aluminum content, its low density and its crack healing ability. Two very different Ti_2AlC microstructures have been produced by HIP and SPS powder metallurgy processes. Impurities have been identified using Energy Dispersive X-ray Spectroscopy analyses. Grain sizes and impurity contents have been carefully measured by Optical and Scanning Electron Microscopies coupled with image analysis. Oxidation behavior has been investigated at 1000°C under synthetic air to highlight the relationship between microstructure and oxidation resistance.

4:10 PM

(ICACC-S12-009-2018) High temperature water vapor oxidation of typical MAX phases for ATFs

Y. Lei*[1]; J. Zhang[1]; X. Wang[1]; J. Wang[1]

1. Institute of Metal Research, Chinese Academy of Sciences, High-performance Ceramics Division, China

Development of accident tolerant fuel (ATF) for light water reactors (LWRs) that possesses enhanced oxidation resistance under extreme conditions has gained attentions after the accident at Fukushima in 2011. One of ATF strategies is applying a protective coating on the zircaloy cladding to delay the onset of severe LWR core degradation. MAX phases are believed to be attractive coating candidates, for they combine the merits of both metal and ceramic, such as high modulus, high thermal conductivities, excellent high temperature corrosion resistance and easy machinability. In this work, typical MAX phases, including Ti_3SiC_2, Ti_2AlC and Ti_3AlC_2, are exposed to controlled-steam environcment at 900-1200 °C simulated as Loss of Coolant Accident (LOCA) scenario in LWRs. For pure Ti_3SiC_2 phase, the oxide scale is almost composed of an outer layer of dense pure rutile and an inner layer of mixture of TiO_2 and SiO_2, while at 1200 °C, cracks are formed in the outer layer. For the Al-containing MAX phase, the oxidation results in a thin continuous protective Al_2O_3 layer at all the temperature with comparable oxidation rate to that in air, exhibiting good oxidation resistance in water vapor environment.

4:30 PM

(ICACC-S12-010-2018) Densification and Phase Evolution of SHS Derived MAX Phase Ti_3SiC_2 Active Precursor Powders during Hot Pressing Processes

L. Chlubny*[1]; J. Lis[1]; P. Borowiak[1]; K. Chabior[1]; K. Kozak[1]

1. AGH-University of Science and Technology, Poland

Ti_3SiC_2 is one of the most popular compounds which belong to the interesting group of ternary nanolaminate materials called MAX phases. These compounds are characterised by heterodesmic chemical bonding and thanks to this fact they are characterized by the unique set of properties situating them between metals and ceramics. Self-propagating High-temperature Synthesis (SHS) is one of the best methods of synthesizing fine active precursors powders of MAX phases. This method utilizes exothermal effect of the chemical reaction in adiabatic conditions and allows obtaining many compounds such as nitrides, carbides, composites, intermetallics, etc. at low energy consumption and with high efficiency and at relatively short time. In this paper authors would like to present results of investigation on densification and phase evolution phenomenon during hot pressing of various Ti_3SiC_2 active precursor powders manufactured by SHS method. Powders were synthesized by SHS with local ignition system from elemental metallic powders with stoichiometric proportions and with excess amount of silicon. The selected powders were hot-pressed at temperatures range from 1350 to 1500°C. The phase evolution and apparent densities of obtained samples were examined in order to establish the optimal conditions for manufacturing of near single phase Ti_3SiC_2 material.

4:50 PM

(ICACC-S12-011-2018) A Review of Different Types of MAX-Metal Composite Systems for Multifunctional Applications

M. Dey*[1]; M. Fuka[1]; S. Gupta[1]

1. University of North Dakota, Mechanical Engineering, USA

It is well known that $M_{n+1}AX_n$ (MAX) phases (over 60+ phases) are thermodynamically stable nanolaminates. These phases display fascinating properties like damage tolerance, thermal shock resistance, machinability, and hardness between 2–8 GPa. Recently, we showed that it is possible to manaufacture novel composites where MAX Phases act as reinforcements in metal matrix (MRMs). In this presentation, we will present a comprehensive review of some of the recent studies on designing MAX phase reinforced composites.

S13: Advanced Ceramics and Composites for Nuclear Fission and Fusion Energy

ATF and Coating Technologies

Room: Coquina Salon H
Session Chairs: Kurt Terrani, Oak Ridge National Lab; Monica Ferraris, Politecnico di Torino

1:30 PM

(ICACC-S13-001-2018) Mechanistic Mesoscale Simulation of UO_2 Sintering and Densification (Invited)

M. R. Tonks*[1]; I. Greenquist[2]; Y. Zhang[3]

1. University of Florida, Materials Science and Engineering, USA
2. Pennsylvania State University, Mechanical and Nuclear Engineering, USA
3. Idaho National Lab, USA

Light water reactor fuel pellets are fabricated by sintering UO_2 powder until it reaches greater than 97% density. During the early stages of reactor operation, the fuel pellets continue to densify, resulting in an increase in the gap width between the fuel and cladding and a resultant increase in fuel centerline temperature. For this reason, it is critical that we understand the mechanisms involved in sintering and how these mechanisms change in reactor. For this reason we are developing a mesoscale model of UO_2 sintering using the phase field method coupled with heat conduction, mechanics, and radiation damage. The model is being developed using the MARMOT mesoscale nuclear materials code. In this presentation, we summarize the sintering model, demonstrate its capabilities, and how it is being used to improve our understanding of nuclear fuel densification.

2:00 PM

(ICACC-S13-002-2018) LCVD Approaches for Novel Accident Tolerant Fuel Concepts

S. Harrison*[1]; J. Pegna[1]; J. L. Schneiter[1]; R. K. Goduguchinta[1]; E. G. Vaaler[1]; K. L. Williams[1]

1. Free Form Fibers, USA

Free Form Fibers (FFF) continues to make inroads on innovative nuclear fuel concepts for accident tolerant fuel using the laser-driven chemical vapor deposition (LCVD) process. Both fuel in fiber (FiF) and fuel as fiber (FaF) designs are being demonstrated in fabrication evaluations, in part to prepare samples for autoclave testing, as well as continued performance modeling in partnership with MIT's Nuclear Science and Engineering department. FFF's high performance fiber manufacturing capabilities have significantly advanced to produce high volume, long, continuous fibers for advanced ceramic matrix composite (CMC) applications. This validates the LVCD approach for producing FiF and FaF-based fiber arrays necessary for composite layup and manufacturing. FiF development is focused on demonstration of precision spot-coating on the LCVD-produced long fiber substrates. FaF efforts are primarily driven to form a fiber forest on a substrate, utilizing a depleted uranium gas precursor to deposit uranium nitride and uranium disilicide materials in the individual fiber rods that compose the forest. FFF is exploring different matrices to complete the forest composite, including glass-based materials. These FaF forest composites are planned for chemistry evaluation in pressurized water reactor (PWR) conditions via autoclave testing.

2:20 PM

(ICACC-S13-003-2018) Synthesis and Thermophysical Properties of ThN

S. S. Parker*[1]; J. T. White[1]; A. Parkison[2]; P. Hosemann[3]; A. T. Nelson[1]

1. Los Alamos National Laboratory, Material Science and Technology Division, USA
2. Los Alamos National Lab, USA
3. University of California, Berkeley, Nuclear Engineering, USA

Thorium-based nuclear fuel cycles represent an intriguing alternative to conventional nuclear fuels. The majority of thorium fuels tested and analyzed to date are oxide or metal fuel forms, but a thorium cycle constituted using a nitride architecture would offer a number of performance benefits. Thorium mononitride (ThN) has a higher actinide density than thorium oxide and a significantly higher melting point compared to thorium metal. The thermal conductivity of ThN far exceeds that of UO_2 in the temperature range relevant to nuclear reactor operation. ThN produced by a novel carbothermic reduction to nitridation (CTRN) process was sintered to produce high density (>90% theoretical) pellets for initial thermophysical property measurements. The thermal diffusivity, heat capacity, and thermal expansion data for ThN from room temperature to 1700 K are presented and discussed with respect to the benefits of a mixed thorium-uranium nitride fuel cycle.

2:40 PM

(ICACC-S13-004-2018) Processing of urania fuel with novel additives starting from sol-gel feedstock

S. C. Finkeldei*[1]; R. Hunt[1]; J. Kiggans[1]; K. Terrani[1]

1. Oak Ridge National Lab, USA

Inclusion of additives in urania may be desired to enhance its thermal properties or fission product retention characteristics. In our earlier proof of concept study an increase in the thermal conductivity, up to 45%, was demonstrated for ZrO_2 based ceramics, by addition of molybdenum and its specific distribution and microstructure. These fabrication routes will be adopted and extended for the fabrication of urania ceramics with various additives. The influence of a variety of additives such as molybdenum, chromia, alumina, and silica as well as the impact of their concentrations will be studied regarding the thermal properties of the final ceramics and their microstructural characteristics. Moreover, the influence of different blending techniques will be explored. Finally, multiple additive urania ceramics will be fabricated to look for correlations between the various additives regarding the final properties of the ceramics.

3:20 PM

(ICACC-S13-005-2018) Electric Current Assisted Joining of SiC-based Ceramic Matrix Composites (Invited)

P. Tatarko*[1]; S. Grasso[2]; T. Saunders[2]; V. Casalegno[3]; M. Ferraris[3]; M. Reece[2]

1. Institute of Inorganic Chemistry, Slovak Academy of Sciences, Department of Ceramics, Slovakia
2. Queen Mary University of London, United Kingdom
3. Politecnico di Torino, Italy

Spark Plasma Sintering (SPS) machine was employed to join CVD-SiC coated C_f/SiC composites with a Ti interlayer utilising both indirect heating via graphite dies (standard SPS process) and direct heating using a die-less configuration. The purpose of the work was to bring together the flash processing and the joining of ceramics, thus allowing flash joining of ceramics. During the flash joining, a sound joint with the highest apparent shear strength of 31.4 MPa, which corresponds to the interlaminar shear strength of the composites, was obtained within just 7 seconds. An extremely rapid heating rate of 9,600°C/min combined with a very short processing time significantly suppressed the diffusion of both C and Si into the Ti interlayer. A maximum corresponding joining temperature was ~ 1250°C at the maximum heating power of 2.2 kW. When a standard SPS process was applied (heating rate of 100°C/min), formation of the reaction products at the interface suppressed the solid state diffusion bonding at the same temperature of ~1250°C. The temperature as high as 1700°C was necessary to achieve bonding. This led to the in-situ formation of a MAX phase Ti_3SiC_2 in the joining area, accompanying with significant shrinkage and infiltration. The flash joining results showed a significant reduction in a processing time and the maximum temperature required, when compared to the standard SPS joining processes.

3:50 PM

(ICACC-S13-006-2018) Arc Welding of SiC Based Ceramics

D. King*[1]; J. Watts[2]; K. Cissel[1]; S. Ganti[1]; A. Kadhim[1]; G. Hilmas[2]; W. Fahrenholtz[2]

1. UES, Inc., USA
2. Missouri University of Science & Technology, USA

Plasma arc welding and gas tungsten arc welding techniques were explored as potential joining technologies for the fabrication of complex ceramic shapes. Flat plates of SiC-based monolithic ceramics with a nominal composition of 50 vol% SiC, 35 vol% ZrB_2, and 15 vol% ZrC were first fabricated by hot-pressing. Hot-pressed plates were then used in welding experiments to determine the plasma arc welding parameters necessary for fusion welding of the parent material. The microstructure of the resulting fusion zones were examined to analyze the solidification path of the weld pool. The strength of the joined monolith was compared with the base material through four-point flexure testing. Successful demonstration of butt welds was utilized to demonstrate that arc welding techniques could be used to join ceramics in other joint configurations, for the fabrication of complex shapes. The fabrication and arc welding of fiber reinforced ceramics was also explored and will be discussed.

4:10 PM

(ICACC-S13-007-2018) Development of the High Temperature Joints for Carbon-Based Materials

R. Piat*[1]; B. Reznik[2]; M. Rohde[2]; S. Lichtenberg[2]; O. Deutschmann[2]; H. J. Seifert[2]

1. Darmstadt University of Applied Science, Germany
2. Karlsruhe Institute of Technology, Germany

Due to their light weight, exceptional strength, stiffness, structural stability at elevated temperatures and the possibility to tailor their thermo-mechanical properties by microstructure design, carbon-based composites are the material of choice for the high-temperature applications. Some examples of the application of these materials are nuclear safety structures or a thermal protection material used for spacecraft heat shields. One of the important questions by utilization of these materials is in which way connect different components produced from these materials to largescale or complex shape component without reduction resulting thermo-mechanical properties or degradation of the whole structure. In last year's several methods were developed for joining carbon-based materials, such as glass material bonding and reactive metal brazing etc. Nevertheless, joints produced with above-mentioned interlayer material only can be used for low temperature application. In presented studies components consisting of the high textured Pyrolytic Graphite were joined using chemical vapour infiltration technique. In this way the resulting joined material is pour carbon-based structure, the joining layer consists of Pyrolytic carbon and can be used for the high temperature applications. The light microscopic, thermal conductivity and resistance studies of the obtained structure were provided.

4:30 PM

(ICACC-S13-009-2018) Thermal conductivity modeling of UN/U_3Si_2 composites

A. W. Travis*[1]; J. T. White[2]; A. T. Nelson[2]

1. University of California, Irvine, USA
2. Los Alamos National Lab, USA

The thermal conductivity of novel uranium nitride (UN) and uranium silicide (U_3Si_2) composites is investigated for potential application as an accident tolerant fuel with higher uranium density than conventional UO_2 nuclear fuel. Specimens of various compositions ranging from 10 vol% to 40 vol% U_3Si_2 with balance UN were fabricated and then characterized via SEM and XRD. Microstructures from SEM images are used as the input for thermal conductivity modeling using a combination of OOF2 (Object oriented finite element analysis version 2) and MOOSE (Multiphysics object oriented simulation environment). Single phase thermal conductivity values for each phase are used as inputs for simulations from 373 K to 1673 K. Using pure UN and U_3Si_2 values yielded MOOSE model results that were ~9% higher than the corresponding experimental results, determined from a combination of thermal diffusivity, specific heat capacity, and density measurements. This deviation is attributed to an unknown U-Si-N phase with unknown thermal properties. The thermal conductivity of this phase is estimated using a Rule of Mixtures calculation and used in subsequent models. With the addition of the third unknown phase into the MOOSE models, the experimental and simulated results for the 20 vol% - 40 vol% U_3Si_2 composites show good agreement. Thermal conductivity of irradiated material is also estimated by assuming thermal degradation in the U_3Si_2 phase.

4:50 PM

(ICACC-S13-010-2018) Numerical Analysis of Multi-pellet Fabrication of UO_2 fuel pellets using Spark Plasma Sintering

B. Nili*[1]; G. Subhash[1]; J. S. Tulenko[1]

1. University of Florida, Material Science and Engineering, Nuclear Engineering Program, USA

In recent years, spark plasma sintering (SPS) techniques has been successfully used to sinter net-shaped uranium dioxide (UO_2) fuel pellets with 97% density. Compared to conventional sintering, in SPS, these fuels have been sintered in a significantly lower processing time (few minutes) and lower sintering temperatures (as low as 1100 °C). The quality of sintered fuel pellets for in-reactor fission performance requires high quality of pellets with uniform microstructure and fewer defects. Therefore, a detailed understanding of the science behind the sintering process using SPS is necessary in order to improve the reproducibility of fuel pellet fabrication, which is expected to enhance reactor performance. Towards this end, a coupled thermal-electrical-mechanical finite element (FE) model is implemented to analyze the consolidation process of UO_2 powder at elevated temperatures and pressures. The results indicate that considerable heterogeneity in temperature distribution exists in the tooling assembly, especially when multi-pellet fabrication is pursued. New strategies for obtaining uniform temperature distribution and consequently homogeneous microstructures are proposed.

S14: Crystalline Materials for Electrical, Optical and Medical Applications

Phosphor

Room: Tomoka C

Session Chairs: Joanna McKittrick, UC San Diego; Isabel Kinski, Fraunhofer IKTS

1:30 PM

(ICACC-S14-001-2018) Optimization of polycrystalline ceramic phosphors for applications with blue-light excitation by laser and light-emitting diodes (Invited)

I. Kinski*[1]; G. Eberhardt[2]; M. Kunzer[3]; M. Arnold[4]

1. Fraunhofer IKTS, Business Division Optics, Germany
2. Laser Display Technology GmbH, Germany
3. Fraunhofer IAF, Optoelectronic, Germany
4. Fraunhofer IKTS, Functional Materials for Hybrid Microsystems, Germany

For the generation of white light the most commonly used approach is based on colour mixing of blue and yellow light to achieve the desired white light. The yttrium aluminium garnet (YAG) doped with cerium is used as inorganic phosphor for the conversion of the blue excitation light into yellow light. For high power white light sources thin ceramic discs are crucial for high luminance, and luminous efficacy. It is important to adapt the microstructure of the phosphor ceramic that is used as light conversion component to the specific excitation source, the geometry, and module design. Depending on the excitation source (LED or laser) as well as the arrangement of the ceramic disc in either transmissive or reflective geometry, the microstructure of the polycrystalline ceramic discs has to be changed, respectively in order to provide still high luminous efficacies. Additionally, the surface finish influences the efficiency strongly next to the surface microstructure and volume scattering centers. In and out coupling of light can be adjusted by different surface finishes, while the microstructure influences the propagation of light and heat dissipation. In order to control the angular-dependent colour impression volume scattering in the ceramic converter discs and structuring of the surface have been investigated regarding their influence.

2:00 PM

(ICACC-S14-002-2018) Crystal Growth of Silicate Phosphors using SiO Vapor (Invited)

K. Toda*[1]

1. Niigata University, Japan

In this syudy, we report a new solid-vapor hybrid synthesis technique of silicate phosphors using gaseous SiO raw material. Vaporization of SiO was performed at over 1673 K from a silica source (or SiO powder) in H_2-Ar reduction atmosphere. The heating of the silica source (or SiO powder) in strong reductive atmosphere occurs to generate gaseous SiO, which reacted with the other raw materials powders on the substrate. In this method, well-grown powders with a micrometer size were mainly observed because of relatively slow nucleation. As an example, green emitting $Ba_2SiO_4:Eu^{2+}$ phosphor can be synthesized by a new solid-vapor phase technique. Emission intensity of the phosphor synthesized by new solid-vapor phase technique at 1723 K is about 2.6 times higher than that of a conventional solid state reaction sample. This is due to high crystallinity and relatively few defects for the solid-vapor phase processed samples. The developed solid-vapor hybrid process is a general and powerful tool for synthesizing the well-crystalline and -grown silicate phosphors. To the best of our knowledge, no such a solid-vapor synthetic process for the production of well-grown silicate phosphors has been found up to now.

2:30 PM

(ICACC-S14-003-2018) Development of phosphor-in-glass for high power solid state lighting system (Invited)

S. Kim*[1]; Y. Nam[1]; B. Kim[1]; J. Hwang[1]; J. Kim[1]; Y. Lee[1]; D. Jeon[1]

1. Korea Institute of Ceramic Engineering and Technology (KICET), Republic of Korea

Recently, the phosphor-in-glass (PiG) has been extensively investigated as the promising materials for high power solid state lighting system due to their excellent thermal and chemical stabilities, good heat- or light-resistance and ease of formability. Several studies have been conducted to achieve the high optical efficiency of the PiG by modifying the refractive index of the glass, composition of glass, as well as the particle size of the phosphor and content of the phosphor in glass. In our research group, we have focused on glass materials, such as the composition, light transmission, glass frit size, to realize the novel PiG with a high luminescent efficiency. It was demonstrated in our previous studies that the luminescence efficiency of the PiG strongly depended on the glass materials. In this study, we present the luminescent properties of the PiG controlled the glass composition, frit size, and transmission and discusses on the effect of the glass materials on the luminescent properties of the PiG.

3:20 PM

(ICACC-S14-004-2018) Phosphor composition prediction and synthesis using a combined experimental and computational approach (Invited)

J. McKittrick*[1]; J. Ha[1]; Z. Wang[1]; O. Graeve[1]; S. Ong[1]

1. UC San Diego, USA

The search for new phosphor compositions that can be activated by near UV-LEDs (370 – 410 nm) is needed to (1) extend the color range and provide a high color rendering index, (2) increase quantum efficiency and (3) improve thermal stability. A high throughput screening of quaternary oxides was carried out. The motivation for this is that ternary compositions are well explored. We chose oxides because they are easier to synthesize than nitrides. A focus was placed on light, electropositive elements to enhance the structural rigidity and to be able to tune the band gap. We have discovered new phosphors for various Eu^{2+}-activated complex oxide compositions by calculating the band structure and projected orbital density of states from which the excitation wavelength can be determined. The new compositions were fabricated by a variety of wet chemical synthesis methods along with solid-state reaction. The excitation and emission wavelengths were measured along with the quantum efficiencies and thermal stability. These and other pertinent topics will be discussed. This research is supported by NSF Ceramics Program Grant # DMR-1411192.

3:50 PM

(ICACC-S14-005-2018) Exploration of novel silicate phosphors by crystal-site engineering approach (Invited)

Y. Sato*[1]; K. Tomita[2]; M. Kakihana[3]

1. Okayama University of Science, Department of Chemistry, Faculty of Science, Japan
2. Tokai University, Department of Chemistry, School of Science, Japan
3. Tohoku University, Institute of Multidisciplinary Research for Advanced Materials, Japan

Silicon-based nitride phosphors such as $CaAlSiN_3$:Eu^{2+} and $Sr_2Si_5N_8$:Eu^{2+} exhibit an excellent red-light emission excited by blue-light region. However, it is difficult to prepare these silicon-based nitride phosphors in pure form with uniform particle size because it should be needed to sinter precursor powders under high pressure and temperature conditions. On the contrary, Eu^{2+}-activated silicates with alkaline-earth ions have been reported as high efficient phosphors. Such silicate phosphors in pure form can be easily obtained by simple preparation techniques without any critical conditions for silicon-based nitrides mentioned above. Therefore, productivity and production cost for silicate phosphors are advantageous for the commercial production of red phosphors. We have recently found new red-emitting Ca_2SiO_4:Eu^{2+} phosphors with higher Eu^{2+} concentrations at 20-40 mol.% excited by blue-light region. In the case of the Ca_2SiO_4:Eu^{2+} phosphors, from a crystallographic point of view, it is possible to customize the luminescence color of Ca_2SiO_4:Eu^{2+} by employing crystal-site engineering (CSE) approach. Other silicate phosphors includeing M_2SiO_4:Eu^{2+} (M =Sr and Ba) and $Ca_3ZrSi_2O_9$:Eu^{2+} are also discovered by the CSE approach, the results of which will be given at the day of our presentation.

4:20 PM

(ICACC-S14-006-2018) Plate and powder form of single crystal phosphors for high-brightness white lighting

K. Shimamura*[1]; V. Garcia[1]; D. Inomata[2]; A. Ito[3]

1. National Institute for Materials Science, Japan
2. Tamura Corporation, Japan
3. Koha Co., Ltd., Japan

Currently, production is based on ceramic powder phosphors (CPPs) embedded in binders with a low thermal conductivity, which easily overheat and degrade under high blue irradiation. In order to overcome these drawbacks in high-brightness (HB) applications, we have proposed the use of single-crystal phosphors (SCPs), which exhibit a superior conversion efficiency, a high thermal stability and a low temperature increase under high-power irradiation. SCPs possess a high purity and crystalline quality, thus guarantying best performance in either bulk or powder form. The best yellow phosphor for white LEDs is Ce:YAG, and its emission can be gradually tuned towards the red and the green by Gd and Lu substitution on the Y-site, respectively. Independently of the Lu content, SCPs exhibit an outstanding internal quantum efficiency (QE); at room temperature it's over 95%, and with the temperature rise it increases even further, reaching a maximum at about 250°C. Instead, the internal QE of CPPs even quenches at much lower temperatures, indicating the presence of undesired non-radiative recombination paths. These show that, SCPs meet the fundamental requirements for emerging HB applications such as laser headlights and laser projectors. Binder-free SCP powder plate is demonstrated, and it has shown the performance as SCP plates.

4:40 PM

(ICACC-S14-007-2018) Microstructures and their relevance to photoluminescence in $SrAl_2O_4$

S. Mori*[1]

1. Osaka Prefecture University, Materials Science, Japan

$SrAl_2O_4$ is a promising host material exhibiting long afterglow luminescence and elasticoluminescence behaviors, which are associated with changes in the local electronic structure due to strain fields induced by the application of an external stress. Recently, some anomalies of elastic properties have been reported in the monoclinic phase below 650 K. It is suggested that these anomalies of elastic behaviors are related with the motion of defects such as domain walls. It is important to elucidate the ferroelastic microstructures for understanding elastic behaviors and mechanoelectroc-optical properties in $SrAl_2O_4$. In order to understand unique photoluminescence effect in Eu^{2+} doped $SrAl_2O_4$, we have investgated crystal structure and its associated microstructures of the monoclinic phase with the $P2_1$ space group mainly by transmission electron microscope (TEM) experiments, combined with powder x-ray diffraction experiments. Electron diffraction experiments showed that the monoclinic phase of $Eu_{0.02}Sr_{0.98}Al_2O_4$ should be characterized as the modulated structure with the modulation vector of q=0 1/2 0. High-resolution TEM experiments revealed that the monoclinic phase can be characterized as microstructures due to the modulated structures with nanometer-sized planer structures. These nanosized modulated structures should be relevant to the characteristic photoluminescence effect found in Eu^{2+} doped $SrAl_2O_4$.

5:00 PM

(ICACC-S14-008-2018) Sol-Gel-Derived $CaTiO_3$:Pr^{3+} Thin Films for Wavelength Conversion (Invited)

T. Hayakawa*[1]; H. Nakamori[1]

1. Nagoya Institute of Technology, Department of Life Science and Applied Chemistry, Japan

Development of down-shift phosphors which convert near-ultraviolet(UV) to visible/near-infrared light is required in active research fields of solar cells and white light emitting diodes (W-LEDs). Recently, we reported synthesis of transparent thin films of Pr^{3+}-doped $CaTiO_3$ and their sharp, red photo-/cathode-luminescence (PL/CL) for optoelectric applications (Y.Ishikawa, T.Hayakawa, K.Inoue, M.Nogami, J.Sol-Gel Sci.Technol. 65 (2013) 324.). The thin films exhibited strong UV absorption in the range of 250-340 nm, which should however be extended to longer wavelengths for photon harvest in solar cells. In the presentation, we'll report our facile synthesis of $(Ca,Bi)TiO_3$:Pr^{3+} and Bi-assisted photon conversion of near-UV to visible light. Under 350 nm excitation, the synthesized Pr^{3+}-doped $Ca_{1-x}Bi_xTiO_3$ (CBTO) thin films (x=0~0.20) exhibited a sharp PL line at 612 nm assigned to $^1D_2$-$^3H_4$ transition of Pr^{3+} ions. For non-Bi-doped CTO:Pr^{3+} film the PL had a excitation peak at 315 nm and rather small PL intensity under 350 nm excitation. When Bi^{3+} ions were introduced, the excitation band was extended to longer wavelength up to 400 nm and the red PL intensity under 350 nm excitation was increased, which was due to metal-to-metal charge transfer (MMCT) band of Bi^{3+} ions, meaning efficient energy transfer from Bi^{3+} to Pr^{3+} ions, applicable as photon harvest materials in solar cells.

S15: Additive Manufacturing and 3-D Printing Technologies

Stereolithography I

Room: Coquina Salon B

Session Chair: Soshu Kirihara, Osaka University

1:30 PM

(ICACC-S15-001-2018) Ceramic Additive Manufacturing: From Basic Science to Aerospace Opportunities (Invited)

M. B. Dickerson*[1]; L. M. Rueschhoff[1]; L. A. Baldwin[1]; C. C. Wyckoff[1]; T. Pruyn[1]; G. Wilks[1]; Z. D. Apostolov[1]; T. Key[1]; H. Koerner[1]; M. Dalton[1]; M. Cinibulk[1]

1. Air Force Research Laboratory, Materials and Manufacturing Directorate, USA

Advancements in additive manufacturing (AM) technologies and the proliferation of relatively inexpensive 3D printing systems have fueled an explosive increase in the capabilities and interest in bottom-up fabrication methodologies. Though much of the AM field is devoted to the printing of select metal alloys and relatively low-performance polymers, recent innovations in the processing of ceramics present considerable opportunities for scientific study and practical application. The digital design, complexity, and locally tailorable properties afforded by AM are especially enabling for the manufacture of ceramics, which have been challenging to shape by traditional top-down approaches. In this presentation, we will provide an overview of the opportunities and limitations of state-of-the-art ceramic AM for aerospace applications. Additionally, efforts in the development of pre-ceramic polymer-based inks and resins for stereo lithography (SLA), multi-photon lithography (MPL), and direct ink write (DIW) extrusion printing will be discussed. Effects of polymer chemistry and rheology on printability, ceramic composition, and high-temperature performance of ceramic AM builds will be detailed.

2:00 PM

(ICACC-S15-002-2018) Advances in lithographic additive manufacturing of dense ceramics – non-oxides and translucent materials

M. Schwentenwein*[1]; A. A. Altun[1]; J. Homa[1]

1. Lithoz GmbH, Austria

This contribution focuses on new developments associated with the lithographic-based ceramic manufacturing (LCM) process. While lithographic techniques in general have taken a leading position in additive manufacturing (AM) of highly precise and strong ceramic parts, the available material portfolio is significantly smaller than for powder bed processes or extrusion-based techniques. This is mainly due to the difficult processing of darker powders because of light absorption and scattering phenomena. By optimizing the photocurable suspensions towards very high reactivity the printing of materials such silicon nitride, silicon carbide and cermets could be realized. Especially in the case of silicon nitride-based ceramics, it was already possible to produce 3D printed parts with mechanical properties which are at eye-level to conventionally formed components. Very recently it could also be demonstrated that lithographic AM can also be used to fabricate translucent alumina parts with a grain size below 1 µm, underlining the extremely high quality of printed parts that can be obtained by using this approach.

2:20 PM

(ICACC-S15-003-2018) Additive manufacturing of flexible 3-3 ferrorelectric ceramic/polymer composite based on triply periodic cellular micro-skeleton

X. Song*[1]; L. He[1]; W. Wang[2]; Z. Wang[2]; L. Chen[2]

1. University of Iowa, Mechanical and Industrial Engineering, USA
2. Mississippi State University, Mechanical Engineering, USA

3-3 ferrorelectric ceramic/polymer composites, i.e. three-dimensional (3D) interconnected piezocomposites with continuous piezoelectric ceramic skeleton, possess an excellent property combination of mechanical flexibility and piezoelectricity, compared with other materials including pure ceramics, 0-3, 1-3 and 2-2 piezocomposites. While piezoelectric performances of 3-3 piezocomposites are dependent on the meso-scale geometry of the active piezoelectric ceramic phase in the composite, current manufacturing processes are extremely limited in defining a desired meso-scale geometry in a 3-3 piezocomposite. In this research, we investigate the fabrication of 3-3 piezocomposites with predefined ceramic micro-skeletons utilizing a Suspension Enveloped Projection Stereolithography process (SEPS). Triply periodic cellular structures are used in the design of the active piezoelectric ceramic phase due to their larger elastic constants with respect to their rod-connected counterparts. Post processes including debinding, sintering, poling and polymer infiltration are presented. Mechanical and piezoelectric properties of final 3-3 piezo-composites are analyzed by experimental characterization and finite element modeling.

2:40 PM

(ICACC-S15-005-2018) Stereolithographic Additive Manufacturing of Micro Ceramic Patterns by Ultraviolet Laser Dewaxing and Sintering

S. Kirihara*[1]

1. Osaka University, Joining and Welding Research Institute, Japan

In stereolithographic additive manufacturing, cross sections were created through photo polymerization by UV laser drawing on spread resin paste including ceramic nanoparticles, and composite models were sterically printed by layer lamination. An automatic collimeter was equipped with the laser scanner to adjust beam diameter. Fine or coarse beams could realize high resolution or wide area drawings, respectively. As the row material, nanometer sized ceramic particles were dispersed in to photo sensitive liquid resins at

50 % in volume fraction. These materials were mixed and deformed to obtained thixotropic slurry for 15 min at 700 and 300 rpm of rotation and revolution speeds, respectively. The resin paste was spread on a glass substrate at 100 μm in layer thickness by a mechanically moved knife edge. An ultraviolet laser beam of 355 nm in wavelength was adjusted at 50 μm in variable diameter and scanned on the spread resin surface. Irradiation power was increased at 1 W for resin dewaxing and powder sintering through heating by ultraviolet ray propagations, resonations and absorptions in the paste materials. In this investigation, micro emboss patterns with square pits of 100×100×70 μm in edge sizes were opened in solid electrolyte lithium-lantern-zirconate (LLZ) sheet of 100 μm in thickness were processed to applied for all solid batteries.

Stereolithography II

Room: Coquina Salon B

Session Chair: Martin Schwentenwein, Lithoz GmbH

3:20 PM

(ICACC-S15-004-2018) Influence of layer orientation on microstructure and mechanical properties of ceramics processed by stereolithography (Invited)

A. L. Leriche*[1]; M. Dehurtevent[3]; F. Petit[2]; J. Hornez[1]; A. Thuault[1]; L. Robberecht[3]; P. Behin[3]; F. J. Cambier[2]

1. University of Valenciennes, France, France
2. BCRC, RSE, Belgium
3. University of Lille 2, Dental Faculty, France

Subtractive CAD-CAM technologies have led to major improvements in dentistry, making possible to produce reliable restorations with accurate dimensions while reducing manufacturing time. Such processes are limited by waste of raw materials, difficulty in recycling excess material and heavy wear of tools. Microscopic cracks weakening restorations can appear due to the milling. Additive manufacturing processes avoid such limitations. Impact of slurry composition on mechanical properties has been extensively studied but not the influence of layer orientation. In this talk, we compare mechanical properties of dense alumina and ZTA processed by stereolithography, as a function of the layers orientation. Samples were processed for 3 orientations: XY, ZX, ZY. Sizes were 1.3x4x22 mm. Physical properties of ceramics were assessed as well as toughness and 3-point flexural strength. No difference of density was found regarding the orientation of printed layers but fracture toughness along XY was found significantly higher than with ZX. All samples had large grains whose major axis was parallel to the layer orientation. The fracture surfaces of XY and ZY manufactured samples were transgranular whereas intergranular for ZX manufactured samples. These results will be discussed and compared with results published in the literature concerning electrophoretic deposition process

3:50 PM

(ICACC-S15-035-2018) Inexpensive additive manufacturing using waste materials

H. Colorado*[1]

1. Universidad de Antioquia, CCComposites Laboratory, Colombia

Additive manufacturing is an emerging technique growing dramatically worldwide and changing the way we do manufacturing and use materials science in many applications. Waste is a problem difficult to solve because the increasing population of the world and complex chemistry and processing involved in many recycling solutions. The method can be applied worldwide because is simple and inexpensive. The circular economy concept is also discussed in this presentation. This research explore these questions and shows progress using diverse wastes in an inexpensive processing using the direct ink writing technique. Some of the materials utilized in this research are considered as hazardous, however, used in combination with clays or cements can be converted in useful solutions. The materials microstructure and their derived properties are also presented.

4:10 PM

(ICACC-S15-006-2018) Digital Light Processing of Wollastonite-Diopside Glass-ceramic Complex Structures

J. E. Schmidt*[1]; H. Elsayed[1]; E. Bernardo[1]; P. Colombo[1]

1. University of Padova, Industrial Engineering, Italy

Highly complex glass-ceramic structures were fabricated starting from a photocurable polymer filled with glass particles (<45 μm) crystallizing into wollastonite and diopside upon heating. Via Digital Light Processing (DLP), the mixture was exposed in a stereolithography 3D printer, which polymerised the photosensitive polymer in an appropriate time frame. The glass particles were trapped in the cured polymer and highly porous structures were built up layer-by-layer. The heat treatment in air, up to 1100 °C, caused the burn-out of the polymer and the sintering of glass particles. The crystallization impeded any viscous collapse, so that a complete shape maintenance was observed after firing, with a homogeneous shrinkage. The developed structures with porosity up to about 80 vol% exhibited a very good compressive strength (> 10 MPa). The bioactivity of wollastonite-diopside glass-ceramics combined with the design freedom and high resolution of 3D stereolithography printing make the structures suitable candidates for bone tissue engineering.

Stereolithography III

Room: Coquina Salon B

Session Chair: Anne Leriche, University of Valenciennes

4:30 PM

(ICACC-S15-007-2018) Additive Manufacturing of Reinforced Polymer-Derived Ceramics

Z. C. Eckel[1]; P. P. Bui[1]; J. M. Hundley[1]; T. Schaedler*[1]

1. HRL Laboratories, USA

We report advances in ceramic stereolithography using resins based on preceramic monomers. UV curable siloxane formulations were 3D printed with conventional stereolithography printers resulting in complex shaped polymer parts that were pyrolyzed to silicon oxycarbide with uniform shrinkage and virtually no porosity. Ceramic particles and whiskers were added to the resins in volume fractions of 10 - 50% and the effects on shrinkage and mechanical properties were investigated. In contrast to conventional ceramic particle filled resins and inks, the liquid resin does not leave the material on heat treatment, but pyrolyzes to a ceramic matrix embedding the particles or whiskers. Novel preceramic resin formulations were synthesized to reduce the oxygen content in the resulting ceramic and increase high temperature properties. Potential applications for this technology will be presented, including first results of a NASA program on additively manufactured ceramic rocket engine components.

4:50 PM

(ICACC-S15-008-2018) Stereolithographic Additive Manufacturing of Ceramic Chambers to Suppress Noise Generations from High Speed Gas Flames

S. Kisanuki*[2]; S. Kirihara[1]

1. Joining and Welding Research Institute, Japan
2. Osaka University, Graduate School of Engineering, Japan

By using stereolithographic additive manufacturing, phononic crystal structures with periodic arrangements of air cavities were fabricated to modulate acoustic wave propagations in an audible range. Acoustic chambers with inner ribs were formed along the even interval to diffract the audible wavelength comparable to the structural periodicity. In the stereolithography process, photosensitive resin including with alumina particles were spread on glass

substrate by using mechanical knife edge. Subsequently, an ultraviolet laser beam of 355 nm in wavelength was focused into 100 μm in diameter and scanned to draw a cross sectional solid pattern. The formed composite precursor was obtained successfully through continuous laminations. Alumina components were obtained through dewaxing and sintering in the air atmosphere. By using computer aided design, manufacture and evaluation, ceramic soundproofs were processed to suppress the generation of noise form thermal spraying guns. The periodic arrangements of acoustic cavities were geometrically modulated according to simulated acoustic spectra by computational fluid dynamics.

5:10 PM

(ICACC-S15-009-2018) Additive Manufacturing of Ceramics from Preceramic Polymers: From Nanometer to Centimeter

J. E. Schmidt*[1]; L. Brigo[1]; G. Brusatin[1]; P. Colombo[1]

1. University of Padova, Industrial Engineering, Italy

A novel approach to fabricate SiOC ceramic structures across different length scales by additive manufacturing using stereolithography has been developed by utilizing a simple physical blend between different commercially available preceramic polysiloxanes. Two silicone resins (Silres 601 and H44), both possessing a high ceramic yield, lead to a homogenous stable solution upon mixing with photosensitive RC 711. Different blend ratios have been realized and respectively optimized concerning printing parameters and additives. Exact replications of highly complex structures were fabricated by Digital Light Processing (DLP) for macrofabrication and 2-Photon-Polymerization (TPP) for microfabrication in an appropriate amount of time. After pyrolysis, a uniform, homogenous shrinkage was observed yielding dense, pore- and crack-free SiOC ceramics. We present the DLP and TPP fabrication of SiOC ceramic macro- (cm scale) and micro-fabricated parts (sub μm scale) starting from commercially available preceramic polysiloxanes. By adjusting the ratio between the different polysiloxanes, parameters such as the ceramic yield, shrinkage, chemical composition and resolution after pyrolysis could be tailored in a wide range of values for macro- and micro- scale ceramics.

S17: Advanced Ceramic Materials and Processing for Photonics and Energy

Synthesis

Room: Halifax A/B

Session Chairs: Christine Luscombe, University of Washington; Clara Santato, Ecole Polytechnque de Montreal

1:30 PM

(ICACC-S17-001-2018) Convergence in the Energy Sector (Invited)

L. D. Madsen*[1]

1. National Science Foundation, Materials Research, USA

The U.S. National Science Foundation (NSF) is well known for its support of basic research. However, efforts are also made by NSF to engage a broad spectrum of the workforce and to address global challenges. To this end, convergence – a new thrust – defined as "the deep integration of knowledge, techniques, and expertise to form new and expanded frameworks for addressing scientific and societal challenges and opportunities". Experts from different disciplines, in the pursuit of common research challenges, intermingle or integrate their knowledge, theories, methods, data, research communities and languages. New frameworks, paradigms or disciplines can result from these sustained interactions across multiple communities. Efforts are driven by specific and compelling challenges arising from deep scientific questions or pressing societal needs. Additionally, federal funding is often needed to effectively transition fundamental academic research into the commercial sector. Many funding modalities support this goal. Examples from the energy sector will be used to illustrate both convergence and translation of research into commercial products.

2:00 PM

(ICACC-S17-002-2018) Solution based synthesis of complex shape and composition nano-structures for energy applications (Invited)

G. Westin*[1]

1. Uppsala University, Sweden

For solar-cells, fuel cells and photo-catalysts for fuel generation the devices are becoming ever more complex and built from several nano-sized components where ultra-thin coatings for control of corrosion, tunneling or band-bending has become an important component. At the same time, to make any societal impact the devices have to be low cost which requires efficient processes capable of making high quality, multi-phase complex materials in one or few steps, which calls for increased understanding of materials synthesis processes. Here we describe solution based synthesis routes using metal alkoxides and organically coordinated metal salts to achieve complex oxides and nano-composites with a structures useful for energy conversion and storage. Oxides of varying complexities including doped and non-doped Fe_2O_3, TiO_2, Ln_2O_3 and ZnO in the forms of nano-particles, nano-sponges and thin- and ultra-thin films will be described, as well as metals and alloys as nano-particles, thin films and nano-sponges. The influence of the precursor, reaction kinetics and thermal treatment will be discussed in relation to the structures and properties obtained, as well as the possible synthesis of oxides with extended, metastable doping-levels. The syntheses and products were studied with a wide array of analytical techniques including; SEM, TEM, XRD, TGA, DSC/DTA, IR and Raman spectroscopy.

2:30 PM

(ICACC-S17-003-2018) Modulating the Surface Chemical Composition of Titania Nanocrystals by Solvothermal Synthesis and Surface Related Applications (Invited)

M. Epifani*[1]

1. CNR-IMM, Italy

Among the various photocatalysts, titania (TiO2) is a widely accepted benchmark material, due to its high photoreactivity, abundance, stability and low cost. On the other hand, the need of increasing the range of optical absorption of pure TiO2 has readily emerged, and has triggered a variety of methods for modifying its composition and optical properties. The modification of the optical properties is only a facet of a more general restructuring of the electronic properties, when the band structure of titania is modified by addition of additional levels due to any additive, in a general sense. In fact, in this work the synthesis and chemical modification of titania will be discussed as triggered by gas-sensors field. The first topic therefore will be the chemical synthesis of titania nanocrystals by solvothermal method. Then the chemical modification of the resulting nanocrystals will be illustrated, highlighting the chemical principles involved in the synthesis. As first examples of the properties of the resulting materials, the gas-sensing properties of W, V and Mo modified titania nanocrystals will be reviewed. Finally, the solar cell application of Mo modified anatase nanocrystals will be discussed, as an example of more general electronic modification induced by chemical modification of titania.

3:20 PM

(ICACC-S17-004-2018) Synthesis and Multifunctions of Titania Nanotubes-based Low-dimensional Anisotropic Nanocomposites (Invited)

T. Sekino*[1]; S. Eom[1]; Y. Yamasaki[1]; H. Nishida[1]; S. Chou[1]; T. Goto[1]

1. Osaka University, The Institute of Scientific and Industrial Research, Japan

This study focuses on the development of titania nanotubes (TNTs) based low-dimensional nanocomposites combined with nanostructured 1-dimensional (1D) or 2-dimensional (2D) carbons. Series of low-D Carbon/TNT nanocomposites have been synthesized through the solution chemical process with various carbon ratio. When Carbon nanotube (CNT) was chosen, unique core-shell 1D/1D CNT/TNT nanocomposite could be successfully obtained, where CNT core was surrounded by rolled shell form of TNT. In contrast, 1D/2D nanocomposites could be fabricated by combining graphene oxide (G0) which was 2D nanocarbon. It exhibited sheet structure, where TNT decorated on the graphene substrate. These low-dimensional carbon/TNT nanocomposites exhibited new absorption bands in UV-visible spectra. Electrical resistivity was decreased with the addition of nanocarbon, however, it was slightly higher than that of simply-mixed TNT and carbon powders. These facts was considered due to their unique composite structures, such as anisotropic core-shell structure. In addition, these nanocomposites exhibited better gas sensing properties, implying the carrier transport performance of titania which intrinsic mobility is sufficiently low might be greatly enhanced. Detailed anisotropic nanocomposite structures and various properties will be discussed.

3:50 PM

(ICACC-S17-005-2018) Controlling defects – and properties - in 3-D assemblies of oxide nanosheets (Invited)

S. T. Misture*[1]; P. Metz[1]; P. Gao[1]

1. Alfred University, MSE, USA

X-ray total scattering was teamed with X-ray spectroscopy and related tools to probe both the mesostructure and the atomic defects of titanate, niobate and MnO_2 nanosheet assemblies, revealing a direct link between surface defects and optical, photochemical and electrochemical function. Of particular interest is the ability to triple the electrochemical specific capacitance of MnO2 nanostructures by introducing surface Frenkel defects. Beginning with high-perfection microcrystalline K_xMnO_2 powders, it is possible to exfoliate defect-free MnO_2 nanosheets and subsequently reassemble them into 3-D porous structures. Controlled reduction of some of the tetravalent Mn leads to tripling of the electrochemical charge storage capacity. While the effects of defects are well-understood for bulk solids, we explore the case where the solid is 2-D and hence "all" surface. The talk will highlight the use of high energy X-ray scattering and X-ray spectroscopy, among other tools, needed to quantify the defects and to link the defect content to the measured properties.

4:20 PM

(ICACC-S17-006-2018) Low-Symmetry Colloidal Nanocrystals (Invited)

P. Cozzoli*[1]

1. University of Salento, Department of Mathematics and Physics "E. De Giorgi", Italy

Colloidal inorganic nanocrystals (NCs), free-standing crystalline nanostructures grown in solution phase, stand out in the realm of nanomaterials owing to the precision with which their properties can be controlled through tailoring their structural and geometric features in the synthesis stage, and to the flexibility with which they can be processed and exploited in disparate fields. A smart, yet challenging paradigm to engineer, diversify and enrich the physical-chemical preorogatives and technological potential of single-material NCs relies on designing and creating unconventional architectures, in which diverse geometric and topological motifs are integrated across multiple structure and dimensionality levels within reduced-symmetry frameworks. However, due to currently restricted synthetic capabilities and mechanistic knowledge, low-symmetry nanostructures are still under-represented in the colloidal domain, remaining limited to NCs with broken circular or spherical symmetry, and, more rarely, with chiral habits. In this talk, I will report on recent progress made by us in the controlled growth and characterization of single-crystalline NCs of oxide, magnetic and plasmonic semiconductor materials, which can be tailored in monodisperse low-symmetric shapes integrating 1D- 2D dimensionality with 2D-3D connectivity and/or anisotropic curvature. The technological potential offered by such brand-new NCs will be highlighted.

4:50 PM

(ICACC-S17-007-2018) Synthesis of surface modified high aspect ratio akaganeite nanorods with exchange bias at room temperature

H. Khalid*[1]; S. Heo[1]; W. Yang[1]; B. Kim[1]; T. Kim[1]; S. Seo[1]

1. Korea Institute of Industrial Technology, Republic of Korea

Akaganeite (β –FeO(OH)) finds its technological importance as catalytic materials, sorbents, gas sensors, ion exchanger and biomedical applications. Akaganeite with its antiferromagnetic behavior is known to show exchange bias at low temperature. In this study surface morphology of the akaganeite nanorods were tailored in order to achieve exchange bias effect close to room temperature i.e 250K. Akaganeite nanorods were synthesized via forced hydrolysis, HCl was added in the aqueous solution of $FeCl_3$ and heated at 98°C for 24 hrs. Different amount of NH_4OH was added in the solution which acts as a surface modifying agent. Exchange bias effect of different sample with varying amount of NH_4OH was analyzed by VSM and parameter were optimized in order to achieve exchange bias near to room temperature. This increase in exchange bias was correlated with surface roughness and these argument were well supported using TEM, AFM and SEM. Akaganeite nanorods with near room temperature exchange bias properties may find its application in read/write head of HDD, MRAM and magnetic sensors.

Honorary Sympoisum: Advancing Frontiers of Ceramics for Sustainable Society Development - International Symposium in Honor of Dr. Mrityunjay Singh

Advancing Frontiers of Ceramics I -Biotechnologies, etc.

Room: Coquina Salon E

Session Chairs: Sanjay Mathur, University of Cologne; Jerzy Lis, AGH University of Science and Technology

1:30 PM

(ICACC-HON-001-2018) Regenerative Engineering of Bone: Next Generation Inductive Graphene-Ceramics (Invited)

L. Daneshmandi[1]; S. Gohil[3]; L. Nair[3]; A. Arnold[2]; B. Holt[2]; S. Sydlik[2]; C. Laurencin*[1]

1. University of Connecticut, Biomedical Engineering, USA
2. Carnegie Mellon University, Chemistry, USA
3. University of Connecticut Health Center, Orthopedic Surgery, USA

Each year, approximately 2.2 million bone graft procedures are performed worldwide. Current clinical treatments have only shown success for smaller sized bone defects and have significant limitations. Recently Sydlik and her colleagues have developed phosphate functionalized graphene oxide (GO) materials that mimic the chemical and mechanical properties of native osseous tissue. These phosphate graphenes (PG) can covalently incorporate

induceron-like ions such as Ca^{2+}, K^{+}, Li^{+}, Mg^{2+}, or Na^{+} to be released over time and act as signaling molecules for bone regeneration. In this study, we assess the in vivo osteoinductive capabilities of phosphate functionalized graphene with Ca^{2+} as the counter ion (CaPG) following subcutaneous implantation into GFP reporter mice. After 8 weeks and when injected with donor bone marrow stromal cells, CaPG induced ectopic bone formation. Moreover, the histology produced by the combination of GO and Ca^{2+} through CaPG was similar to that of GO delivery with bone morphogenetic proteins. These are the first studies to demonstrate the combination of graphene modified to release Ca^{2+} and PO_4^{3-} ions as a new osteoinductive matrix for bone regeneration. The use of simple signaling molecules in combination with GO presents exciting possibilities for tissue regeneration.

2:00 PM

(ICACC-HON-002-2018) Green Biomaterials: Pioneering Environmentally-Safe Nanophase Materials for Tissue Engineering Applications (Invited)

T. Webster*[1]

1. Northeastern University, USA

With materials such as titanium, stainless steel, nitinol, CoCrMo and others, biomaterials may be one of the last fields to fully embrace sustainability or the use of environmentally-safe materials. Moreover, while nanotechnology, or the use of materials with at least one dimension less than 100 nm, has revolutionized the field of medicine, numerous nanomaterials still employ toxic catalysts or simply are not fabricated using sustainable processes. Bioceramics are an excellent example of the above. Bioceramics have been competently engineered into nanophase particles or scaffolds for the selective transport of remedial agents or the stimulation of cellular adhesion and proliferation. Fundamental design preconditions have postulated the fabrication of pure particulate carriers which possess large surface area-to-volume ratios and that are easily transferable within volumetrically confined regions, or, alternatively, of mechanically robust nanoscaffolds or fibers with surface topographies that promote the rapid induction of bone. Recent research has also found that nanophase ceramics can decrease implant infection without contributing to antibiotic-resistant bacteria. This presentation will cover recent advances in a new field, termed green biomaterials, to highlight efforts for the design and fabrication of nanophase ceramics for a more sustainable environment.

2:30 PM

(ICACC-HON-003-2018) Elements of Medical Product Translation: Importance of Ceramic Components (Invited)

A. J. Coury*[1]

1. Northeastern University, Chemical Engineering, USA

Mineral-based components (ceramics and glasses) comprise essential elements of important implantable medical devices. Electronic cardiovascular and neurological devices and mechanical orthopedic devices constitute significant segments of the implantable device sector. These and all implantable medical devices must conform to certain specifications to achieve regulatory approval. Beyond approval, commercial success mandates the achievement of dozens of "imperatives," which should be considered before beginning a serious product development process. Operationally, a series of translational stages leading to device approval, marketing and post-market surveillance is required and has been validated over two decades. Examples of successful and unsuccessful FDA-approved medical devices (cardiac pacemakers and surgical sealants) illustrate the importance of meeting all of the criteria required for product success.

3:20 PM

(ICACC-HON-004-2018) Life-saving applications of strengthened glass (Invited)

A. K. Varshneya*[1]

1. Saxon Glass Technologies, Inc., USA

Several layers of polymer laminated chemically strengthened glass enable cockpit windshields in commercial aircrafts to safely resist impact of flying birds. Likewise, bullet-resistant glass is often a laminate of thermally or chemically strengthened glass. Over the past decade or so, the introduction of chemical strengthening to a Type I glass cartridge in an autoinjector device, such as an EpiPen, is able to provide safe delivery of emergency antidote epinephrine in true life-threatening anaphylactic shock from extreme allergies to bee-stings, peanuts and shell foods. Newer pharmaceutical applications of strengthened glass which benefit humans at large are on the horizon. Requirements and performance characteristics of glass in several of these examples are briefly presented.

3:50 PM

(ICACC-HON-005-2018) Oxynitride Glasses for Potential Biomedical Usage (Invited)

S. Hampshire*[1]

1. University of Limerick, Materials and Surface Science Institute, Ireland

The low strength of bioglasses have restricted their use to non-load bearing applications and therefore improvements in mechanical properties are needed. One solution to this challenge is to incorporate nitrogen into the glass silicate network. This paper outlines the effect of nitrogen on properties and structure of two series of glasses: (1) a "bioglass" composition (without P_2O_5) in the Na_2O–CaO–SiO_2 system plus Si_3N_4 to give different levels of N and (2) glasses of series (1) with a simple substitution of CaF_2 for CaO. In both cases, the Na:Ca:Si ratio was kept constant so that changes in properties and structure were simply a result of changes in O:N ratio. Properties were all observed to increase linearly with nitrogen content. These increases are consistent with N in the glass structure in 3-fold coordination with silicon and extra cross-linking of the glass network. The combination of both nitrogen and fluorine in series (2) glasses gives better mechanical properties at much lower melting temperatures since fluorine reduces Tm, allows higher solubility of nitrogen and does not affect the higher mechanical properties arising from incorporation of nitrogen. The local structure around silicon and nitrogen was investigated by ^{29}Si MAS-NMR spectroscopy which confirms formation of SiO_3N, SiO_2N_2 tetrahedra with extra bridging anions. Bioactivity was assessed.

4:10 PM

(ICACC-HON-006-2018) Bioinspired materials templates by nature species (Invited)

D. Zhang*[1]; J. Gu[1]; W. Zhang[1]; Q. Liu[1]; S. Zhu[1]; H. Su[1]

1. Shanghai Jiao Tong University, China

Biological materials naturally display an astonishing variety of sophisticated nanostructures that are difficult to obtain even with the most technologically advanced synthetic methodologies. Inspired from nature materials with hierarchical structures, many functional materials are developed based on the templating synthesis method. This review will introduce the way to fabricate novel functional materials based on nature bio-structures with a great diversity of morphologies. We focused on replicating the morphological characteristics and the functionality of a biological species (e.g. butterfly wings). We change their original components into our desired materials with original morphologies faithfully kept. Properties of the obtained materials are studied in details. Based on these results, we discuss the possibility of using these materials in photonic control, solar cells, energy harvesting, and gas sensitive devices, et al. In addition, the fabrication method could be applied to other nature substrate template and inorganic systems that could eventually lead

to the production of optical, magnetic. or electric devices or components as building blocks for nanoelectronic, magnetic, or photonic integrated systems. These bioinspired functional materials with improved performance characteristics are becoming increasing important, which will have great values on the development on structural function materials in the near future.

4:30 PM

(ICACC-HON-007-2018) Ceramics Research Endeavor at the Korea Institute of Ceramic Engineering and Technology (KICET) (Invited)

S. L. Kang*[1]; S. Lee[1]

1. Korea Institute of Ceramic Engineering and Technology (KICET), Republic of Korea

KICET, a semi-governmental institution, dates back to 1912 as a ceramic engineering division in the central testing laboratory of the government. The function of testing and evaluation of ceramic products has been maintained, but a research function was added in 2000. Since then, ceramics research has been the major activity of KICET in addition to industry support, testing and evaluation, and support for the government policy of national research and industry development. The current research activities are based on five themes: Smart for Electronic Ceramics, Green for Energy/Eco Ceramics, Wellness for Nano/Bio Convergence Ceramics, Safety for Engineering Ceramics, and Sensibility for Ceramic Ware. This presentation reports the research highlights and introduces future research directions at KICET.

5:00 PM

(ICACC-HON-008-2018) Modern ceramic education as a response to the needs of economy and industry - from the experience of AGH UST (Invited)

J. Lis*[1]; M. M. Bucko[1]

1. AGH University of Science and Technology, Faculty of Materials Science and Ceramics, Poland

AGH University of Science and Technology is currently the largest research universities in Poland. A wide scope of research and education conducted at AGH UST is a result of over 100 years of history and it was connected with the development of the country's economy. One of the most developed directions are chemical technology and materials science in the field of ceramics conducted by the Faculty of Materials Science and Ceramics. The presentation characterizes of the FMSC AGH UST, the alone faculty in Poland focused on ceramics. In the structure of the Faculty, the unit especially dedicated to research and education in the field of ceramics is the Department of Ceramics and Refractories. Students graduating from the Faculty have strong technological backgrounds associated with the manufacturing of ceramics, glass, building materials and refractories, but also with novel materials for specific applications. FMSC is engaged in extensive international cooperation and has ambitions to be a partner in the world's leading ceramic centers. Very effective and beneficial for the Polish ceramic environment is collaboration with the American Ceramic Society, long-term promoted and developed by Dr. Mrityunjay Singh, especially during Dr. Singh function of the President of AmCerSoc. The examples of this fruitful cooperation and plans for the future are presented and discussed.

5:20 PM

(ICACC-HON-037-2018) Dynamic Inelastic Deformation Mechanisms in Al_2O_3, AlN and AlON (Invited)

J. W. McCauley*[1]

1. Johns Hopkins University/Army Research Laboratory, USA

To systematically develop new or improved materials for use in protection systems a "Materials by Design" approach is being taken. This is a process of designing materials from the atomic to the macroscopic scale for a suite of mechanisms and properties that are required for defined performance/applications. Inelastic deformation mechanisms can dominate in extreme dynamic events. The total mechanical response and energy dissipation is a result of mechanisms controlled by the atomic structures of the phase/s, grain sub-structure, microstructure, grain boundary/inter-phase characteristics and defects at the various scales. Rather than toughness, which is the energy to propagate a single crack, normally in quasi-static conditions, while ignoring any plasticity at the crack tip, the concept of high strain rate "quasi-plasticity" for structural ceramics will be discussed. This can be described as inelastic (non-linear) deformation (sub-macrocrack mechanisms in a volume of material) prior to failure (macro-cracking), at both intragranular and microstructural scales. The talk will describe the apparent mechanisms observed in Al_2O_3, AlN and AlON materials resulting from high strain-rate tests, including nano/micro-deformational twinning, nano/micro-cleavage, dislocations, and grain boundary failure.

7th Global Young Investigator Forum

Frontiers in Ceramic Chemistry and Physics: New Precursors for Functional Ceramics, Ceramics and Catalysis, Functional Surfaces

Room: Coquina Salon G

Session Chair: Manoj Mahapatra, University of Alabama at Birmingham

1:30 PM

(ICACC-GYIF-001-2018) Electrospun Metal Oxide Fiber Meshes for Improved Sensing of Toxic Analytes in the Gas Phase (Invited)

T. Fischer*[1]; D. Graf[1]; A. Lepcha[1]; Y. Gönüllü[1]; S. Mathur[1]

1. University of Cologne, Institute of Inorganic Chemistry, Germany

Gas sensors need to combine a high Sensitivity and analyte Selectivity with acceptable readout Speed and long term Stability by not sacrificing the fabrication Scalability and System Integration capability, when it comes to commercialization. Metal oxide semiconductors are applied as chemirosistive gas sensing materials, but lack a specific target selectivity especially in humid environments as well as exhibit rather slow sensing kinetics when used as pure unmodified material. Resistive metal oxide gas sensors with nanowire based sensing materials offer high surface areas and defined signaling pathways for improved device performance thus fulfilling most of the aforementioned criteria, but most often lack the potential of large scale integration, due to complex fabrication techniques. In contrast, electrospun metal oxide fibers and fiber meshes can be fabricated in comparable large amounts at ambient conditions, thus providing ideal materials for either the active sensing layer or necessary preconcentrators and filters, respectively. A direct integration onto multifunctional gas sensing platforms is also possible in modified electropinning setups, thus providing inteconnected sensing meshes with high surface areas.

2:00 PM

(ICACC-GYIF-002-2018) Formation mechanism of critical microstructures in ceramics by infrared spectroscopy

C. A. Lee*[2]; j. Ren[1]; K. Chen[1]; M. Li[1]; Y. Chen[1]

1. The Chinese University of Hong Kong, Energy and Catalysis Laboratory, Department of Mechanical and Automation Engineering, China
2. Energy and Catalysis Laboratory, Department of Mechanical and Automation Engineering/The Shun Hing Institute of Advanced Engineering, The Chinese University of Hong Kong, China

Ceramics and catalysis are widely used in energy and environmental applications. Their functionality, performance and reliablility are closely related to their structure and defects, which are generally

controlled by processing parameters and heat treatments. Several techniques have often been applied to investigate thermal behaviors and chemical reactions in ceramics, including thermal gravity analysis, differential thermal analysis, dilatometer, etc. We are developing an analytical technique using in-situ infrared spectroscopy to monitor gas evolution kinetics associated with ceramic processing and applications. This information sheds a new light on the microscopic structures of ceramics such as defects that are critical to their performance.

2:20 PM

(ICACC-GYIF-003-2018) Magnetic textures in the ferromagnetic insulating phase of $La_{0.875}Sr_{0.125}MnO_3$

A. Kotani*[1]; H. Nakajima[2]; K. Harada[3]; Y. Ishii[1]; S. Mori[1]

1. Osaka Prefecture University, Materials Science, Japan
2. Kyushu University, Applied Quantum Physics and Nuclear Engineering, Japan
3. the Institute of Physical and Chemical Research, Center for Emergent Matter Science, Japan

Intricate magnetic domain patterns, called magnetic textures, have been expected to be useful for development of new spin devices. In particular, magnetic vortices such as magnetic skyrmions are formed in some helical magnets (MnSi, FeGe) without the inversion symmetry, which has been revealed by neutron scattering experiment and Lorentz microscopy (LM) observation. In present work, we have investigated evolution of magnetic textures by applying external magnetic fields in $La_{1-x}Sr_xMnO_3$ with the centrosymmetric crystal structure, using LM and small angle electron diffraction (SmAED). It is revealed that in the ferromagnetic (FM) phase of $La_{0.875}Sr_{0.125}MnO_3$ magnetic stripes at zero magnetic field evolved to magnetic bubbles by applying magnetic fields along the magnetic easy axis. The magnetic bubbles were formed in the FM insulating phase, due to the charge and orbital ordering, which originate from magnetic dipole-dipole interactions and the high magnetocrystalline anisotropy. From our experiments, magnetic phase diagrams of $La_{0.875}Sr_{0.125}MnO_3$ as functions of magnetic field and temperature were constructed.

Applications: Ceramic Sensors and Actuators, Energy Generation, Saving and Storage, Photo-catalysis and Biomedical Applications

Room: Coquina Salon G

Session Chairs: Giorgia Franchin, University of Padova; Daniele Benetti, Institut National de la Recherche Scientifique

3:20 PM

(ICACC-GYIF-004-2018) Graphite – The Critical Carbon (Invited)

R. M. Paul*[1]

1. GrafTech International Holdings Inc., Technology, USA

Graphite is one of the allotropes of carbon. However, the term 'graphite material' is also used to refer to a class of engineered materials that have been in use going back hundreds of years. Today, graphite materials represent a $14 billion global industry, with over 3 million tons produced every year, growing at a rate of about 4%. As engineered materials, natural and synthetic graphite materials are made in various forms and are used as critical components in a variety of products, including carbon fiber composites, nuclear moderators, lithium-ion battery anode powders, graphite electrodes, and flexible graphite heat spreaders. This talk will highlight the fundamentals of natural and synthetic graphite, including the processing, structure, and properties that enable graphite materials to play a critical role in today's products and applications. Natural graphite is even considered a critical material, as its mining is concentrated to a few key countries outside the United States. The limitations of graphite will also be highlighted, for example, the oxidation-induced degradation of nuclear-grade graphite used as a neutron moderator. This talk will summarize the current state-of-the-art of natural and synthetic graphite and offer comments on the future of a material that is one of the most critical forms of carbon.

3:50 PM

(ICACC-GYIF-005-2018) Additive manufacturing of TiC porous targets for nuclear physics applications in radiopharmacy

G. Franchin*[1]; A. Zanini[1]; A. Girotto[1]; S. Corradetti[2]; A. Andrighetto[2]; P. Colombo[1]

1. University of Padova, Industrial Engineering, Italy
2. Istituto Nazionale di Fisica Nucleare, Laboratori Nazionali di Legnaro, Italy

The SPES (Selective Production of Exotic Species) project is devoted to basic and applied research in nuclear physics and astrophysics involving among all the generation of neutrons for material studies and of radionuclides of medical interest. It provides the construction of a ISOL (Isotope Separation On-Line) facility for producing neutron-rich ions by collision of protons onto a target. In the case of the ISOLPHARM project, such radioisotopes will be studied for the production of radiopharmaceutical for diagnostics and/or treatment. The target is designed for producing specific radioisotopes: here, we use TiC as target material for the production of Sc radioisotopes. Previous studies show that residual porosity derived from incomplete sintering increases the release efficiency of the targets; structures with interconnected pores are desired to minimize impacts between target and isotopes and to allow for effusion. We fabricated TiC lattice targets via DIW (Direct Ink Writing) of an aqueous suspension with optimized rheology and we selected a sintering treatment able to consolidate the structures without promoting grain growth. We designed and produced geometries with similar total porosity but different stacking of layers and measured their permeability and emissivity. The results will lead to the selection of an target geometry which maximizes the system performances.

4:10 PM

(ICACC-GYIF-006-2018) Reaction-bonded silicon carbide for nuclear fusion

A. J. Leide*[1]; R. I. Todd[1]; S. G. Roberts[1]; K. Yoshida[2]; T. Yano[2]; M. Gorley[3]; D. E. Armstrong[1]

1. University of Oxford, Department of Materials, United Kingdom
2. Tokyo Institute of Technology, Laboratory for Advanced Nuclear Energy, Institute of Innovative Research, Japan
3. Culham Centre for Fusion Energy, United Kingdom

Silicon carbide has potential applications in the blanket of fusion reactors, where its chemical inertness, low neutron cross-section, and radiation tolerance are especially useful. However, manufacturing large, complicated blanket components for a fusion reactor using "nuclear grade" SiC composites is currently very challenging. Large components can be made reliably to near net shape at an economical price using reaction-bonded silicon carbide (RB-SiC). In unirradiated form, the properties are suitable for fusion applications, however, early irradiation work on commercial grades of RB-SiC suggested that radiation-induced swelling in SiC and residual silicon caused internal stresses leading to failure. In an attempt to solve this, SiC-$CrSi_2$ and SiC-WSi_2-Si composites with fine grain structures have been made by the reaction-bonding process, and have been investigated using micromechanical techniques, SEM, and Raman microscopy. Ion implantation is used to investigate the changes to these materials under irradiation. This presentation will begin with a description of nuclear fusion, radiation damage and the unique challenges of this environment, followed by processing of these novel ceramics, and their properties pre-and post-irradiation. The talk will end with ideas for future work and potential collaborations, along with comments on working in an interdisciplinary field of materials scientists, physicists, and engineers.

4:30 PM

(ICACC-GYIF-007-2018) Evaluation of power generation from biomass using Solid Oxide Fuel Cell (SOFC) and downdraft gasifier

S. Yamaguchi*[1]; K. Katagiri[1]; T. Ozaki[1]; T. Ehiro[1]; A. Kakitsuji[1]

1. Osaka Research Institute of Industrial Science and Technology Izumi Center, Research Division of Applied Material Chemistry, Japan

Performances of power generation system from biomass composed of downdraft gasifier and SOFC were evaluated. For experimental analysis of downdraft gasifiers, a small sized gasifier (0.1 kW, with composite catalysts, using external heater) and big sized gasifier (20 kW, without catalysts, self-combusting) were examined. The gasified gases from each downdraft gasifier were analyzed by numerical simulation model based on thermodynamics. By analyzing the temperature distribution in the big sized gasifier, the compositions of gasified gases by experiments were in good agreement with those predicted by the numerical simulation. The cold gas efficiencies converting biomass to gasified gas by the big sized gasifier operating at steady state were also evaluated. To analyze the characteristics of gasification and power generation, gasification with different conditions of air, steam flow rate by small sized gasifier using composite catalysts were experimented and analyzed by the numerical simulation. By stabilizing the flow rate of gasified gases from the small sized gasifier to SOFC, the current-voltage (I-V) characteristics of SOFC fed by each gasified gas were examined stably. By analyzing the I-V characteristics by the gasification and power generation using downdraft gasifier and SOFC, total thermal efficiency from biomass to electricity were evaluated.

4:50 PM

(ICACC-GYIF-008-2018) Performance enhancement of medium-temperature anhydrous fuel cells by incorporation of proton conductive material to the three-phase interface

K. Maegawa*[1]; K. Ya[1]; G. Kawamura[1]; T. Hattori[1]; H. Muto[2]; A. Matsuda[1]

1. Toyohashi University of Technology, Electrical and Electronic Information Engineering, Japan
2. Toyohashi University of Technology, Institute of Liberal Arts and Sciences, Japan

Development of medium-temperature anhydrous fuel cell, which is one type of Polymer Electrolyte Fuel Cell (PEFC) is becoming popular recently. By utilizing ionic conduction in this system, the usage of expensive platinum could be reduced in order minimize operational cost. To obtain good fuel cell performance, proton conductive material which is called CHS-WSiA ($CsHSO_4$-$H_4SiW_{12}O_{40}$) that exhibit high proton conductivity at the temperature of 150°C in anhydrous condition was fabricated. Excellent power density enhancement was achieved when CHS-WSiA was added into the electrolyte. In this work, three-phase interface which consists of the electrode, catalyst, and electrolyte is being investigated. The structure of the interface influences the transport mechanism of electron and ion especially. In order to enhance the interfacial ion conductivity as well as suppressing the overvoltage phenomenon CHS-WSiA fabricated by mechanical milling was applied at the cathode side of catalyst. As a result, the interface resistance and overvoltage between the catalyst and the electrolyte were reduced and the maximum power density was enhanced. The results obtained clearly that characteristics improvement of the interface in the medium-temperature anhydrous fuel cell can be achieved by applying mechanically milled CHS-WSiA to the catalyst layer.

5:10 PM

(ICACC-GYIF-009-2018) Carbon Dots and their application in energy harvesting devices

D. Benetti*[1]; Y. Zhou[1]; H. Zhao[1]; A. Vomiero[2]; F. Rosei[1]

1. Institut National de la Recherche Scientifique, Materials, Energy and Telecommunication, Canada
2. Lulea University of Technology, Engineering Sciences & Mathematics, Sweden

Carbon dots (C-dots) are composed of non-toxic elements (mainly C, N and O) and can be synthesized in large quantities via a simple solvothermal approach. Compared to traditional semiconducting quantum dots, such as CdSe or PbS, they are more environmental friendly, and easier and cheaper to produce. By mean of doping procedures, it is possible to tune their optical and electrical properties. Exploiting this ability, we produced different kind of Cdots, tuning their emission from 365nm up to 630nm. The as prepared Cdots were then load on wide bandgap semiconductor materials (MOS), such as TiO2, and characterized by Time-Resolved Photoluminescence Spectroscopy in order to verify the ability fo Cdots to inject electron in the conduction band of the MOS. Thanks to their properties, the Cdots were then employed for realizing different energy harvesting devices.

Tuesday, January 23, 2018

S1: Mechanical Behavior and Performance of Ceramics & Composites

Strength and Fracture Toughness of Monolithics

Room: Coquina Salon D

Session Chairs: Jonathan Salem, NASA Glenn Research Center; Raul Bermejo, Montanuniversitaet Leoben

8:30 AM

(ICACC-S1-011-2018) Fracture Toughness of Modern and Ancient Glasses and Glass Ceramics as Measured by the SEPB Method

G. D. Quinn*[1]; J. Swab[3]; R. Brill[2]; S. Koob[2]

1. National Institute of Standards and Technology, Materials Measurement Sciences Division, USA
2. Corning Museum of Glass, USA
3. U.S. Army Research Laboratory, USA

The fracture toughness of two modern glasses and two transparent glass ceramics was measured by the single-edged precracked beam (SEPB) method. This work is the continuation of our studies to characterize the mechanical properties of transparent armor grade materials. This year we tested two new commercial, low-iron soda lime silicas (St. Gobain Diamant and Guardian UltraWhite) and two commercial transparent glass ceramics (Schott Zerodur and Robax) with nanometer scale crystallites. Results are compared to our earlier data on six other glasses. A soda lime silica made in antiquity (circa 381 C.E.) was also evaluated. This glass was recovered in 1965 from an archeological excavation of a Roman empire era glass factory located near Mt. Carmel in modern day Jalame, Israel. This location is very near the site described by Pliny the Elder in Volume36 of his first century book series Natural History. This glass was exposed to the earth and elements for over 16 centuries.

8:50 AM

(ICACC-S1-012-2018) Observations in Fracture Toughness Testing of Glasses and Optical Ceramics

J. Salem*[1]

1. NASA Glenn Research Center, Materials and Structures, USA

Fracture toughness is a critical structural design parameter and an excellent metric to rank materials. It determines fracture strength by way of the flaws, both inherent and induced, and defines the endpoint of the slow crack growth curve. The fracture toughness of structural and optical ceramics, and glasses as measured by several techniques is compared. When good metrology is employed, the results are very comparable with two exceptions: materials exhibiting crack growth resistance and those with a low SCG exponents. For materials with R-curves, the result is a function of extension and can be minimized with short cracks. For materials with low SCG exponents, such as glasses, elimination of the corrosive media and/or increasing the stress intensity rate minimizes effects. A summary of values is given, and it appears that highly modified glasses exhibit lower fracture toughness and slow crack growth exponent than high purity glasses such as fused silica.

9:10 AM

(ICACC-S1-013-2018) Toward seashells under stress: Novel concepts to design tough layered ceramic composites

R. Bermejo*[1]; Y. Chang[2]; G. L. Messing[2]

1. Montanuniversitaet Leoben, Institut fuer Struktur- und Funktionskeramik, Austria
2. Pennsylvania State University, Materials Science and Engineering, USA

The design of "bio-inspired" layered ceramic composites with weak or strong interfaces can yield significant increase in the failure resistance of the material and, in some cases, hinder, or even arrest, the propagation of cracks. In this work, novel concepts are presented which combine different approaches used in layered architectural design to obtain highly reliable ceramic materials with enhanced fracture resistance. The use of tailored residual stresses in embedded layers within the structure is demonstrated to act as an effective barrier to the propagation of surface flaws, providing the material with a minimum design strength, below which no failure occurs. Moreover, texturing of the microstructure in embedded layers is utilized to provide preferential paths for conducting propagating cracks, thus protecting the underlying structure. A combination of experiments and modelling is presented, showing the potential of layered architectures in the design of future ceramic components with spatially resolved strength and toughness.

9:30 AM

(ICACC-S1-014-2018) Effect of Control Mode and Load Rate on Fracture Toughness

J. Salem[1]; B. Hausmann*[2]

1. NASA Glenn Research Center, Materials and Structures, USA
2. Case Western Reserve University, Materials Science, USA

The effects of control mode and rate on the fracture toughness of ceramics were measured by using chevron-notched flexure specimen in accordance with ASTM C1421. The use of stroke control gave the most consistent results with less than 2% variation in measured fracture toughness for a very wide range of rates (0.005 to 0.5 mm/min). Use of strain or CMOD control gave ~5% variation over a very wide range of rates, with the measurements being a function of rate. However, the effect was eliminated by use of dry nitrogen, implying a stress corrosion effect rather than a stability effect. With the use of nitrogen, both control modes were within 1% over a wide range of rates (40 to 60 με/s and 0.005 to 0.2 mm/min). COMD or strain control did allow stable crack extension well past maximum load, and thus is better for energy calculations.

10:10 AM

(ICACC-S1-015-2018) Crack-Path Dependent Fracture Toughness in Additively Manufactured Ceramic Composites with Anisotropic Heterogeneities

N. R. Brodnik*[1]; C. Hsueh[3]; S. Biesboer[2]; T. Schaedler[2]; Z. C. Eckel[2]; G. Ravichandran[3]; K. Bhattacharya[3]; K. Faber[1]

1. California Institute of Technology, Materials Science, USA
2. HRL LLC, USA
3. California Institute of Technology, Mechanical Engineering, USA

Ceramics have long been of interest as engineering materials because of their high stiffness and good thermal and chemical stability. However, their efficacy across different applications has historically been limited by their low fracture toughness. To better understand the fracture of brittle ceramics, we have developed additively manufactured composites that use precisely positioned anisotropic heterogeneities to increase fracture toughness and induce a dependence on the direction of crack growth. These composites are produced using stereolithography, which allows for precise control of both the shape and position of inclusions. Specimens are designed as thin structures whose heterogeneities are uniformly spaced parallel channels with asymmetrical cross-sectional areas. Thin composites allow for study of systems in a two-dimensional stress state, and directional alignment of channels produces fracture toughness values that vary based on orientation with respect to the direction of crack propagation. Methods for evaluation of fracture toughness have been established using simulation as well as experiment on both model brittle photopolymer and silicon oxycarbide produced from printable preceramic polymers. Based on these evaluations, increases in toughness of up to 50% can be achieved for a given composite compared to its homogeneous matrix material.

10:30 AM

(ICACC-S1-016-2018) Overcoming challenges and obstacles in measurement of fracture toughness of plasma sprayed ceramics

G. Smith*[1]; S. Sampath[1]

1. Stony Brook University, Center for Thermal Spray Research, USA

Spray formed structures, due to their layered build up, comprise of multiscale defects (pores/cracks) and anisotropy. These affect various mechanical and thermal properties such as elastic modulus, toughness, conductivity, etc. Since these defects govern much of the thermo-mechanical response, it is of importance to accurately characterize and consider orientation dependence. Most coatings are considered in-plane orthotropic, with different through thickness properties due to the lamellar buildup of the coating. Due to thickness limitations and constraints associated with substrate bound coatings, it is difficult to rigorously characterize the mechanical behavior in multiple orientations. Past work with atypically thick, free-standing coating specimens in near bulk form allowed direct property measurement in multiple orientations, however this presents fabrication challenges. Alternatively, traditional thin coats can be examined with a combination of less conventional mechanical methods presented here to extrapolate fracture toughness, orientation dependence, and anisotropy ratios. In many cases, these measurement values can be corroborated with those from the bulk thick sample. Results point to a 15-30% difference in properties as a function of orientation.

10:50 AM

(ICACC-S1-017-2018) Mechanical behavior of single domain polycrystalline and single crystal ferroelastic ceramics

C. S. Smith*[1]; J. A. Krogstad[1]

1. University of Illinois at Urbana-Champaign, Materials Science and Engineering, USA

Ferroelastic deformation allows for increased toughness in a variety of electroceramic and structural ceramic materials through the nucleation and motion of ferroelastic domains. Unlike other toughening mechanisms, ferroelastic toughening occurs without sacrificing the microstructural and phase homogeneity of the ceramic. In this study, the mechanism of ferroelastic deformation and its behavior in real ceramic microstructures has been examined. Mechanical tests have been performed on ferroelastic ceramics over a range of length scales. Due to the high mechanical anisotropy of ferroelastic crystals, careful tracking of individual crystal orientations has been done using EBSD. Microindentation has been used to induce ferroelastic deformation in bulk polycrystals, while deformations of single crystals have been studied using in situ transmission electron microscopy. Using these methods, the coercive stress in single crystal as well as polycrystalline ferroelastic ceramics has been exceeded without nucleation of multiple domains. Furthermore, for single domain polycrystals, a lack of permanent domain nucleation is observed in absence of associated material cracking. These results have implications for the designing of microstructures to utilize ferroelastic toughening.

11:10 AM

(ICACC-S1-018-2018) Orientation dependent fracture behaviour of $LiTaO_3$ and $LiNbO_3$ brittle single crystals and its atomistic origin

M. Gruber[1]; M. Popov[3]; P. Supancic[1]; D. Kiener[2]; R. Bermejo*[1]

1. Montanuniversitaet Leoben, Institut fuer Struktur- und Funktionskeramik, Austria
2. Montanuniversitaet Leobe, Material Physics, Austria
3. Materials Center Leoben, Austria

Single crystal piezoelectric materials such as $LiTaO_3$ and $LiNbO_3$ have found important application as surface acoustic wave filter substrates for high frequency data transfer. In order to optimize the functionality of the filters, a particular orientation and surface conditioning (e.g. grinding) of the crystal must be ensured. This can affect the mechnical response of the functional material to external thermo-mechanical loading. In this work, the strength and crack growth resistance (fracture toughness) of $LiTaO_3$ and $LiNbO_3$ materials were investigated macroscopically using biaxial bending and microscopically by miniaturized V-notched cantilevers in a SEM, respectively. It was found that the orientation of the notch, either parallel to the corresponding cleavage plane or with an angle, yields to a significant difference in the toughness values. As a consequence, the orientation of the grinding directions during flexural bending with respect to the cleavage planes showed a high impact on the strength for both materials.

11:30 AM

(ICACC-S1-019-2018) In Situ Fracture Tests of Ceramic Grain Boundaries

F. Giuliani*[1]; G. Sernicola[1]

1. Imperial College London, United Kingdom

The fracture toughness of ceramics is often dominated by the structure of their grain boundaries. Our capacity to improve the performance of ceramic components depends on our ability to investigate the properties of individual grain boundaries. This requires development of new fracture testing methods providing high accuracy and high spatial resolution. Recently, several techniques have been developed using small scaled mechanical testing, based within a nanoindenter, using a variety of sample geometries. However, the majority of the published work relies on load-displacement curves for the identification of crack initiation and can result in a complex analysis. Our approach uses a double cantilever geometry to obtain stable crack growth under a constant wedging displacement. The tests are carried out within an SEM, this has two benefits: the sample is well aligned for a controlled test and images are recorded during the test for later analysis. Crucially this allows us to use beam deflection and crack length rather than critical load to measure fracture toughness. Our tests have proved it is possible to initiate and stably grow a crack in a controlled manner in ceramic materials for several microns. This approach has been validated on SiC where it gives a good approximation of the surface energy and then extended to SiC bi-crystals along with Ni-Al_2O_3 interfaces where crack blunting and bridging mechanism can be measured.

S2: Advanced Ceramic Coatings for Structural, Environmental, and Functional Applications

Environmental Barrier Coatings - Fundamentals

Room: St. John

Session Chair: Dongming Zhu, NASA Glenn Research

8:30 AM

(ICACC-S2-012-2018) Effect of Electrical Characteristics on Oxygen Shielding Properties and Structural Stability of Bilayer Oxide Films

S. Kitaoka*[1]; T. Matsudaira[1]; M. Tanaka[1]; T. Sato[2]; O. Sakurada[2]; Y. Kagawa[3]

1. Japan Fine Ceramics Center, Japan
2. Gifu University, Japan
3. Tokyo University of Technology, Japan

High-temperature mass-transfer mechanisms in oxide films were investigated by evaluation of oxygen permeability through the films, which served as models for anti-oxidation films. When an alumina film was subjected to an oxygen potential gradient ($d\mu_O$), the electronic transference number for the film increased, resulting in the development of a negative charge on the high oxygen partial pressure (P_{O2}(hi)) surface, and a positive charge on the opposing low oxygen partial pressure (P_{O2}(lo)) surface. The grain boundary (GB) diffusion coefficient for oxygen ions under a $d\mu_O$ was significantly smaller than that in the absence of a $d\mu_O$. Space charges which are opposite to the charges of the P_{O2}(hi) and P_{O2}(lo) surfaces are likely to spontaneously develop beneath the surfaces, resulting in the formation of electrical double layers. In contrast to this situation, a mullite film exhibited ionic conductivity and its oxygen-GB diffusion coefficient was independent of the presence or absence of a $d\mu_O$. The resulted in the polarity of its electrical double layers being opposite to that for alumina. The use of a bilayer film consisting of oxides with markedly different electrical characteristics, such as alumina and mullite, significantly affected the oxygen shielding properties and structural stability of the entire film.

8:50 AM

(ICACC-S2-013-2018) Effect of water vapor on mass transfer in polycrystalline $Yb_2Si_2O_7$ under oxygen potential gradients at high temperatures

M. Wada*[1]; T. Matsudaira[1]; N. Kawashima[1]; D. Yokoe[1]; T. Kato[1]; S. Kitaoka[1]; M. Takata[1]; M. Takeuchi[2]

1. Japan Fine Ceramics Center, Japan
2. University of Tokyo, Japan

We attempted to elucidate the mass transfer mechanism in a polycrystalline $Yb_2Si_2O_7$ film under wet conditions at high temperatures, which served as a model for an environmental barrier coating layer, based on oxygen permeation into the film. The oxygen permeation under a large oxygen potential gradient ($d\mu_O$) and dry conditions

is controlled by the interdiffusion of Yb and oxide ions along the grain boundaries (GBs), which results in the formation of Yb_2SiO_5 at the high oxygen partial pressure (P_{O2}) surface and the decomposition of $Yb_2Si_2O_7$ at the low P_{O2} surface. The presence of water vapor with the application of a $d\mu_O$ clearly accelerated the GB diffusivity of Yb ions, resulting in the formation of a thick Yb_2SiO_5 layer at the high P_{O2} surface and the segregation of Yb_2SiO_5 particles inside the film. Nevertheless, the oxygen permeability was below the detection limit. This is thought to be due to the consumption of ambient O_2 molecules in the vicinity of the high P_{O2} surface, which are associated with the formation of Yb_2SiO_5 through the reaction of $Yb_2Si_2O_7$ with water vapor and O_2.

9:10 AM

(ICACC-S2-014-2018) Local Bonding as a Dominant Factor Governing Thermal Expansion of High Temperature Ceramic Materials

M. Yoshiya*[1]; Y. Akada[1]; Y. Sumi[1]

1. Osaka University, Department of Adaptive Machine Systems, Japan

Thermal expansion of crystal lattice is inevitable at elevated temperature and it is often out of the scope upon pursuing target properties of thermal barrier coatings (TBC) or environmental barrier coatings (EBC). Differences in coefficients of thermal expansion (CTEs) in multi-layered TBC/EBC occasionally leads to spallation or fracture upon thermal cycle. Thus, from the perspective of lifetime of TBC/EBC, CTE needs to be controlled to minimize strain energy stored upon the thermal cycle. While CTEs of materials having simple crystal structures including pure metals are correlated to their melting point, all the differences in CTEs of high temperature are often attributed mainly to Grüneisen parameter, impeding to control CTEs for longer lifetime without deteriorating other target properties of TBC/EBC. In this study, we perform ab initio lattice dynamics within quasi-harmonic approximation to systematically evaluate CTEs. It is found that stable atomic positions at elevated temperature that modifies local interatomic bonding are different from those at room temperature and thus taking those into account is crucial for understanding the mechanism governing thermal expansion.

9:30 AM

(ICACC-S2-015-2018) Calorimetric Studies of Refractory Oxides at High Temperature (Invited)

A. Navrotsky*[2]; S. Ushakov[1]; D. Kapush[1]

1. University of California, Davis, Peter A. Rock Thermolab and NEAT ORU, USA
2. University of California, Davis, Peter A. Rock Thermochemistry Laboratory, USA

Thermodynamic properties of solid and liquid refractory oxides, relevant to thermal barrier coatings, nuclear energy, and general phase diagram modeling, are poorly known, especially above 1500 oC. Using a variety of newly developed or optimized techniques, we have developed direct measurements of heats of phase transition and fusion of oxides. Commercial ultra-high temperature differential thermal analyzers (DTA) allow investigation of phase transformations and melting in inert environment to the temperatures up to 2500 °C; Combination of laser heating and a splittable nozzle aerodynamic levitator allows splat quenching and drop calorimetry from temperatures limited only by sample evaporation; iii) Synchrotron X-ray and neutron diffraction on laser heated aerodynamically levitated oxide samples allow in situ observation of phase transformations in variable atmosphere, refinement of high temperature structures and thermal expansion. These methods provide temperatures, enthalpies and volume change on phase transformations above 2000 °C which are complementary experimental data for optimization of CalPhaD databases. New data for rare earth oxides are presented as examples.

Environmental Barrier Coatings - Processing & Properties II

Room: St. John
Session Chair: Kang Lee

10:20 AM

(ICACC-S2-016-2018) Weibull-Based Stochastic Simulation of Mud-Crack Damage Formation in an Environmental Barrier Coating

N. Nemeth*[1]; S. Mital[3]; P. L. Murthy[1]; B. A. Bednarcyk[1]; E. J. Pineda[1]; D. Zhu[1]; H. Wadley[2]; S. M. Arnold[1]

1. NASA Glenn Research Center, USA
2. University of Virginia, USA
3. University of Toledo, USA

The integrated Finite Element Analysis–Micromechanics Analysis Code/Ceramics Analysis and Reliability Evaluation of Structures (FEAMAC/CARES) program was used to simulate the formation of mudflat-cracks from thermomechanical loading on a multi-layered Environmental Barrier Coating (EBC) system deposited on a ceramic substrate. FEAMAC/CARES combines MAC/GMC multiscale composite micromechanics code with CARES/Life probabilistic multiaxial failure criteria code and Abaqus finite element analysis. In this work, step function elastic modulus reduction of randomly damaged finite elements was used to represent discrete cracking events. The use of many small-sized low-aspect-ratio finite elements enabled the depiction of crack boundaries and formation of mudflat patterned damage. Demonstrated examples include finite element models of button–sized square– and disk–shaped 3-D specimens, and a 2-D model of through-the-thickness cross-section. All models were subjected to a progressive cool down from 1300° C to room temperature. Mudflat crack damage in the coating system resulted from the buildup of residual tensile stresses between the individual material constituents from thermal expansion mismatch. A 2-parameter Weibull distribution characterized the coating layer stochastic strength response and the effect of the Weibull modulus on the formation of damage was studied here.

10:40 AM

(ICACC-S2-017-2018) Yb silicate anti-oxidation fiber/matrix interface coating for SiC/SiC for higher temperature durability

K. Goto[1]; A. Ito[2]; M. Sekiyama[4]; T. Matsuda[3]; S. Takahashi[3]; S. Kitaoka[3]; T. Goto*[4]

1. Japan Aerospace Exploration Agency, Institute of Space and Astronautical Science, Japan
2. Yokohama National University, Japan
3. Japan Fine Ceramics Center, Japan
4. Tohoku University, Japan

Yb silicate (YbSiO5 and/or Yb2Si2O7) was examined as Interface coating for SiC fiber reinforced SiC composite to have superior anti-oxidation and water vapor. Two coating method were conducted to make coating onto the SiC fibers, sol-gel process and chemical vapor deposition. Yb2Si2O7 was selected as targeted coating with its higher resistant property for high temperature water vapor oxidation. Mechanical properties of coated fibers were examined by tensile test of a fiber bundle mini-composite. Yb2Si2O7 coating were successfully fabricated through both coating method, sol-gel process and CVD. However, tensile strength of a mini-composite made by sol-gel process and CVD became half of the tensile strength of a mini composite without coating. Tensile strength of a mini-composite without coating reflects tensile strength of SiC fiber. Hence the strength of SiC fiber degraded by coating, because the coating affected as a brittle defect on SiC fibers. Coating was improved to possess weak bonding between fiber and coating and finally the properly controlled interface coating were realized

and tensile strength of mini-composite maintained original fiber strength. Finally, the coating performance was proved by SiC/SiC bundle mini-composite fabricated by polymer infiltration and pyrolysis.

11:00 AM

(ICACC-S2-018-2018) Ultrathin Ceramic Coatings to Stabilize SiC Against Steam Oxidation

A. Hoskins*[1]; A. Coffey[1]; C. B. Musgrave[1]; A. W. Weimer[1]

1. University of Colorado Boulder, Chemical and Biological Engineering, USA

Silicon Carbide (SiC) is an ideal material for many high-temperature applications due to its resistance to thermal shock and high thermal conductivity. However, SiC degrades in water-rich environments limiting its applications in extreme oxidative environments such as combustion engines, heat exchangers, and high temperature reactor materials. Atomic layer deposition (ALD) generates nanoscale films that are conformal, crack-free, and chemically bonded to the substrate surface. We have previously found that the application of mullite nanostructured films grown with ALD significantly improves the oxidation resistance of SiC in extreme environments with a film orders of magnitude thinner than standard applications. In addition to mullite, we have targeted a variety of high temperature coating materials chosen based on thermal properties, structural characteristics, and stability in oxidative environments. Using thermogravimetric analysis, it has been shown that these coatings improve the oxidation resistance of SiC by up to 64% at 1000°C. We have also developed a computation materials screening method implementing density functional theory (DFT) to model the diffusional and thermal properties of candidate coating materials. Computational modeling of the chosen coating materials can help to identify promising coatings to further extend the lifetime of SiC, as well as engineered application specific coatings.

11:20 AM

(ICACC-S2-019-2018) Performance and Durability of Advanced Environmental Barrier Coating Systems

D. Zhu*[1]; B. J. Harder[1]; G. Costa[1]; V. L. Wiesner[1]; K. Lee[1]; B. Puleo[1]; J. B. Hurst[1]

1. NASA Glenn Research, Materials and Structures Division, USA

This paper summarizes recent NASA environmental barrier coating (EBC) material advances for protecting the SiC/SiC Ceramic Matrix Composites for meeting next generation turbine engine performance requirements. We particularly present an advanced multicomponent rare earth silicate EBCs with HfO_2-Si bond coat system, the temperature capability, environmental stability, and thermomechanical durability, advanced laboratory testing toward subelement demonstrations. Fundamental coating properties of the coating system will also be discussed, with the emphasis on the coating diffusion barrier performance and Calcium Magnesium Alumino-Silicate (CMAS) resistance.

11:40 AM

(ICACC-S2-020-2018) Damage Evolution of Environmental Barrier Coatings under Mechanical Loading Condition (Invited)

Y. Kagawa*[1]; Y. Aoki[1]; Y. Arai[1]; H. Hatta[1]

1. Tokyo University of Technology, The Center for Ceramic Matrix Composites, Japan

The effects of external mechanical loading on the damage evolution of a typical oxide/Si bond coat/(SiC/SiC) environmental barrier coatings have been investigated. The influence of local high stress/strain concentration and local crack evolution of oxide layer on the damage evolution have been elucidated through experimental approaches. To understand the effects clearly, model experiments coupled with finite element simulations are carried out. Brittle polymer/metal model materials are used for the experiments. Discussions are made on the correlation between unique deformation and fracture behavior of environmental barrier coating layer on SiC/SiC substrate.

S3: 15th International Symposium on Solid Oxide Fuel Cells (SOFC): Materials, Science and Technology

Electrolytes, Oxygen Ion, Proton and Mixed Conductors, Conduction Mechanisms

Room: Crystal
Session Chair: Scott Barnett, Northwestern Univ

8:30 AM

(ICACC-S3-010-2018) Proton Conductive Multilayers: manufacturing key issues (Invited)

A. Sanson*[1]; E. Mercadelli[1]; A. Gondolini[1]

1. CNR-ISTEC, Italy

Perovskites of general formula ABO_3 ($BaCeO_3$, $SrCeO_3$, $BaZrO_3$ and $SrZrO_3$), have received increasing interest as high temperature proton conductors for hydrogen separation and as electrolytes in intermediate-temperature solid-oxide fuel cells and/or electrolysers (SOFCs/SOECs). These materials however still require ad-hoc investigations from the ceramic-technology point-of-view to boost their widespread deployment in the abovementioned applications. This work will highlight and examine two of the main issues linked to the production of the abovementioned devices: production of planar defect-free multilayers and retention of stoichiometry. In particular, the role of the lamination step on the production scheme will be studied using an alternative characterisation technique. The results will show a new way to properly design and set the processing variables in order to achieve an high performance proton conductive device.

9:00 AM

(ICACC-S3-011-2018) Electrolyte Film Deposited on Porous Anode by Ultrasonic Spray Pyrolysis and Microwave Energy Modification for Anode-supported SOFC

F. Ko*[1]; H. Chang[1]

1. National Taiwan Ocean University, Department of Marine Engineering, Taiwan

The electrolyte thickness reduction and the triple phase boundaries (TPBs) extension into electrodes can reduce the ohmic and polarization resistance to achieve high performance of solid oxide fuel cell (SOFC). In order to deposit dense electrolyte film and also to impregnate electrolyte into porous anode, ultrasonic spray pyrolysis method is used to deposit the electrolyte film. A $(La_{0.75}Sr_{0.2}Ba_{0.05})_{0.175}Ce_{0.825}O_{1.891}$ (LSBC) electrolyte thin layer was then deposited on $La_{0.3}Sr_{0.7}TiO_3$ (LST) porous anode by ultrasonic spray pyrolysis. During the spraying process, the LSBC could partly impregnate into porous LST. After co-fired at 1350°C, LSBC/LST half-cell was then obtained. A microwave process was also used to further modify the LSBC densification. The microwave heat treatment could help LSBC thin layer more densely and also improve the connectivity between LSBC and LST. From the AC-impedance analysis, the polarization impedance of LSBC/LST half-cell was reduced by microwave modification. The LSBC impregnated into LST successfully by spray pyrolysis as to extending TPBs from LSBC to LST. Microwave energy improved the diffusion between LST and LSBC effectively.

9:20 AM

(ICACC-S3-012-2018) Electrochemical studies on a Na-β"-Alumina+ YSZ composite mixed ionic conductor

L. Ghadbeigi*[1]; T. D. Sparks[1]; A. V. Virkar[1]

1. University of Utah, Material Science and Engineering, USA

An experimental approach was developed to investigate electrochemical behavior of Na-β" Alumina+YSZ ceramic composites under open circuit condition. This composite is a two-phase mixed sodium ion – oxygen ion conductor. Under open circuit condition, three types of experiments in the range of 500°C-1000°C were conducted to study the effect of; (i) applied chemical potential differential of Na across the membrane, (ii) applied chemical potential differential of O_2 across the membrane (fuel cell mode) and (iii) applied simultaneous chemical potential differentials of both Na and O_2. Under an applied chemical potential differential of Na, sodium Nernst potential was observed. Under fuel cell loading, the sample exhibited degradation on the side exposed to hydrogen, which was attributed to Na-β"-alumina in the sample. The measured potential varied with the extent of degradation. Ionic conductivity was measured by a DC method and by electrochemical impedance spectroscopy. Under applied both sodium and oxygen chemical potential differentials, the open circuit voltage (OCV) exhibited Goldman-type behavior. The results will be discussed using a multi-species transport model. Acknowledgements: This work was supported by the National Science Foundation under Grant Number DMR-1407048.

9:40 AM

(ICACC-S3-013-2018) Electrolyte Conductivity and Area Specific Electrode Polarization Resistance of Pt/8YSZ/Pt Over a Wide Temperature Range using a DC Method

A. Szendrei*[1]; T. D. Sparks[1]; A. V. Virkar[1]

1. University of Utah, Materials Science and Engineering, USA

Contemporary transport measurements on solid state electrolyte electrochemical systems are typically done using Electrochemical Impedance Spectroscopy (EIS) from which electrolyte resistance and electrode polarization resistance can be obtained. DC experiments have only focused on electrolyte conductivity typically using a 4-point method to remove electrode polarizations. Using a sensitive ammeter, a high impedance voltmeter, and a constant voltage source the conductivity of a polycrystalline 8YSZ sample was measured down to 119°C using a 4-probe method. The area specific electrode polarization resistance of platinum electrodes was estimated using Kirchhoff's voltage law, thus obtaining electrolyte and electrode properties in a single experiment using the DC method. Arrhenius plots of the electrolyte and the electrode data exhibited linear behavior in the temperature range investigated. The electrode polarization/current density relationship was analyzed considering both the low and high electrode polarization limits of the Butler-Volmer equation. EIS measurements showed good agreement with the DC data.

Sealing Materials, Designs and Approaches

Room: Crystal

Session Chair: Federico Smeacetto, Politecnico di Torino

10:20 AM

(ICACC-S3-014-2018) Development of SOFC sealing materials at the Technical University of Denmark – Towards solutions for today's and tomorrow's challenges (Invited)

R. Kiebach*[1]; I. Ritucci[1]; K. Agersted[1]; P. Zielke[1]; P. V. Hendriksen[1]; A. Wulff[1]; F. Smeacetto[2]; A. Sabato[2]

1. DTU, Denmark
2. Politecnico di Torino, Department of Applied Science and Technology, Italy

Glasses and glass-ceramics are commonly used as sealants in planar solid oxide fuel cell (SOFC) stacks. These seals must provide essential functions like separating fuel and oxygen streams, electrically insulating various cell components, and providing sufficient mechanical support. Unfortunately, improving on one functionality usually comes at the expense of other properties, and the right balance is often difficult to achieve. This presentation will start with a short review of existing solutions and challenges. Presented results will focus on i) the development of Ba-free glasses with high coefficient of thermal expansion (CTE), ii) the interaction of sealing glasses with ferritic steels and (commercially available) coatings, and iii) the long-term stability of commercial and in-house developed sealing solutions. Following recent trends, it can be expected that new generations of SOFCs will be operating at lower temperatures (< 700 °C). Consequently, future stack assembly will take place at lower temperature (e.g. to prevent coarsening of nanostructured electrodes or corrosion of metal supported cells), which will require a re-design of existing solutions or the development of new sealing materials. The final part of the presentation will look into these challenges and first ideas and results to overcome these will be presented.

10:50 AM

(ICACC-S3-015-2018) Effects of contaminants on the ageing of glass ceramic sealants for solid oxide cells (Invited)

D. Montinaro*[1]

1. SOLIDpower SpA, R&D Materials & Process, Italy

Barium-base glass ceramics, normally used as sealants for intermediate temperature applications, are considered the most critical materials limiting the lifetime of SOFC- and SOE-systems and, in the most critical situations, leading to failure. In the present work the ageing behaviour of glass sealants from the Ba-base family of glasses, was simulated by exposing single components to typical SOC operating environment. In addition, the effects of impurities, contained in the raw materials or released from other stack components, were investigated by introducing traces of such elements during the preparation of the glass. Results obtained from this study suggest that the crystalline phases developed after a short (<1000h) ageing period are stable as long as the glass ceramic is not contaminated by foreign elements. Contaminants selectively react with some elements of the glass ceramic, depleting the crystalline or the residual glass phases and promoting the formation of new phases. It was found that the formation of the Barium Chromate at the interface between the glass and the ferritic stainless steel is the main source of delamination after short ageing time. On the other hand, the formation of Si-reach crystalline or glassy phases, probably related to Ba-depletion, were considered the main sources of cracks and delamination after a long (>10000h) ageing time.

11:20 AM

(ICACC-S3-016-2018) Mechanical properties and strength between an SOFC glass ceramic seals and Crofer22APU

I. Ritucci*[1]; R. Kiebach[1]; L. Han[1]; P. Zielke[1]; K. Agersted[1]; P. Hendriksen[1]; H. L. Frandsen[1]

1. DTU, Energy, Denmark

Glass-ceramics are commonly used as seals in solid oxide fuel cells (SOFCs) due to their ability to provide gas tight joints between the different stack components and the versatility in terms of processing. Mechanical integrity of the seals is however a key challenge for the SOFC technology. Mismatch of the coefficient of thermal expansion (CTE) between seal and other materials commonly used in SOFC stacks leads to high residual stresses and greater risk of failure. The Ba-free glass presented here has a promising CTE of 12.8 x 10^{-6} K^{-1} matching the CTE of the remaining materials. The seal was here tested against three different surface modifications of the Crofer22APU: pre-oxidation at 900°C, an alumina coating and a $MnCo_2O_4$ (MCO) coating, which represent some interesting variations in surface roughness and porosity. The pre-oxidized layer is dense and flat, while the alumina coating is rather rough, and the MCO layer is porous. Given a weaker interface, surface roughness and porosity should increase the interface fracture energy. Here, the interfacial fracture energy is measured using the Charalambides method, by sandwiching the glass between treated Crofer22APU bars. The fracture interfaces are characterized by means of scanning electron microscopy and energy-dispersive X-ray spectroscopy to evaluate possible interactions between the seal and the metal that might cause the seal to fail.

S4: Armor Ceramics - Challenges and New Developments

Materials Characterization I

Room: Coquina Salon F

Session Chair: Christopher Marvel, Lehigh University

8:30 AM

(ICACC-S4-013-2018) Measuring hardness when indents are severely damaged

L. J. Vandeperre*[1]

1. Imperial College London, Materials, United Kingdom

When measuring the hardness of hard materials, the number of indents whose size can actually be measured decreases rapidly with increasing load due to extensive cracking and the removal of material during unloading. Only measuring the indents, which have survived is not good laboratory practice as there could be a systematic reason why some indents survive and others not and hence the hardness obtained from a subset of indents might be biased. There is therefore a need for a method of determining the hardness of such indentations which does not rely on examination of the indentations after unloading. For small indentations, the use of load-displacement data recorded during loading and unloading is now well established for this purpose but when the loads are increased on a range of hard materials it was found that the derived values of the elastic modulus of the materials decreased at the loads where cracks started to be observed. Since the elastic modulus of the material is constant this indicates that some of the cracks form during loading and alter the interrelation between displacement and contact area by adding further compliance to the system. A study of the variation of compliance confirmed this. Therefore a new analysis was carried out and yielded a method, which can account for the cracking and give averaged hardness values from series of indentations at different loads.

8:50 AM

(ICACC-S4-015-2018) Disordering energy and excess free energies effects on grain growth in $MgAl_2O_4$ spinels

D. Ferreira Muche*[1]; R. Castro[1]

1. University of California, Davis, Materials Science and Engineering, USA

Nanocrystalline $MgAl_2O_4$ have shown to be a strategic material for armor applications. However, together with nanocrystalline structures, the excess grain boundary free energies (γb) drives the system to massive growth even at relatively low temperatures. Therefore, the understanding of the thermodynamics and kinetics are the key for controlling the microstructure evolution in nanocrystalline these systems. This work quantifies their contribution by a systematic experimental investigation using ultrasensitive Differential Scanning Calorimetry (DSC) on fully dense transparent $MgAl_2O_4$ spinel. The determination of γb as function of the grain size ranging from ~ 8nm to ~150nm has been discussed and interestingly, the γb in $MgAl_2O_4$ spinel showed to possess a notable influence of its grain size and degree of disorder, known to be dependent of grain size. Furthermore, the influence of excess energies on grain growth is addressed at temperature range 1100 to 1300C on fully dense MgAl2O4. Grain boundary mobility and activation energy were experimentally determined showing that excess energies are responsible for the increased mobility, promoting faster growth at nanoscale range.

9:10 AM

(ICACC-S4-016-2018) Applying Data Science to Material Science to Advance Armor Ceramic Research (Invited)

M. C. Golt*[1]

1. U.S. Army Research Laboratory, USA

Within the discipline of data science, researchers have developed methods to obtain knowledge of trends, correlations, and causality in systems by analyzing and visualizing collected data. These methods, when applied to the development and testing of ceramic material systems, could help material scientists gain insights into how these materials can be efficiently manufactured to have superior performance. Discussed are several data science methods that have been employed on characterization data collected on large batches of armor ceramic parts to understand the relationships between processing, microstructure, and ballistic performance. Among the methods used is a technique to efficiently collect ballistic performance data such that the information contained in the dataset is maximized. Once collected, various machine learning and data visualization techniques are applied to the characterization dataset to discover and understand how performance is related to microstructure and processing.

9:40 AM

(ICACC-S4-017-2018) Grain Boundary Segregation of Rare-Earth Additives in Boron Suboxide

C. J. Marvel*[1]; K. D. Behler[2]; J. S. Dunn[2]; J. LaSalvia[2]; M. P. Harmer[1]

1. Lehigh University, USA
2. US Army Research Laboratory, USA

This study is aimed at determining the effects of rare-earth additives on the structure and chemistry of grain boundaries in boron suboxide. Boron suboxide powders were co-doped with combinations of silica and yttrium, ytterbium, and other rare-earth oxides totaling between 5-20 wt. %, and specimens were densified using hot-pressing at 1850 °C for 3 hours. Different rare-earth elements were chosen to investigate the role of dopant size and valence on grain boundary segregation behavior. Grain boundaries were characterized using aberration-corrected scanning transmission electron microscopy (ac-STEM) to determine atomic structures using high-angle annular dark field imaging (HAADF) and chemistries using energy dispersive spectroscopy (EDS). Observations are compared to site occupancy predictions made using first principle calculations of

dopants on free surfaces. The main observations include clear differences in segregation behavior between different dopants, namely modified atomic structures and different excess grain boundary segregation concentrations. Experimental and computational results will be presented.

Materials Characterization II

Room: Coquina Salon F
Session Chair: Jerry LaSalvia, Army Research Laboratory

10:20 AM

(ICACC-S4-018-2018) Mitigation of Amorphization in Boron Carbide Achieved by Silicon doping Through High Temperature Coupling of Boron Carbide and Silicon Hexaboride

A. M. Etzold[1]; V. Domnich*[1]; K. D. Behler[3]; K. Xie[2]; J. LaSalvia[3]; R. A. Haber[1]

1. Rutgers University, Materials Science and Engineering, USA
2. Johns Hopkins University, USA
3. US Army Research Laboratory, Multifunctional Materials Branch, USA

Boron carbide suffers a loss in shear strength under high nonhydrostatic stresses due to nanoscale amorphization. Density functional theory (DFT) simulations indicate silicon doping of the 3-atom linear chains of boron carbide might suppress this amorphization. In this work, diffusion zones in boron carbide/silicon hexaboride diffusion couples were microstructurally, chemically, and mechanically characterized to investigate the possible formation of silicon-doped boron carbide. Diffusion zones were microstructurally and chemically distinct, consisting primarily of silicon-doped boron carbide with widths between 80-280μm depending on temperature and time. While measured nanohardness values for silicon-doped boron carbide were lower than high-purity boron carbide, Raman spectra from indents showed a significant decrease in amorphization peak intensities. Rietveld refinements of XRD patterns indicate silicon replaces boron at the center atom position in the 3-atom chains. Consistent with DFT predictions, results demonstrate that silicon atoms can be incorporated into boron carbide's lattice and mitigate stress-induced amorphization.

10:40 AM

(ICACC-S4-021-2018) The Kinetics and Mechanisms of Abnormal Grain Growth Grain in Si-doped Boron Suboxide

C. J. Marvel*[1]; K. D. Behler[2]; J. LaSalvia[2]; M. P. Harmer[1]

1. Lehigh University, USA
2. US Army Research Laboratory, USA

Boron suboxide is a promising armor ceramic because of its low density and high hardness. One challenge for boron suboxide is its inherent low fracture toughness. This can be significantly improved by engineering grain boundaries with liquid-like nanolayer films. This study is aimed at characterizing abnormal grain growth in boron suboxide processed with silica additives. Abnormal grain growth is used as a marker for possible complexion transitions and existence of liquid-like nanolayer films. Boron suboxide samples with 1-5vol.% silica (densified by pressure-assisted sintering at 1750°C) were heat-treated at various temperatures and times to track the evolution of abnormal grain growth. The number and fraction of abnormal grains were plotted on a time-temperature-transformation diagram to represent the kinetics of abnormal grain growth. Furthermore, grain boundaries of both abnormal and small grains were characterized by sub-Å resolution electron microscopy to determine structural and chemical differences. Experimental procedures and results will be presented.

11:00 AM

(ICACC-S4-020-2018) Nanotwinning in Boron Suboxide

C. Kunka*[1]; Q. An[2]; G. Subhash[1]

1. University of Florida, Mechanical and Aerospace Engineering, USA
2. University of Nevada, Reno, Chemical and Materials Engineering, USA

Recent experimental and computational investigations have demonstrated that nanotwinning of both metals and ceramics can dramatically increase mechanical properties. This effect is largely attributed to the arrest of slip, but other deformation mechanisms can be important considerations, especially for icosahedral systems. Hence, this study explores the effects of nanotwinning in boron suboxide, an advanced ceramic with extraordinary theoretical strength but a susceptibility to amorphization. With transmission electron microscopy, quasistatic/dynamic indentation, and ultrasonic testing, we rationalize how nanotwinning benefits the mechanical response of boron suboxide.

11:20 AM

(ICACC-S4-019-2018) An Analysis and Interpretation of Planar Features in Boron Carbide (Invited)

J. W. McCauley*[1]

1. Johns Hopkins University/Army Research Laboratory, USA

Recent work on various hot pressed SiC materials, has suggested that their transitional velocities depend on hardness and a form of ceramic plasticity. It has also been suggested that the presence of planar features may have been a major contributor to this "quasi-plasticity". There is evidence that the presence of "twinning" in boron carbide enhances the thermoelectric Seebeck coefficient and certain mechanical properties. Their presence may also enhance quasi-plasticity, so it would be important to try to understand why they form and how to control and model them. In the assumed solid solution between $B_{12}C_3$ and $B_{13}C_2$, it has also been observed that the planar features increase with increasing Boron content and certain processing techniques. A review of selected examples in the boron carbide system will be presented. While some may be twins, many may not be crystallographic twins, but forms of compositionally modulated structures that may exhibit grain boundary characteristics; among these may be Wadsley defects (crystallographic shear planes (CS)), which are elegant atomic structure mechanisms to create electronic stability. Examples from other systems will also be presented.

S6: Advanced Materials and Technologies for Direct Thermal Energy Conversion and Rechargeable Energy Storage

Materials Design for Lithium Batteries and Supercapacitors I

Room: Tomoka A
Session Chair: Naoaki Yabuuchi, Tokyo Denki University

8:30 AM

(ICACC-S6-010-2018) Precise Surface Control of Cathode Materials for Improved Battery Performance (Invited)

A. Cao*[1]

1. Institute of Chemistry, Chinese Academy of Sciences, China

Along with the increase in energy density of LIBs, major concerns have been aroused on their reliability and safety. In this contribution, we'll reported our progress on the protection and stabilization of cathode materials by precisely controlling the atomic structure on the particle surface. Two different strategies will be discussed. First, the nanocoating technique to form uniform surface nanoshells with their thickness controlled at

one nanometre accuracy: Solution-based synthetic routes will be discusses to form different coating materials, typically metal oxides, metal phosphates, and carbon for the protection of cathode materials. We demonstrated that the battery performance was directly related to the coating state. Second, a surface doping strategy to achieve a precise manipulation of the surface vacant lattices: We identified that the diffusion path for metal ions during the charge/discharge process could be successfully controlled by inserting suitable metal ions, typically Al^{3+} for $LiMn_{1.5}Ni_{0.5}O_4$, which ensured a very effective way to suppress the structural degradation and parasitic side reactions at the surface for such a high voltage cathode material.

9:00 AM

(ICACC-S6-011-2018) Electrochemical activity in borates and oxyborates toward lithium

V. Pralong*[1]; B. LeRoux[2]; S. Malo[1]; A. Guesdon[1]; F. Laine[1]; J. F. Colin[2]; C. Martin[1]

1. CRISMAT ENSICAEN Université de Caen, Laboratoire de Cristallographie et Sciences des Matériaux, France
2. CEA, Laboratoire d'Innovation pour les Technologies des Energies Nouvelles et les Nanomateriaux, France

Regarding the field of energy storage, the design of new materials that are showing high ionic mobility together with being economic and environmental benign is crucial. Our research is focused on the synthesis by soft chemistry of new frameworks with large tunnels or layered structures in order to favor ionic mobility. As an example, we will discuss the lithium insertion into a copper iron oxyborate, i.e. Cu_2FeBO_5. Four electrons are involved in the course of the first discharge, which is ascribed to the Cu^{2+}/Cu redox couple. The reaction is associated with a collapse of the structure but this material shows a reversible and stable capacity of 180 mAh/g at 2.35V vs. Li^+/Li. Moreover, A new borate compound LiFe0.5Co0.5BO3 was been successfully synthesized for the first time by a multiple-step process. This compound delivers a very interesting first discharge capacity of 120mA h g_1 at C/20 rate without in situ carbon coating. In this presentation we will discuss the possible use of transition metal borates and oxyborates as electrode materials for Li/Na ions batteries.

9:20 AM

(ICACC-S6-012-2018) Thermodynamic characterization of de-lithiated $LiNi_{0.4}Mn_{0.4}Co_{0.2}O_2$ (NMC) cathode materials for Lithium-ion batteries

W. Zhao*[1]; H. J. Seifert[1]

1. Karlsruhe Institute of Technology, IAM-AWP, Germany

$LiNi_xMn_yCo_zO_2$ (NMC) phases are commercially most promising cathode materials for Lithium-ion battery design. However, there is lack of knowledge of fundamental electrochemical-thermodynamic correlations of these materials. This work is focused on the thermodynamic stabilities of NMC phases of various compositions, especially in charged state (partially de-lithiated state) to contribute to better understanding of thermal and safety behavior. Compared with other cathode materials, NMC safety performance is relatively poor, according to thermal runaway tests. A systematic study is performed to investigate the structural changes and thermodynamic characters of $LiNi_{0.4}Mn_{0.4}Co_{0.2}O_2$ upon chemical extraction. The de-lithiated experiments are performed by using the oxidizing solution $(NH_4)_2S_2O_8$. The impacts of lithiation degree in $Li_{1-x}Ni_{0.4}Mn_{0.4}Co_{0.2}O_2$ on the structural and thermodynamic stabilities are investigated. The crystal chemistry is determined by X-ray diffraction, and the enthalpy of formation is measured by high temperature oxide solution calorimetry. In addition, the NMC phases are investigated by differential thermal analysis. At the same time, the released gases of the reactions are investigated by mass spectrometry. The presented results are significant for better understanding the thermal and safety behavior of changed cathode materials of NMC phases.

9:40 AM

(ICACC-S6-046-2018) Crystallization behavior of the Li_2S–P_2S_5 glass electrolyte for sulfide-based all-solid-state lithium batteries

S. Mori*[1]

1. Osaka Prefecture University, Materials Science, Japan

Lithium-ion secondary batteries exhibit excellent charge-discharge cycle characteristics and high energy density. However, there is a problem in safety for using a flammable organic solvent. Therefore, we focused on the sulfide-type all-solid-state batteries using a non-flammable inorganic solid electrolyte. In the present study, thermal stabilities of the positive electrode consisting of the active material $LiNi_{1/3}Mn_{1/3}Co_{1/3}O_2$ (NMC) and the $75Li_2S$-$25P_2S_5$ (LPS) glass electrolyte have been investigated by transmission electron microscopy (TEM). It was found that the exothermal reaction originated from the crystallization of the LPS glasses. To better understand the origin of the exothermic reaction, furthermore, the precipitated crystalline phase of the LPS glass was identified. In situ TEM observation revealed that -Li_3PS_4 precipitated at approximately 200 °C, and then $Li_4P_2S_6$ and Li_2S precipitated at approximately 400 °C. Since the precipitation of $Li_4P_2S_6$ and Li_2S could not detected in the single LPS glass, interfacial contact with NMC would have a significant influence on the crystallization behavior in the LPS glass electrolyte and the exothermal reaction. On the basis of the first principle calculation, the origin of exothermic reaction in the NMC–LPS composites will be discussed.

Materials Design for Lithium Batteries and Supercapacitors II

Room: Tomoka A

Session Chair: Valerie Pralong, CNRS ENSICAEN

10:20 AM

(ICACC-S6-013-2018) Hierarchically structured cathode materials for lithium ion batteries (Invited)

J. R. Binder*[1]; A. Höweling[1]; N. Bohn[1]; A. Wagner[1]

1. Karlsruhe Institute of Technology, Institute for Applied Materials (IAM), Germany

Electrochemical energy storage systems will play an outstanding role for flexible, decentralized and mobile energy concepts due to their high degree of efficiency and the flexible application possibilities. The key challenge in the context of new electrochemical energy storage devices is the development of improved materials which are adjusted to specific applications. Common electrode materials for lithium ion batteries can be modified in various ways to achieve an enhancement of thermal, mechanical, and chemical stability as well as electronic and ionic conductivity, e.g. by variation of the chemical composition using dopants or by functional coatings to protect the particle surfaces. Another possibility to improve the electrochemical performance of active battery materials is the realization of a hierarchical structure. In this presentation the approach of hierarchically structuring of cathode materials by an established process of grinding, spray drying and calcination will be presented using the example of NCM materials and high voltage spinels. The relationship between process parameters, morphology/microstructure and electrochemical performance of the as prepared lithium ion batteries will be presented.

10:50 AM

(ICACC-S6-014-2018) A Step Toward Designing Electrodes for Higher Energy Density Lithium-ion Batteries

P. Antitomaso*[1]; L. Savignac[2]; L. Taylor[3]; S. Généreux[1]; S. Rousselot[1]; T. Bibienne[4]; M. Pasquali[3]; S. Schougaard[2]; M. Dolle[1]

1. University of Montreal, Canada
2. University of Quebec in Montreal, Canada
3. Rice University, USA
4. Nemaska Lithium, Canada

Conventional composite electrodes consisting of a mixture of active material (AM), carbon black and polymeric binder spread on a metallic current collector could impede the development of high energy density batteries as a large part of its composition only act as dead weight up to 25%. Binder free, self-standing and flexible electrode only made of AM and single wall carbon nanotubes (SWNT) were previously investigated with up to 95 wt.% of AM and promoted high specific capacity and steady with cycling. The high electronic conductivity obtained thanks to the CNT network in the self-supported electrodes allowed the cycling of uncoated $LiFePO_4$ (LFP) particles. However, some performance losses (especially at high C-rate) evidence that the SWNT network does not allow reaching all single LFP particles. In order to reach a multiscale conductivity, polymerisation of the 3,4-ethylene-dioxythiophene (EDOT) by reinsertion of lithium into $Li_{(1-x)}FePO_4$ was attempted. Indeed, PEDOT on uncoated LFP is well known to create a conductive network between the particles. Electronic conductivity at the macroscopic and microscopic scale in the electrode can be reached thanks to the SWNT and the PEDOT leading to an uncoated $LiFePO_4$ PEDOT self-standing electrode. The electrode preparation, their physico-chemical properties and electrochemical performance will be discussed and compared to that of a conventional electrode.

11:10 AM

(ICACC-S6-015-2018) Strain effect on cathode properties of $LiNi_{0.5}Mn_{1.5}O_4$ spinel for Li-ion batteries

T. Kozawa*[1]

1. Osaka University, Joining and Welding Research Institute, Japan

Among the cathode materials for Li-ion batteries, $LiNi_{0.5}Mn_{1.5}O_4$ (LNMO) is a promising high-voltage cathode with a spinel structure. The way to improve cathode properties includes a particle size reduction, surface coating with conducting materials, and control of particle morphology. It is generally considered that lattice strains and defects in the crystal are undesirable due to hinder the ion diffusion. However, we have revealed that the insertion of lattice strains into LNMO particles leads to improve its electrochemical performances. In this study, the LNMO powder was treated by simple ball milling to insert lattice strains. The treated LNMO exhibited a better cathode performance than the pristine LNMO sample.

S7: 12th International Symposium on Functional Nanomaterials and Thin Films for Sustainable Energy Harvesting, Environmental, and Health Applications

Functional Nanostructures for Energy Conversion and Storage and Catalysis I

Room: Coquina Salon C

Session Chairs: Dawit Gedamu, École de technologie supérieure (ETS); Daniel Chua, National University of Singapore

8:30 AM

(ICACC-S7-010-2018) In situ Synchrotron X-ray Spectroscopic Characterization of Energy Material (Invited)

C. Dong*[1]

1. Tamkang University, Department of Physics, Taiwan

A new age of human demand for sustainable energy is dawning and material scientists are dedicated to finding novel materials for clean energy. It has always been a considerable challenge to advance the efficiencies of energy conversion, generation, and storage of current energy materials. In numerous energy materials, such as smart materials, artificial photosynthesis, nanoscatalysts, and energy storage devices, it is critical to examine the local atomic/electronic structures of the interfacial region under real working environment. Without knowing fundamental properties such as atomic/electronic structures and especially how they adjust in the interfacial region upon chemical/physical reaction, it is arduous to manufacture a better material for boosting energy conversion/generation/storage efficiency. Synchrotron x-ray spectroscopies, including x-ray absorption and emission spectroscopies are very powerful and effective tactics to characterize the local unoccupied and occupied states. Taking advantage of the in situ technique, monitoring the variation of atomic/electronic structures of the energy material in operational environment now becomes possible. This presentation will report the synchrotron in situ x-ray spectroscopies, including soft- and hard-x-ray spectroscopies for various energy materials. Recent development of in situ technique, and emergent synchrotron microscopy will be also presented.

9:00 AM

(ICACC-S7-011-2018) Molten salt oxidation of nickel in KOH melts for the direct production of electroactive nickel oxides to be used in supercapacitor applications (Invited)

M. Urgen*[1]; N. Tokmak[1]

1. Istanbul Technical University, Turkey

Since the invention of capacitor in 1745, very high capacity capacitors that fill the gap between batteries and conventional capacitors could only found widespread use in the beginning of this century. Today these capacitors also named as super capacitors/ ultra capacitors or electrochemical capacitors are extensively used in automotive industry, electrical vehicles and also for power quality. Between the two basic types of supercapacitors, electrical double layer capacitors (EDLC) are more widely used. However, for increasing energy density, pseudo capacitors or a combination of EDLC and pseudo capacitors provides more opportunities. Electroactive nickel oxides have attracted substantial interest as pseudo capacitor electrodes due their high capacity. In this presentation benefits of direct production of these oxides on nickel substrates will be explained and the methods for direct production will be compared. Our recent results on the usage of molten salt oxidation of nickel foams for super capacitor applications will be presented.

9:30 AM

(ICACC-S7-012-2018) Freestanding Holey Thin Films for Battery Energy Storage Systems (Invited)

K. Marcus*[1]; Y. Yang[1]

1. University of Central Florida, Materials Science and Engineering, USA

The rapid advancement of portable and wearable technologies has challenged research to improve upon current battery energy storage systems. By using nanotechnology, it is now possible to access more of the energy storage theoretical values that have been unattainable thus far. We have developed a method to create freestanding holey thin films through combinations of electrochemical and chemical vapor deposition (CVD) techniques to be used in battery energy storage systems. Freestanding thin films promote excellent contact between the residual conductive framework and any functionalized active component specific to the designed material. Without requiring any other additives, the as-prepared freestanding thin films can be mechanically and chemically tuned to allow for use in a wide range of applications. Incorporation of micro- and nano-sized holey structures dramatically enhances the electrochemically active surface area, which is essential for facilitating appropriate reactions in conversion type energy storage systems. Combining the freestanding and holey components with an active layer effectively enhances conductivity and reduces the electron transfer distance at the electrode-electrolyte interface. Herein, two separately designed freestanding holey thin films were successfully used as cathode materials for lithium-sulfur battery (Li-S) and magnesium-ion battery (MIB) energy storage systems.

Perovskites and Other Optical Materials for Light Management

Room: Coquina Salon C
Session Chair: Chung-Li Dong, Tamkang University

10:20 AM

(ICACC-S7-013-2018) Frequency Converting Lanthanide-Based Materials and Molecules (Invited)

E. Hemmer*[1]

1. University of Ottawa, Chemistry and Biomolecular Sciences, Canada

Based on their outstanding optical properties, frequency converting Ln^{3+}-based compounds have been suggested for a whole gamut of applications including the field of biomedicine and solar energy conversion. For instance, the capability of Ln^{3+}-based materials to emit visible and near-infrared (NIR) light under NIR excitation is sought after when aiming for biomedical applications. This is due to the fact that NIR light penetrates deeper into biological tissue when compared to UV or visible light. Fluorides, e.g. $NaGdF_4$, are commonly considered as suitable host materials for upconverting and NIR emitting Ln^{3+} ions (e.g. Er^{3+}) and their preparation via the thermal decomposition process has been widely studied. A microwave-assisted approach offers a promising alternative for the synthesis of Ln^{3+}-nanoparticles of controlled size and crystalline phase. Ln^{3+}-based compounds have also been suggested to reduce efficiency loss in solar cells. In this context, Ln^{3+} ions such as Eu^{3+} or Tb^{3+} are suitable candidates emitting visible light under UV excitation. Moreover, Eu^{3+} is a model ion of particular interest since its emission spectrum allows us to draw conclusions about the chemical environment of Eu^{3+} ions as shown on the example of a Tb^{3+}/Eu^{3+}-complex. This presentation will shine a light on the versatile landscape of Ln^{3+}-based materials and molecules focusing on materials synthesis and Ln^{3+}-specific optical features.

10:50 AM

(ICACC-S7-014-2018) Semiconducting halide perovskite materials for PV and as photodetectors (Invited)

D. M. Gedamu*[1]; I. M. Asuo[2]; F. Rosei[2]; S. G. Cloutier[1]; R. Nechache[1]

1. École de technologie supérieure (ÉTS), Department of Electrical Engineering, Canada
2. INRS-EMT, Centre for Energy, Materials and Telecommunications, Canada

Hybrid perovskite semiconductors recently emerged as one of the promising material systems particularly for solar cell applications because of low cost material synthesis, high photon absorption, tunable bandgap and versatile material properties and fabrication processes. In the last 5 years, an unprecedented effort has been devoted in tailoring perovskite grain size and control of pinhole in the perovskite thin film. However an efficient technique in tailoring the microstructure at ambient environmental conditions in a simple experimental strategy has not been realized. In addition, processing of the film at ambient environmental condition poses a huge problem since hybrid perovskite materials have strong affinity to atmospheric moisture. In here we will show ambient processed perovskite thin films using different solvent treatment techniques to control grain size and a possible mechanism to reduce the number of pinholes on perovskite film that would allow for the better performance of the cell. Single and triple cation based perovskite thin films which the microstructure is controlled by solvent treatment will be demonstrated and the PV performance will also be presented. In addition, we also present results from the recently developed highly performing halide perovskite nanowire based photo detectors.

11:20 AM

(ICACC-S7-015-2018) High-performance organohalide perovskite nanowire photodetector (Invited)

I. M. Asuo*[1]

1. L'École de technologie supérieure (ETS), Electrical, Canada

Organometal halide perovskites have exhibited outstanding optoelectronic properties such as tunable optical properties, high charge mobility, and long charge diffusion length. In this work, we demonstrate the synthesis of organohalide perovskite nanowires by spin coating method and fabrication of simple-planar photodetector without charge extraction layers. The photoresponse measurements indicate a broad spectral response from 300 to 850 nm, high spectral responsivity, time response and photocurrent. These results indicate that photocurrent generation is due to the charge transfer within the nanowire network.

11:40 AM

(ICACC-S7-016-2018) Electrospun Perovskite Fibers – New Flexible 1D Nanocomposites for Light Harvesting Applications (Invited)

C. Bohr*[1]; S. Oez[1]; A. Lepcha[1]; M. Schütz[1]; F. Staub[2]; T. Kirchartz[2]; S. Mathur[1]

1. University of Cologne, Institute of Inorganic Chemistry, Germany
2. Forschungszentrum Juelich, IEK-5 Photovoltaics, Germany

Due to their high efficiencies (up to 22%, comparable to established thin-film solar cells like $Cu(In,Ga)Se_2$ or CdTe), interest in perovskite solar cells is growing rapidly. In contrast to the high quantity of publications dealing with planar, rigid solar cells, one dimensional, fiber like solar cells have not been focused. There for, single step fabrications of phase-pure organic-inorganic lead and tin halide perovskite ($MeNH_3MI_3$; M= Sn,Pb) fibers are presented. The experimental results demonstrate first comprehensive data on inert electrospinning of an air sensitive organic-inorganic hybrid material. X-ray diffraction and steady state photoluminescence data confirmed the phase purity, as well as photonic properties. Planar heterojunction solar cells prepared by direct electrospinning of composite fibers onto compact TiO_2 coated FTO substrates showed a

photoelectric response under simulated sunlight conditions. Despite their lower efficiencies (compared to planar PV cells), 1D hybrid perovskite fibers are potential elements for flexible optoelectronics.

S8: 12th International Symposium on Advanced Processing and Manufacturing Technologies for Structural and Multifunctional Materials and Systems (APMT12)

Polymer-Based Processing

Room: Coquina Salon A
Session Chairs: Enrico Bernardo, University of Padova; Lisa Rueschhoff, Air Force Research Lab

8:30 AM

(ICACC-S8-008-2018) Multifunctional polymer-derived (carbo) nitride ceramics (Invited)

A. Lale[1]; S. Bernard*[1]

1. CNRS UMR 7315, Ceramic Research Institute, France

There is a trend toward more flexibility and an increased interest in "smart" and "adaptive" materials with the objective to meet most industrial specifications. Nitrides and carbonitrides can be considered as such strategic materials. They attract increasing interest due to their properties targeted for future materials and technologies especially because they bear intrinsic multifunctionality through the synthesis of multi-element compounds. Inherent difficulties to the traditional techniques for manufacturing such multi-element materials can be overcome by the development of synthetic paths where chemistry of materials and ceramic science are combined rationally to process multi-scale complex solid state architectures. The Polymer-Derived Ceramics (PDCs) route offers new preparation opportunities in ceramic sciences. The molecular origin of preceramic polymers and the possibility to shape then pyrolyze them into advanced materials play a major role in the preparation of ceramics endowed with properties that reach far beyond those of existing materials. Here, we report the design of multi-element ceramics in the carbonitride and nitride systems. This presentation will be particularly focused on the polymer synthesis/processing/pyrolysis to generate amorphous ceramics, composites and nanocomposites in the Si-Ti-N-C systems that display high potential as catalyst and electrocatalyst supports.

9:00 AM

(ICACC-S8-009-2018) Preparation of High-Pressure Phases from Polymer-Derived Amorphous Materials (Invited)

Y. Sugahara*[1]

1. Kagami Memorial Research Institute for Materials Science and Technology, Waseda University, Department of Applied Chemistry, School of Advanced Science and Engineering, Japan

High-pressure phases are attracting increasing attention for example as hard materials. The use of amorphous materials could allow the crystallization of high-pressure phases at relatively low pressure and temperature in comparison with their standard synthesis using crystalline materials stable under ambient conditions. Polymer-derived ceramics (PDCs) have been developed for various directions, such as non-oxide fibers and composites, and the PDC process is also applicable for the preparation of non-oxide amorphous materials by employing relatively mild pyrolysis conditions. Thus, it is interesting to prepare high-pressure phases by using amorphous materials derived by the PDC process. One example is preparation of cubic silicon nitride from amorphous materials prepared by the pyrolysis of perhydropolysilazane using a shock-compression apparatus. The use of amorphous materials was advantageous for the crystallization of cubic silicon nitride. Another example is the preparation of cubic boron nitride using a belt-type apparatus from amorphous materials obtained via the reaction between boric acid and urea and subsequent annealing under ammonia. The formation of cubic boron nitride was promoted by the use of amorphous materials if water was added.

9:30 AM

(ICACC-S8-010-2018) Highly Porous Mullite Ceramics from Engineered Alkali Activated Suspensions

E. Bernardo*[1]; A. Rincon[1]; H. Elsayed[1]

1. University of Padova, Dipartimento di Ingegneria Industriale, Italy

Air may be easily incorporated by vigorous mechanical stirring, with the help of surfactants, of activated geopolymer-yielding suspensions. The cellular structure is stabilised by the viscosity increase caused by curing reactions, configuring an 'inorganic gel casting'. The present investigation is aimed at extending this approach to mullite foams, obtained by the thermal treatment of engineered alkali activated suspensions. 'Green' foams were first obtained by gel casting of a suspension for Na-geopolymer enriched with reactive γ-Al_2O_3 powders. Sodium was later extracted by ionic exchange with ammonium salts. In particular, a complete removal of Na^+ ions was achieved by immersion in ammonium nitrate solution (at 60 °C, overnight), with retention of the cellular structure. Finally, the ion-exchanged foams were successfully converted into pure mullite foams by application of a firing treatment at 1300 °C, for 1 h. Activated suspensions were also used as novel base materials for direct ink writing of Na-geopolymer/γ-Al_2O_3 three-dimensional composites scaffolds, later subjected to the same treatments (ion exchange and conversion into mullite) applied to foams. Preliminary results concerning the obtainment of porous SiAlON ceramics, from the same process (except for the firing in N_2 instead of air) will be presented as well.

10:10 AM

(ICACC-S8-011-2018) Synthesis of $M_1Ti_2(PO_3)_4$ (M_1= Li, Na, K,) compounds by the polymeric steric entrapment method and their thermal expansion behavior

D. Ribero*[1]; K. Tseng[1]; K. C. Seymour[2]; W. M. Kriven[1]

1. University of Illinois, Materials Science and Engineering, USA
2. Pratt & Whitney, Aviation & Aerospace, USA

Pure crystalline $LiTi_2(PO_4)_3$ (LTP), $Li_{1.3}Al_{0.3}Ti_{1.7}(PO_4)_3$ (LATP), $NaTi_2(PO_4)_3$ (NTP) and $KTi_2(PO_4)_3$ (KTP) compounds were produced in a single step by the polymeric steric entrapment method. Ethylene glycol (EG) monomer ($HOCH_2CH_2OH$) was used as the organic carrier for the precursors. It was found that the proper dissolution and mixing of the reagents was even more important than the nitrates:polymer ratio. Excess of isopropyl alcohol, pH modification and mechanical re-dispersion of the gels formed during the synthesis enabled the production of single phase powders. In general, powders at low calcining temperatures were in the nano range with high specific surface area (500°C – 700°C) with rapid crystal growth. However, reduction in the specific surface area as well as pre-sintering were notice at high temperature (1000°C). The thermal expansion behavior of LTP, LATP, NTP and KTP were measured by in situ, high temperature, synchrotron powder diffraction. It was demonstrated that the CTEs were neither constant nor linear. LTP and LATP exhibited a positive but decreasing thermal expansion along the a-axis in the 25°C - 1000°C range, NTP displayed a negative but increasing thermal expansion in the 25°C - 1200°C range. KTP revealed a negative but increasing behavior up to ~500°C which then became positive and increasing up to ~1300°C.

10:30 AM

(ICACC-S8-012-2018) Effect of Atmospheric Composition on Thermal Condensation Reaction of Polysilane as Ceramic Precursor

M. Narisawa*[1]; R. Hanatani[1]; K. Yamada[1]; H. Inoue[1]

1. Osaka Prefecture University, Japan

Polydimethylsilane (PDMS) is known to be useful as ceramic precursor after condensation reaction in an inert atmosphere at temperatures of 400-450 C. In this reaction. Si-Si bonds in PDMS main chains are considered to be converted to Si-C bonds by methyl group insertion (called "Kumada rearrangement"). Thus obtained condensates (named polycarbosilane (PCS)) acquire melt spinnability and solubility for organic solvents, although PDMS itself does not have meltability, and is not soluble to any organic solvents. The conversion yield of PCS from PDMS are, however, usually 50-60 mass%. In spite of many attempts to improve the PCS yield from PDMS using an autoclave or catalytic additives, substantial progress has not been reported for a long period. Recently, we investigated the effect of CO_2 coexistence under relatively high pressure (2-5 bar) for thermal condensation reaction of PDMS. The starting temperature of "Kumada rearrangement" was lowered, and the apparent PCS yield from PDMS was increased beyond 80 mass%. The yields seem to depend on the pressure of CO_2 and a temperature profile of the used chamber. In the presentation, we will describe the basic information about the CO_2 effect on the PDMS condensation, and some characterization results of thus obtained "PCS".

10:50 AM

(ICACC-S8-013-2018) Processing of Organized Ceramic Thin Film Nanocomposites via Macromolecular Self-Assembly

L. M. Rueschhoff*[1]; L. A. Baldwin[1]; Z. D. Apostolov[1]; H. Koerner[1]; J. D. Berrigan[1]; T. Pruyn[1]; M. Cinibulk[1]; M. B. Dickerson[1]

1. Air Force Research Laboratory, USA

Nanoscale controlled ceramics and ceramic composites exhibit extraordinary mechanical properties, including elastic deformation and high toughness, but are difficult to fabricate using scalable production methods. The ability to control pre-ceramic polymer (PCP) patterning by using bottom-up approaches enables production of hierarchical ceramic components with potentially enhanced mechanical properties. The combination of block copolymers (BCPs) and pre-ceramic polymers (PCPs) enables forced patterning of the PCPs as a result of the self-assembly of the BCPs. BCPs contain two or more chemically-distinct blocks that phase separate into well-defined nanostructures (e.g. lamella). Subsequent pyrolysis of the material converts the PCP into a structural ceramic material while removing the self-assembled BCP from the structure. Nanocomposite materials can then be created through backfilling these structures with a second polymer or ceramic phase. Preliminary results on processing optimization of silicon carbide PCP and polystyrene-poly(methyl methacrylate) (PS-b-PMMA) lamellae forming BCP thin films will be presented.

11:10 AM

(ICACC-S8-014-2018) Synthesis and characterization of novel lignin based composites

K. Hall*[1]; Y. Ji[2]; S. Gupta[1]

1. University of North Dakota, Mechanical Engineering, USA
2. University of North Dakota, Chemical Engineering, USA

Fused deposition process (FDP) is defined as a type of Additive Manufacturing (AM) process where polymeric feedstock is extruded into filaments which then are deposited by 3D printing, and the solidification occurs during cooling of the melt. Currently, complex structures are being developed by FDP. In this presentation, synthesis and characterization of novel lignin based composites will be fabricated by additive manufacturing. More particularly, the effect of critical parameters like printing speed, particulate content and chemistry will be identified.

11:30 AM

(ICACC-S8-040-2018) Compositional effect on the ionic conductivity in doped $LaAlO_3$ base ceramic system (Invited)

P. Singh*[1]

1. Indian Institute of Technology(BHU), Department of Physics, India

In the present work, we investigated on compositional effect on the ionic conductivity of $LaAlO_3$. For this, at first, we co-substituted 10 mole% of Sr and Mg at A- and B- site of $LaAlO_3$, respectively and then we synthesized double substituted with A″ (with A″ = Ba and Sm) at A-site to form $A''_xLa_{0.9-x}Sr_{0.1}Al_{0.9}Mg_{0.1}O_{3-d}$ with x = 0.01 and 0.03. The samples were synthesized by auto-combustion technique and found to possess high density. The X-ray diffraction patterns indicated that both Ba and Sm doubly substituted systems formed single phase solid solution having structure similar to pure $LaAlO_3$. EDX results confirmed elemental compositions and revealed variation in Oxygen deficiency which was in correlation with mobile charge concentration. The conductivity was found to be enhanced considerably in substituted samples in comparison to pure $LaAlO_3$. Also, it was observed that the enhancement in the conductivity was higher for Ba double substituted system. The transference number of the systems was calculated in a unique way and that suggested that Ba substituted system is of type I ionic conductor while Sm substituted system is of type II ionic conductor.

S11: Advanced Materials and Innovative Processing Ideas for the Production Root Technologies

New Concepts and Emerging Technologies for Enhanced Product Performance

Room: Ponce de Leon
Session Chairs: Heechae Choi, Virtual Lab Inc.; Byung-Koog Jang, National Institute for Materials Science (NIMS)

8:30 AM

(ICACC-S11-012-2018) One-step synthesizable heterostructure photocatalyst discovered by DFT thermodynamics calculations (Invited)

H. Choi*[1]

1. Virtual Lab Inc., Republic of Korea

ZnS is a promising photocatalytic material having a strong reduction power (-1.04 V to NHE). However, the severe electron-hole recombination of ZnS in its direct band gap implies that there are many chances to improve its photocatalytic activities via materials engineering for accelerated electron-hole separation. Using density functional theory (DFT) calculations, we found that ZnS photocatalyst has large band offset and strong tendency of electron-hole transfers with nickel sulfide phases (NiS, Ni_9S_8). In addition, nickel sulfide phases (Ni_xS_y) are predicted to be thermodynamically immiscible with ZnS. Our experiments clearly demonstrated that ZnS-Ni_xS_y phases are well separated and exhibit excellent photocatalytic activities in heterojunctions. The immiscibility of ZnS and Ni_xS_y phases and large band offsets make the ZnS-Ni_xS_y composite as an ideal heterostructure photocatalyst which can be synthesized via one-step processes and possess strong electron-hole separation capability.

8:50 AM

(ICACC-S11-013-2018) Synthesis method of carbon fiber reinforced thermoplastic with high performance (Invited)

T. Yamamoto*[1]; K. Uematsu[1]; S. Yabushita[1]

1. Nagoya University, Materials and Design Innovation Engineering, Japan

The composite materials were synthesized to create high functionality. For example, carbon fiber reinforced plastic (CFRP) was light and high stiffness to be used as constructional materials in the field of aircraft industry. The mechanical properties of the composite materials depended on the surface adhesion between raw materials. Hence, this study focused on the surface properties between them. We improved the surface adhesion to enhance the mechanical properties of carbon fiber reinforced thermoplastics (CFRTP). Polymer colloid including the same components as the thermoplastic was prepared through the polymerization or using thermoplastic powder and surfactant. The polymer particles were adsorbed on the carbon fibers (CFs) by electrophoresis. The amount of the adsorbed particles on CFs were controlled with applied voltage. The surface adhesion between the modified CFs and thermoplastic were evaluated to measure the interfacial shear strength (ISS). As a result, the ISS was increased with the amount of the adsorbed particles increasing. CFRTP was prepared through hot press method using the modified CFs, and the three-point bending test was examined. Improvement of surface adhesion lead to the high mechanical properties of the CFRTP. Thus, CFRTP with high performance was synthesized with colloidal techniques. CFRTP is expected to be applied to automobiles to realize the sustainable development.

9:10 AM

(ICACC-S11-014-2018) Design and Process of a Ceramic and Polymer Composite with High Strength and Low Weight Using Binder-Jet 3D Printing

D. Kim*[1]; J. Bae[1]; S. Park[1]; J. Choi[1]; J. Lee[1]; E. Kim[1]

1. Korea Institute of Industrial Technology, Republic of Korea

3D printing technology is recently being highlighted as a innovative manufacturing process. Among various 3D printing methods, a binder-jet 3D printing using ceramic powder is particularly interesting since considerably reducing production time of a traditional sand mould. However, high building cost by the binder-jet 3D printer needs to be lower to increase an industrial application of the 3D printing. In this study, a high strength and low weight ceramic and polymer composite is studied for less material consumption and build time saving. The adhesive property of a current binder such as furan are improved using various chemical additives, and low weight structure are investigated with designing a microstructure. Our results will play a significant role in manufacturing of 3D ceramic products having a complex shape as well as conventional sand moulds using the 3D printing technique.

9:30 AM

(ICACC-S11-015-2018) Mechanical properties of CNTs reinforced ceramics nanocomposites (Invited)

B. Jang*[1]; K. Lee[2]; Y. Han[3]

1. National Institute for Materials Science, Research Center for Structural Materials, Japan
2. Kookmin University, Republic of Korea
3. Wuhan University of Technology, China

CNT(Carbon Nanotube) have attracted great interest because of their unique structural, electronic, physical, and thermal properties, such as high electrical conductivity, thermal conductivity, and elastic modulus. It has been reported that CNT are 100 times stronger and 6 times lighter than steel. CNT addition into engineering ceramics is expected to offer good damage and wear resistance, exhibited by the lower friction and damage absorption characteristics of carbon material. The goal of the present study is to improve the damage and wear resistance of alumina ceramics by the addition of CNT, considering only the content of CNT in the composites. The hardness and toughness of Al_2O_3-CNT nanocomposites were also affected by CNT contents, which, in turn, influenced the wear characteristics of the composites. In addition, the effect of CNT reinforcement on the mechanical properties of yttria-stabilized zirconia (YSZ) and hydroxyapatite has been investigated.

10:10 AM

(ICACC-S11-016-2018) Production Root Technology has Another ACE (Applicability, Cost efficiency, and Environmental friendly) (Invited)

K. Yasuda*[1]

1. Tokyo Institute of Technology, Japan

For several years in this symposium, we discuss the concept of "Production Root Technology", and found many success stories and noteworthy examples, in which we recognize transformation from 3D (dangerous, dirty, and difficult) aspects into ACE (automatic, clean, and easy) ones. But the present author thinks that another ACE is also needed for production root technology. That is, "(Wide) Applicability", "Cost efficiency", and "Environmental friendly" for the next generation ceramic industry. For example, if we develop an advanced production route, it should have these ACEs. Or, if we adopt an reliability estimation method, it also should have the ACEs. In this presentation, the present author introduces an example of extension of Weibull statistics from dense ceramics to porous ceramics from the viewpoint of wide applicability, cost efficiency, and environmental friendly.

10:30 AM

(ICACC-S11-017-2018) Interfacial adhesion between carbon fiber and thermoplastics effected for appropriate/sufficient mechanical properties of CFRTP (Invited)

T. Iirisawa*[1]; K. Ujihara[1]; S. Kobayashi[1]; Y. Tanabe[1]

1. Nagoya University, Japan

CFRPs is an excellent material and the demands for the latest automotive of them are considered to be increasing for its lightweight. Especially, carbon fiber reinforced thermoplastics (CFRTPs) have been expected for their low cost, high productivity. Therefore, CFRTPs made with commodity plastics have been developed worldwide. Improvement of interfacial shear strength (IFSS) between carbon fibers (CFs) and thermoplastics has been focused for development of CFRTP having high mechanical properties. It is known that the surface of CFs has the oxygen-containing functional groups by surface treatment such as electrolytic oxidation process, and that these functional groups form covalent bonds to epoxy resin at the time of traditional CFRP molding, which causes high IFSS. On the other hand, it has not been clear that the functional groups efficiently work for the IFSS between CFs and various thermoplastics. In this study, PA6 were chosen as matrix polymer for CFRTPs, because PA6 have been expected as matrix polymer for automotive application in terms of the balance of properties and cost. Each CFs with/without oxygen-containing functional groups on the surface were prepared, and difference of IFSS between each CFs and PA6 was discussed. The mechanical properties of CFRTP made with each CFs and PA6 were also evaluated.

10:50 AM

(ICACC-S11-018-2018) Current Status of ISO/TC206 Fine Ceramics (Invited)

S. Sakaguchi*[1]

1. AIST, Japan

ISO/TC206 is a technical committee (TC) in ISO, which deals with the ISO standards relating to fine (advanced) ceramics. This technical committee was established in 1992, and the first plenary meeting was held in 1994. After more than 20 years of the activities of this TC, we already have around 100 ISO published standards,

and still around 40 new work items under development. For the discussion of the standards for this field, we have 12 working groups in our TC. They are (1) terminology/classification, (2) powders, (3) chemical analysis, (4) composites, (5) porous ceramics, (6) monolithic ceramics - mechanical properties, (7) monolithic ceramics - physical and thermal properties, (8) joining, (9) photocatalysis, (10) coatings, (11) electrical and optical applications, (12) engineering applications. In the beginning of this TC, we had work items for fundamental properties on ceramics, such as strength, hardness, thermal expansion etc., but already the discussing items tends to shift to some focused applications, such as bearing balls, photocatalytic materials, piezoelectric materials etc. As the presenter is in charge of the secretary of this TC, current status of this TC will be explained in the presentation.

11:10 AM

(ICACC-S11-019-2018) Innovative Technology for ^{6}Li Enrichment using Electrodialysis with Lithium Ionic Superconductor (Invited)

T. Hoshino*[1]

1. National Institutes for Quantum and Radiological Science and Technology (QST), Breeding Functional Materials Development Group, Department of Blanket Systems Research, Rokkasho Fusion Institute, Fusion Energy Research and Development Directorate, Japan

Tritium needed as a fuel for fusion reactors is produced via neutron capture by lithium-6 (^{6}Li). However, natural Li contains only about 7.8% ^{6}Li, and enrichment of ^{6}Li up to 90% is required for adequate tritium breeding in fusion reactors. In Japan, lithium isotope enrichment methods have been developed to avoid the environmental hazards of using mercury. However, the isotope separation coefficient and efficiency is too low to meet the practical need of large mass production of ^{6}Li. Therefore, new Li isotope separation technique using a Li ionic superconductor functioning as a Li isotope separation membrane (LISM) have been developed. First of all, I investigated the ionic mobility of lithium isotopes in ionic superconductor. Combing the first principle and the kinetics Monte Calro simulation, I calculate the diffusion constant of ^{6}Li and ^{7}Li. Furthermore, examinations of Li isotope separation using LISM with electrodialysis ware performed. Because the mobility of ^{6}Li ions is higher than that of ^{7}Li ions, ^{6}Li can be enriched on the cathode side of a cell. Using $Li_{0.29}La_{0.57}TiO_3$ (LLTO) as the Li ionic superconductor was prepared. After electrodialysis, I obtained a maximum of 1.04 for the ^{6}Li isotope separation coefficient. This result showed that the ^{6}Li isotope separation coefficient of this method is the same as that of the amalgamation process using mercury (1.06).

11:30 AM

(ICACC-S11-008-2018) Observation of internal structure of ceramic slurry, green body and sintered body by optical coherence tomography (Invited)

J. Tatami*[1]; T. Takahashi[2]

1. Yokohama National University, Japan
2. Kanagawa Institute of Industrial Science and Technology, Japan

It is well-known that the properties of ceramics strongly depend on their internal structure, which is changed during the fabrication process. Optical coherence tomography (OCT) is the novel technique to be able to obtain the image of the internal structure of opaque materials rapidly and 3-dimensionally, which has been developed in the field of medical application. In this study, the OCT was applied to observe internal structure of ceramic slurry, green and sintered body. Brownian motion of Si_3N_4 particles in the slurry and anisotopic drying of the slurry were observed in real time. Internal structure of the green body prepared by dry pressing resulted from the used granules. Artificially induced pores and cracks in the Al_2O_3 sintered body were clearly found by OCT observation.

S12: Advanced MAX/mxene Phases and UHTC Materials for Extreme and High Temperature Environment

MAB Phases and Next Generation Development in Designing of MAX Phases

Room: Tomoka B

Session Chairs: Babak Anasori, Drexel University; Leszek Chlubny, AGH-University of Science and Technology

8:30 AM

(ICACC-S12-012-2018) Ti_3AuC_2, $Ti_3Au_2C_2$ and Ti_3IrC_2 by noble-metal substitution reaction in Ti_3SiC_2 (Invited)

P. Eklund*[1]

1. Linkoping University, Dept. of Physics, Chemistry, and Biology, Sweden

The large class of layered ceramics encompasses both van der Waals (vdW) and non-vdW solids. While intercalation of noble metals in vdW solids is known, formation of compounds by incorporation of noble-metal layers in non-vdW layered solids is largely unexplored. The MAX phases constitute an important model system for this general research question. Here, I will present our recent demonstration [Fashandi, …, Eklund, Nature Materials 16 814 2017] of formation of Ti_3AuC_2 and $Ti_3Au_2C_2$ phases by a substitutional solid-state reaction of Au into Ti_3SiC_2 single-crystal thin films with simultaneous out-diffusion of Si and discuss the formation mechanism. Ti_3IrC_2 is subsequently produced by a substitution reaction of Ir for Au in $Ti_3Au_2C_2$. These phases form Ohmic electrical contacts to SiC and remain stable after 1000 h of ageing at 600 C in air. We have also demonstrated that the same phenomenon occurs in several other MAX phases including Ti2AlC and Ti3AlC2. These results, by combined analytical electron microscopy and ab initio calculations, open avenues for processing of noble-metal-containing layered ceramics that have not been synthesized from elemental sources, along with tunable properties such as stable electrical contacts for high-temperature power electronics or gas sensors.

9:00 AM

(ICACC-S12-013-2018) Processing and Characterization of $Ti_2(Al_{1-x},Bi_x)C$ Solid Solutions

E. Prehn*[1]; Z. Tan[1]; T. Duong[1]; R. Arroyave[1]; M. Radovic[1]

1. Texas A&M University, MSEN, USA

Herein we report for the first time on processing and structural parameters of one of the MAX phase solid solution systems, namely $Ti_2(Al_{1-x},Bi_x)C$. This system is particularly interesting because (a) Bi has significantly higher atomic radius than Al; and (b) our previous cluster expansion calculations suggest that those solids solutions would be stable, regardless of the fact that one of the end members, namely Ti_2BiC does not exist. A series of $Ti_2(Al_xBi_{1-x})C$ were high-throughput synthesized using Pulsed Electric Current Sintering (PECS), and the phase composition and structure of the sintered solid solutions was characterized using Scanning Electron Microscopy with Energy Dispersion Spectroscopy (EDS) and X-Ray Diffraction. Results indicate that stable solid solutions with at least 33.3% bismuth on the A-site can be synthesized. Furthermore, A-site vacancy formation is noticed in samples with higher (≥ 17%) bismuth substitution on A site. It was also found that substitution of Al with Bi results in the significant increase of lattice parameter a and only moderate changes in lattice parameter c. Effects of increasing amount of Bi in $Ti2(Al_{1-x},Bi_x)C$ on elastic properties, hardness, and electrical conductivity are also discussed in detail.

9:20 AM

(ICACC-S12-014-2018) Synthesis and Characterization of the Ternary, Nanolaminated Boride: Cr_2AlB_2

S. Kota*[1]; W. Wang[1]; J. Lu[3]; O. Chaix-Pluchery[2]; G. Ying[1]; L. Hultman[3]; S. May[1]; M. Barsoum[1]

1. Drexel University, Materials Science and Engineering, USA
2. Université Grenoble-Alpes, CNRS, LMGP, France
3. Linköping University, The Department of Physics, Chemistry and Biology, Sweden

Cr_2AlB_2 is an atomically laminated compound comprised of Cr_2B_2 slabs interleaved by single layers of Al and among the small family of similar layered borides known as the MAB phases. In this work, the effects of starting composition and synthesis temperature was investigated to make predominantly single-phase powders. Fundamental characterization of the structure, bonding, defects and possible formation mechanisms, was carried out using transmission electron microscopy and Raman spectroscopy. Magnetization measurements over 10-350 K are consistent with paramagnetism. The structure and properties observed are compared to those of the corresponding chromium monoboride, CrB.

9:40 AM

(ICACC-S12-015-2018) Synthesis and Characterization of Novel Ni-MAB composites

M. Fuka*[1]; M. Dey[1]; S. Gupta[1]

1. University of North Dakota, Mechanical Engineering, USA

$M_{n+1}AX_n$ (MAX) phases (over 70+ phases), where n =1,2,3; M is an Early Transitional Metal, A is a Group A element (mostly groups 13 and 14); and X is C and/or N, are novel nanolaminate ternary carbides and nitrides, They crystallize in the space group of $P6_3/mmc$. It is also well known that these solids are bestowed with excellent properties like damage tolerance, thermal shock resistance and machinability. However these solids do not have a ternary composition where X = B. Recently, single crystals of $Cr_2AlB_2(CrB_2)_x$ (x = 0, 1, 2), M_2AlB_2 (M = Cr, Mn, Fe), and MAlB (M = Mo, W), and due to their similarity with MAX Phases - these novel ternary solids are classfied as "MAB-phases". In this presentation, we will report the synthesis and characterization of novel structural Ni-MAB composites. There has been very limited or no studies on these novel composite systems. .

Novel Applications and Processing Methods for Synthesizing MAX Phases III

Room: Tomoka B

Session Chairs: Babak Anasori, Drexel University; Leszek Chlubny, AGH-University of Science and Technology

10:20 AM

(ICACC-S12-017-2018) Probing the local atomic structure in MAX phases and MXenes using EELS and DFT simulations: From solid solution effects to surface functionalization (Invited)

V. Mauchamp*[1]; D. Magné[2]; M. Nechiche[3]; P. Chartier[1]; V. Gauthier[1]; S. Celerier[4]; S. Dubois[1]; T. Cabioch[1]

1. Institut PPRIME, Physics and Mechanics of Materials, France
2. Groupe de physique des Matériaux, France
3. Université Mouloud Mamerri, Algeria
4. Institut de Chimie des Milieux et Matériaux de Poitiers, France

Electron Energy Loss Spectroscopy (EELS) in the Transmission Electron Microscope (TEM) is a very powerful nanometer scale probe of the local order in complex materials. Through the analysis of core electrons excitations, EELS combined with density functional theory simulations provide information on the local organization around the investigated atoms over distances up to about 1 nm: being chemical sensitive and closely related to the electronic structure of the material, it is very complementary to diffraction techniques for materials analysis. In this presentation, I will illustrate the benefit of combining EELS with DFT simulations for the characterization of MAX phases and MXenes by focusing on two important issues. First, the characterization of solid solution effects in MAX phases will be addressed through the example of copper substitution in Ti_3AlC_2: particular attention will be paid to the determination of the substitution sites and corresponding hybridizations. Second, the surface functionalization of $Ti_{n+1}C_nT_x$ MXene (T ≡ -O, -OH and –F) will be discussed with particular emphasis on the determination of the surface groups localization and their chemical nature. In both cases, the structural information will be correlated to the electronic properties (e.g. optical or transport properties) of these materials.

10:50 AM

(ICACC-S12-018-2018) Anisotropic properties of MAX phase single crystals

T. Ouisse*[1]; D. Pinek[1]; I. Gélard[1]; L. Shi[2]; B. Hackens[2]; T. Ito[3]; T. Fujita[3]; f. Bourdarot[4]; P. Bourges[5]; P. Piekarz[6]

1. Grenoble INP, France
2. UCLouvain, Belgium
3. Nagoya University, Japan
4. CEA, INAC, France
5. CEA Saclay, LLB, France
6. Institute of Physics Polish Academy of Sciences, Poland

MAX phases combine interesting properties of ceramics and metals. The common point of these materials is to share a highly anisotropic crystal structure, which is put to good use to produce a new family of two-dimensional materials named MXenes. However, until recently, the main synthesis processes resulted in highly polycrystalline materials, making difficult a quantitative assessment of the expected physical anisotropies. High temperature solution growth allows us to obtain single crystals of macroscopic size. Therefore, we can directly assess their physical anisotropies. Here we wish to review the results we obtained so far. They include magneto-transport: We evidence very high ratios between the in-plane and out-of-plane resistivity of some compounds. We also use Angle Resolved Photoemission Spectroscopy to measure the complex Fermi Surfaces (FS) of MAX phase single crystals, and confirm the quasi 2D character of the investigated phases (we demonstrate, e.g., that in Cr_2AlC both electron and hole FS's form bulged open tubes with an axis directed parallel to c). Eventually, single crystals of suitable dimensions (areas exceeding several cm^2) allow us to measure neutron inelastic scattering and to recover the phonon dispersion branches along the main crystallographic directions.

11:10 AM

(ICACC-S12-019-2018) Synthesis, Characterization, and Bonding of Ti_3SiC_2 through Spark Plasma Sintering, Additive Manufacturing, and Cold Spray

E. Faierson*[1]; V. Ageh[2]; T. Scharf[2]

1. Quad City Manufacturing Lab-Western Illinois University, USA
2. University of North Texas, USA

MAX phase materials exhibit unique mechanical, thermal, and electrical properties. Their material properties can be similar to both that of metals and ceramics in certain circumstances. Ti_3SiC_2 is a MAX phase material with a low density, high Young's modulus and damage tolerance, as well as good machinability. It also retains its strength at high temperatures, and has been shown to be able to fully recover from high loading, while dissipating a significant amount of the loading energy. Within this study, a planetary ball mill was used to mill titanium, silicon carbide, and carbon powders. The milled powder was then processed through Spark Plasma Sintering. Methods of bonding Ti_3SiC_2 to metals and ceramics such as titanium alloys, boron carbide, and silicon carbide were explored using spark plasma sintering, additive manufacturing, and cold spray processes. Xray diffraction was used to perform bulk phase analysis of the synthesized specimens. Microstructural and mechanical properties

were evaluated using optical microscopy, SEM/EBSD, densimetry, hardness, and bend testing.

11:30 AM

(ICACC-S12-020-2018) Design of Novel Ni-Ti_3SiC_2 based Multilayered Composites

Q. Tran*[1]; M. Fuka[1]; M. Dey[1]; S. Gupta[1]

1. University of North Dakota, Mechanical Engineering, USA

It is well known that $M_{n+1}AX_n$ (MAX) phases (over 60+ phases) are thermodynamically stable nanolaminates. They have fascitinating properties like damage tolerance, thermal shock resistance, machinability, and low Vickers hardness values of 2–8 GPa., During mechanical testing, these solids display nonlinear, hysteretic, elastic behavior due to kink band formation in the basal planes. Recently, we showed that lower concentrations of MAX Phases (< 30 vol%) can also reinforce metal matrix and improve its tribological behavior. We have coined the term MAX Reinforced Metals (MRMs) for these composites. In this study, we will explore the effect of laminate design on the mechanical and tribological behavior of MRMs by further exploring the Ni-Ti3SiC2 system system as Ni is a technologically important material.

S13: Advanced Ceramics and Composites for Nuclear Fission and Fusion Energy

ATF and Radiation Effects

Room: Coquina Salon H

Session Chairs: Yutai Katoh, Oak Ridge National Laboratory; Peng Xu, Westinghouse Electric Company

8:30 AM

(ICACC-S13-011-2018) Improving the accident-tolerance of Zircaloy cladding by integrated gradient ceramic coatings (Invited)

J. Zhang*[1]; Y. Lei[1]; L. Chen[1]; J. Wang[1]

1. Institute of Metal Research, Chinese Academy of Sciences, High-performance Ceramics, China

The development of accident-tolerant fuel/clad system during Loss-of-coolant accident (LOCA) has been considered as challenges of light water reactors (LWR) after the Fukushima Dai-ichi accident. To enhance the accident-tolerance of the nuclear system, one strategy is modification of Zircaloy cladding surface with advanced ceramic coatings. MAX phases display a high resistance to oxidation and ion irradiation from which a promising coating materials. Integrated gradient MAX coatings with optimal irradiation resistance, steam oxidation resistance as well as CTE match were synthesized by PVD method with temperature friendly to Zircaloy cladding. The mechanical and chemical compatibility between integrated coating and substrate in the as-deposited state as well as under LOCA scenario were investigated. Finally, the HT steam oxidation tests were performed to evaluate the feasibility of integrated ceramic coatings in accident-tolerant fuel/clad system.

9:00 AM

(ICACC-S13-012-2018) Evaluation of seal-coated SiC ceramics and composites after neutron irradiation at LWR-relevant temperatures

C. Ang*[1]; Y. Katoh[1]; T. Koyanagi[1]; K. Linton[1]; K. Terrani[1]; D. Carpenter[2]; G. Kohse[2]; L. Snead[2]

1. Oak Ridge National Laboratory, USA
2. Massachusetts Institute of Technology, USA

The radiolytically assisted hydrothermal corrosion and the probabilistic compromise of fission product gas containment are the most critical technical feasibility issues for silicon carbide (SiC) composite-based cladding as the enhanced accident tolerance fuel technology for light water reactors (LWRs). Dual-purpose coating, that provides both the corrosion barrier and the hermetic seal functions, is among the leading mitigation technologies for such issues. In the present work, a variety of developmental dual-purpose coatings applied SiC ceramic and composite test articles were examined following neutron irradiation in the MIT Reactor at the LWR-relevant temperatures. Results are reported with the main focus on structural integrity due to the differential irradiation strain between the coating materials and the SiC substrate. This research was sponsored by the Advanced Fuels Campaign, Nuclear Technology Research and Development Program, Office of Nuclear Energy, United States Department of Energy under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

9:20 AM

(ICACC-S13-013-2018) Non-Destructive Evaluation of Sealed SiC-SiC Composite Cladding Structures using X-Ray Computed Tomography, Helium Pycnometry and Permeability Testing

J. Sheeder*[1]; G. Jacobsen[1]; H. Khalifa[1]; C. P. Shih[1]; E. Song[1]; C. Deck[1]

1. General Atomics, USA

As advancements are made in the fabrication of silicon carbide fiber reinforced silicon carbide matrix (SiC-SiC) composites toward the production of accident tolerant fuel rods, non-destructive evaluation methods must be used as a quality control method to verify that cladding samples meet demanding dimensional and permeability specifications. This work describes the characterization of the composite structure and endplug seal of SiC-SiC rodlets using X-Ray Computed Tomography (X-Ray CT), helium pycnometry and permeability testing. The samples have been sealed with an internal helium back pressure similar to what would be required for fuel rods going into a light water reactor. The ultimate goal of this effort is to validate the fabrication methods used in making sealed SiC-SiC parts and to show that sealed composite samples can be produced consistently. The combined information from the three measurement techniques provide assurance that the SiC-SiC composite cladding samples are impermeable and suitable for reactor insertion. *This work is supported by General Atomics internal funding and US DOE under contract number DE-NE0008222.

9:40 AM

(ICACC-S13-014-2018) Performance of silicon carbide plasma facing coatings under fusion conditions in DIII-D*

S. Gonderman*[1]; H. Khalifa[1]; G. Vasudevamurthy[1]; J. Zhang[1]; T. Abrams[1]; S. Bringuier[1]; D. Thomas[1]; L. Holland[1]; D. Rudakov[2]; A. Briesemeister[3]

1. General Atomics, USA
2. UCSD, USA
3. Oak Ridge National Lab, USA

Silicon carbide (SiC) has garnered interest as a material for fusion energy due to its excellent mechanical properties both at high temperature and under neutron irradiation. To further investigate the response of SiC as a plasma facing component (PFC), chemical vapor deposition SiC coatings (~270 μm) have been applied to graphite samples which were then exposed in the DIII-D tokamak via the DiMES system, a removable sample exposure probe. These SiC coatings were exposed to plasma bombardment with steady-state heat fluxes up to 3 MW m^{-2} and transient loads typically peaking at ~10 MW m^{-2}. Spectral analysis of the Si impurity influx was monitored to quantify erosion rates of the SiC coating using the S/XB method. This was compared to pre and post exposure, x-ray computed tomography imaging of the DiMES sample and correlated with erosion measurements of the surface. Additional SEM imaging showed pronounced features at locations of initial coating imperfections indicating a correlation between coating quality and surface response. SEM analysis indicated some cracking was present on the SiC surface but these cracks did not widen or elongate due to plasma bombardment. *Preparation and analysis of the samples supported

by General Atomics Corporate Funds, testing in the DIII-D Tokamak supported by US DOE under DE-FC02-04ER54698.

10:20 AM

(ICACC-S13-015-2018) Status Update on Westinghouse EnCore™ SiC/SiC Composite Cladding Development (Invited)

P. Xu*[1]; E. J. Lahoda[1]; F. Boylan[1]; R. L. Oelrich[1]

1. Westinghouse Electric Company, USA

SiC/SiC composite cladding is a game-changing accident tolerant fuel (ATF) technology and a key component of Westinghouse's new EnCore™ fuel. The lead test assembly (LTA) for EnCore™ SiC/SiC composite cladding is planned for 2021. To meet this aggressive schedule, significant progress was made to advance the SiC technology, and various developmental activities will be reported here. The water corrosion resistance was improved to meet the hermeticity and coolant chemistry requirements. Various exploratory tests were conducted to understand the effect of surface roughness on corrosion, rod loading, grid-to-rod fretting, pressure drop and heat transfer. Four point bend tests and grid impact tests are being performed to evaluate the response of SiC cladding to seismic loads and hermeticity retention. The SiC tubing specification is being finalized. Test reactor irradiation tests at the MIT reactor, the Advanced Test Reactor (ATR) and the Halden Reactor have been planned. Previous MIT reactor test results will be discussed.

10:50 AM

(ICACC-S13-016-2018) Thermal Hydraulic and Neutronic Analysis of a SiC/SiC Channel Box (Invited)

J. Gorton[1]; N. R. Brown*[1]; G. Singh[2]; K. Terrani[2]; Y. Katoh[2]; B. Wirth[3]

1. Pennsylvania State University, Mechanical and Nuclear Engineering, USA
2. Oak Ridge National Laboratory, USA
3. University of Tennessee, Nuclear Engineering, USA

Silicon carbide fiber/silicon carbide matrix (SiC/SiC) composite channel boxes are being considered as a potential component in a boiling water reactor (BWR). Temperature and neutron flux boundary conditions have been generated for planned 3D thermo-mechanical analysis of a SiC/SiC based channel box in a BWR. Under differential temperature and irradiation, SiC-based materials swell based on the local conditions, so the objective is to use some simplified boundary conditions to predict the dimensional change of a channel box during a typical reactor cycle. The goal of this model is to accurately calculate thermal and neutronic boundary conditions for the surface temperature and flux of a silicon carbide fuel bundle channel box in a GE14-like BWR lattice. Reasonable inputs from a variety of sources in the open literature were used to assembly GE14-like data. The Consortium for Advanced Simulation of Light Water Reactors thermal hydraulic analysis tool CTF and the neutronics tool Serpent were used to generate the boundary conditions. The sensitivity to direct energy deposition from neutrons and gamma rays in the coolant and the channel box was also investigated.

11:20 AM

(ICACC-S13-017-2018) Thermo-mechanical Parametric Evaluation of SiC/SiC Cladding with Fuel Creep

G. Singh*[1]; R. Sweet[2]; B. Wirth[2]; K. Terrani[1]; Y. Katoh[1]

1. Oak Ridge National Lab, USA
2. University of Tennessee, Department of Nuclear Engineering, USA

SiC/SiC cladding is one of the leading concepts being considered for the accident tolerant fuel-cladding systems. Currently there is not good understanding of the creep behavior of UO2 fuel. A thermo-mechanical analysis of the cladding was performed to evaluate the effect of fuel creep on the stresses, temperature distribution and displacements of the SiC/SiC cladding. Other important parameters such as linear heat rate and initial fuel pellet-cladding gap thickness were also considered in the analysis. State of the art properties of SiC/SiC composite were employed to accurately determine the thermo-mechanical behavior of the cladding.

11:40 AM

(ICACC-S13-018-2018) Fabrication and Performance of Engineered SiC-SiC Accident Tolerant Fuel Cladding

C. Deck*[1]; H. Khalifa[1]; G. Jacobsen[1]; J. Sheeder[1]; J. Zhang[1]; C. Bacalski[1]; G. Vasudevamurthy[1]; C. P. Shih[1]; S. Oswald[1]; K. Shapovalov[1]; E. Song[1]; J. Stone[1]; R. Haefelfinger[1]; R. Jacko[2]; C. A. Back[1]

1. General Atomics, USA
2. Westinghouse Electric Company LLC, USA

High purity silicon carbide (SiC) and SiC fiber reinforced, SiC-SiC composites offer high temperature strength and exhibit stable behavior under neutron irradiation, with minimal dimensional and mechanical property changes under reactor-relevant conditions. General Atomics has developed a multi-layered engineered cladding design which couples the advantages of monolithic SiC with SiC-SiC composite to offer a combination of toughness, hermeticity, and enhanced performance in accident conditions. Fabrication of cladding to meet demanding dimensional, uniformity, and structural requirements is essential, and manufacturability was demonstrated through repeatable production of sealed and open ended tubes in batches with high yield. Behavior of this multi-layered cladding structure was quantified through mechanical and thermal characterization, and performance was assessed by measuring permeability, corrosion, and the response to simulated severe accident conditions. To further demonstrate this accident tolerant fuel, rodlets were fabricated consisting of SiC-SiC engineered composite cladding with uranium silicide fuel pellets hermetically sealed inside. These will be irradiated in the Advanced Test Reactor at INL, and the differences between the anticipated irradiation conditions for this test and those conditions expected for normal operation in a pressurized water reactor will be discussed.

S14: Crystalline Materials for Electrical, Optical and Medical Applications

Semiconductor

Room: Tomoka C

Session Chairs: Yoshihiko Imanaka, Fujitsu Laboratories Ltd.; Rafael Jaramillo, Massachusetts Institute of Technology

8:30 AM

(ICACC-S14-009-2018) Development of Perovskite Solar Cells Inspired by Ceramic Processing (Invited)

Y. Suzuki*[1]

1. University of Tsukuba, Faculty of Pure and Applied Sciences, Japan

Perovskite solar cells (PSCs) were first reported by Kojima et al. with a power conversion efficiency (PCE) of 3.81% in 2009. Recently, the PCE of PSCs has been much improved to 22.1%, and thus PSCs are focused all over the world. There exists some problems of PSCs, e.g. (1) expensive organic hole transportation material, (2) lead (Pb) in the active layer, and (3) insufficient reactions between starting materials. Here we introduce our recent development of PSCs to solve these problems: (1) An organic material, (spiro-OMeTAD), is generally used as a hole conductor, but spiro-OMeTAD is much more expensive than other materials used in PSCs. We have prepared PSCs with a cost-effective CuI hole transport layer by spin coating. The CuI-based PSC recorded PCE of 6.52% (max) after the 20 days. (2) Methylammonium bismuth iodide, $(CH_3NH_3)_3Bi_2I_9$, is a promising lead-free perovskite active layer. By using gas-assisted deposition method, we have successfully prepared dense and smooth $(CH_3NH_3)_3Bi_2I_9$ active layer. (3) A new 3-step method for the active layer has been developed, based on the 2-step method

with an additional spin-coating of $CH_3NH_3(I,Br)$ solution on the $CH_3NH_3PbI_3$ film to scavenge remnant PbI_2. The 3-step method improved light absorption of the film by converting the residual PbI_2 into $CH_3NH_3PbI_{3-x}Br_x$. The PCE was improved from 12.9 % (2-step) to 14.4 % (3-step).

9:00 AM

(ICACC-S14-010-2018) Plasmon sensitized silicon nanowires/ titanium dioxide bulk heterojunction solar cells

D. Banerjee*[1]; J. Benavides[1]; S. G. Cloutier[1]

1. École de technologie supérieure (ÉTS), Electrical Engineering, Canada

Vertically aligned silicon nanowires are potential candidates for low-cost solar cells since past decade. Silver plasmon sensitized free standing n-type silicon nanowires and titanium dioxide heterojunction is reported. The optical properties of the heterostructure are significantly influenced by the surface plasmons of silver nanoparticles. A promising light trapping mechanism for these plasmon sensitized nanowires are discussed.

9:20 AM

(ICACC-S14-011-2018) Processing of particulate photocatalysts into sheets for efficient and scalable sunlight-driven water splitting (Invited)

T. Hisatomi*[1]; K. Domen[1]

1. The University of Tokyo, Department of Chemical System Engineering, Japan

Sunlight-driven water splitting employing particulate semiconductor photocatalysts has attracted much attention as a means of renewable solar hydrogen production. A solar-to-hydrogen energy conversion efficiency (STH) of 5% or higher is considered to be necessary for practical photocatalytic solar hydrogen production. It is therefore necessary to activate and stabilize visible-light-driven photocatalysts. Scalability of the system is also a critical issue. Water splitting systems based on particulate photocatalysts are promising in terms of scalability because they do not need any secure electric circuits and thus can be spread over a wide area easily. The author's group has recently studied photocatalyst sheets based on particulate hydrogen evolution photocatalysts (HEPs) and oxygen evolution photocatalysts (OEPs) embedded into a conductive layer by particle transfer. The STH of water splitting exceeds 1.1% when photocatalyst sheets composed of La- and Rh-codoped $SrTiO_3$ as a HEP and Mo-doped $BiVO_4$ as an OEP embedded into a gold thin layer are employed. The water-splitting activity of the photocatalyst sheet based on a gold conductor decreases with increasing the reaction pressure owing to the oxygen reduction reaction. However, the use of carbon as a conductive layer allows the photocatalyst sheet to maintain the water splitting activity at ambient pressure.

10:10 AM

(ICACC-S14-012-2018) Artificial photosynthesis anode composed of nano particulate photocatalyst film using nanoparticle deposition (Invited)

Y. Imanaka*[1]; T. Manabe[1]; H. Amada[1]; T. Anazawa[1]

1. Fujitsu Laboratories Ltd., Japan

Artificial photosynthesis technology known as Honda-Fujishima effect which produces oxygen and hydrogen or organic energy from sunlight, water, and carbon dioxide, is an ultimate energy and environmental technology. The key device for the higher efficiency of this reaction system is the anode electrode, generally composed of a photocatalyst formed on glass substrate with an electrically conductive Fluorine doped Tin Oxide (FTO). To obtain the highly efficient electrode, the dense film composed of nano particulate visible light responsible photocatalyst having usually complicated multi-elements composition need to be deposited and adhered on the FTO. In this study, we discovered a method capable of controlling electronic structures of a film by controlling the aerosol-type nano particle deposition (NPD) condition and thereby forming films of materials with a smaller band gap than prepared raw material powder, and succeeded in extracting a higher current from the anode electrode. As a result, we confirmed that a current approximately 100 times larger than currents produced by conventional processes could be obtained using the same material. This effect can be expected not only from the materials discussed (GaN-ZnO) in this paper but also from any photocatalyst, particularly materials of solid solution compositions.

10:40 AM

(ICACC-S14-013-2018) Importance of thermal processing and defect engineering on the doped TiO_2 photocatalyst: Combination of DFT calculations and experiments

H. Choi*[1]

1. Virtual Lab Inc., Republic of Korea

Inspite of a huge number of reports on the excellent photocatalytic activities of doped TiO_2 particle, such superior photocatalyst products have been hardly commercialized. One of the most important reason for the retarded mass-productions and commercializations of the materials is the significantly varying properties and performances, depending on the thermal processing conditions. It is necessary to find the origins of fluctuating optical properties and photocatalytic activities of doped TiO_2 particles. Mostly, the large variations in the optical properties of wide band gap oxides are from the formations of point defects, which can induce in-gap levels. Therefore, it is important to obtain the defect formation energies and understand role of each defects on the optical and photochemical properties. However, it is impossible to find the dominant point defects within doped oxides due to the limitation of existing measurement technology. Density functional theory (DFT) calculations with hybrid functionals have been successfully employed to calculate accurate point defect formation energies and their effects on the electronic structures. In this talk, we introduce some of our recent works: DFT-guided defect engineering toward strong photocatalysis of TiO_2 photocatalyst particle.

11:00 AM

(ICACC-S14-014-2018) Persistent photoconductivity due to hole-hole correlation in sulfide semiconductors, with applications to neuromorphic computing and chemical sensors (Invited)

R. Jaramillo*[1]

1. Massachusetts Institute of Technology, USA

Persistent photoconductivity (PPC) is due to the trapping of minority carriers at defects. PPC in chalcogenide semiconductors is relevant to the operation of technologies including flame detectors and thin film solar cells. Theory has suggested that anion vacancies are responsible for PPC due to negative-U behavior, whereby two minority carriers become trapped by a lattice relaxation. We provide experimental support for this model of PPC in CdS. We can vary the photoconductivity of CdS films over nine orders of magnitude by controlling the activities of Cd^{2+} and S^{2-} ions during chemical bath deposition. We suggest a screening method to identify other materials with long-lived, metastable states based on the results of ground-state calculations of atomic rearrangements following defect redox reactions. We demonstrate that this control approach can be extended to other chalcogenide semiconductors, and we discuss results that speak to the usefulness of this control in two classes of technology: neuromorphic computing and chemical sensing. PPC can enable artificial synaptic behavior by using optical transitions to excite and inhibit synaptic weights. PPC can also enable a type of chemical sensor in which giant photoconductivity is modified by oxidation-reduction reactions with adjacent chemical analytes.

11:30 AM

(ICACC-S14-015-2018) GaN based Hydrogen Sensor for High Temperature and Humid Ambient Sensing

S. Jung[3]; H. Kim[3]; K. Baik[1]; F. Ren[2]; S. Pearton[2]; S. Jang*[3]

1. Hongik University, Republic of Korea
2. University of Florida, USA
3. Dankook University, Republic of Korea

GaN based materials including AlGaN is well-suited to hydrogen sensing due to its wide bandgap and low intrinsic carrier concentration for high temperature operation, and mechanical and chemical robustness for device reliability. Among the many types of devices based on the GaN, AlGaN/GaN high electron mobility transistor (HEMT) structure with a 2 dimensional electron gas (2DEG) channel induced by piezoelectric and spontaneous polarization at AlGaN/GaN interface shows high sensitivity to change in surface charge created by catalytic reaction of Pt with hydrogen. For the Pt-AlGaN/GaN heterostructure Schottky diode, hydrogen molecules dissociate to atomic form on the Pt surface, which leads to an adsorbed dipole layer at the Pt/AlGaN interface. As a result, the Schottky barrier height of the diode is reduced, increasing a diode current with the concentration of the exposed hydrogen gas. One of issues with semiconductor based hydrogen sensors is that their sensitivity is significantly degraded in humid ambient. Water molecules block the catalytically active sites of the Pt, resulting in the significant reduction in detection signal. In this study, we demonstrate that the water-blocking layer effectively prevents the sensor from any reduction in hydrogen detection current in the presence of moisture and that the devices show excellent the hydrogen sensitivity up to 300°C without any change in characteristics.

S15: Additive Manufacturing and 3-D Printing Technologies

Selective Laser Sintering

Room: Coquina Salon B

Session Chair: Jens Guenster, BAM Federal Institute for Materials Research and Testing

9:00 AM

(ICACC-S15-010-2018) Laser Shock Processing of Structural Ceramics

B. Cui*[1]; F. Wang[1]; X. Yan[1]; S. Sun[2]; L. Deng[2]; Y. Lu[2]; M. Nastasi[1]

1. University of Nebraska, Lincoln, Mechanical & Materials Engineering, USA
2. University of Nebraska, Lincoln, Department of Electrical Engineering, USA

Laser shock processing (LSP) is a novel surface modification process which is traditionally applied to metals to prevent failures such as fatigue. This research has applied LSP to structural ceramics such as Al_2O_3 and Ti_2AlC, and aims to understand the fundamental processing-microstructure-property relationship of structural ceramics in this process. LSP utilizes high-energy laser pulses to irradiate ceramic surface to form a plasma. The explosive expansion of the plasma generates shock waves which penetrate into the bulk ceramic and induce significant compressive residual stresses which can extend to a depth of more than 1 mm from the surface of ceramics. The presence of compressive residual stresses improves the resistance of alumina ceramics to indentation cracking. Microstructural characterizations by transmission electron microscopy suggested that the mechanical response of alumina ceramics to LSP is that elastic deformation occurs in alumina grains while plastic deformation along grain boundaries. During LSP of ceramics at room temperature, microcracks may be induced by laser-driven shock waves, which will limit the further improvement of mechanical properties by LSP. Our recent research shows that by performing post-LSP annealing of α-Al_2O_3 ceramics, cracking healing may occur during the annealing process which can eliminate the surface flaws caused by LSP.

9:20 AM

(ICACC-S15-011-2018) Additive manufacturing of a metal to ceramic assembly

L. Ferrage*[1]; G. Bertrand[1]; P. Lenormand[1]

1. CIRIMAT, France

Selective laser sintering/melting (SLS/M) is the additive manufacturing technology chosen in the present work to elaborate a metal to ceramic assembly. If this process is currently well mastered for the production of metallic parts, the shaping of ceramic materials by means of direct SLS (without the use of a binder material) is more challenging. Indeed, most of the commercial SLS machines are equipped with a Nd:YAG laser emitting at λ=1.065 μm, which is a wavelength almost fully reflected by oxide ceramics. The first step of this experimental work was to optimize both the optical properties of the raw powder and the process parameters in order to successfully fabricate dense ceramic parts (tests were conducted on yttria stabilized zirconia). The second step consisted in the additive manufacturing of a bi-material part designed as a simple stacking of a metal (here aluminum) and a ceramic. It was found that an adequate combination of parameters permitted to shape yttria stabilized zirconia parts with a relative density of 96 %, with no need for thermal post-treatment. Experiments also demonstrated that it was possible to manufacture a metal to ceramic assembly. Its structure, microstructure and chemical composition were characterized and an emphasis was put on their evolution at the interface. A study of the mechanical properties provides additional insight on this assembly.

9:40 AM

(ICACC-S15-012-2018) Stereolithographic Additive Manufacturing of Ceramic Objects with Geometric Fluctuation to Control Fluid Phenomena

H. Nozaki*[1]; S. Kirihara[1]

1. Osaka University, Technology, Japan

Structural patterns in nature, for example angularities of quay walls or wind ripples formed by intermittent stimulations of water or air, have fluctuated vibrations of geometric profiles. Through Fourier transformation for the fluctuated vibrations, functional profiles of Power Spectral Density (PSD) adjusting the amplitudes toward the frequencies show inverse proportions defined as 1/F fluctuations. Through inverse Fourier transformation, fluctuated patterns can be introduced effectively into surfaces of designed solid models automatically. Geometrically modulated artifacts composed of alumina were reproduced by stereolithographic additive manufacturing. The ceramic particles were dispersed into photosensitive resin at 40 - 60 vol. % to create paste material. An ultra violet laser beam was scanned on the paste material spread by a knife edge to solidify a two dimensional cross section. Through the layer laminations, a three dimensional object with fluctuated patterns was fabricated. These created objects should be applied to fluid flows modulations of liquid or gas phases in the waterways or air duct. Furthermore, fluctuated patterns should be applied to control electric waves by structure properties in future.

Powder Bed Fusion

Room: Coquina Salon B
Session Chair: Bai Cui, University of Nebraska, Lincoln

10:20 AM

(ICACC-S15-013-2018) Powder-based Additive Manufacturing at Micro-Gravity (Invited)

J. Guenster*[3]; A. Zocca[3]; P. Lima[3]; J. Lüchtenborg[3]; T. Mühler[1]; M. Sparrenberg[2]; J. Melcher[2]

1. Clausthal University of Technology, Germany
2. Deutsches Zentrum für Luft- und Raumfahrt (DLR) - German Aerospace Center, Germany
3. Bundesanstalt für Materialforschung und –prüfung (BAM) - Federal Institute for Materials Research and Testing, Germany

Many of the most successful and precise additive manufacturing technologies are based on the deposition layer-by-layer of a flowable powder. Just to cite the most well-known technologies, the "powder-based three-dimensional printing" and the "selective laser sintering (SLS)" or "selective laser melting (SLM)" processes all share the same layer deposition method: A flowable powder is spread layer by layer, while the layer information of the part is scribed or printed after each consecutive layer deposition step. Besides outstanding improvements in the development of new technologies and new material systems, little progress has been made on the stabilization and densification of the deposited powder, the so called powder bed. Generally, layer deposition requires gravitational forces acting on each particle as a prerequisite for compaction of the particles and formation of a smooth layer. We have introduced an additional force acting on the particles by establishing a gas flow though the powder bed. The presentation will introduce the gas flow assisted powder deposition as a novel, easy and economic approach for the stabilization of powder beds, which allows the printing of parts without the use of support structures and at micro-gravity.

10:50 AM

(ICACC-S15-014-2018) Progress towards direct additive manufacturing of ceramics using laser beam melting

F. Petit*[1]; E. Juste[1]

1. Belgian Ceramic Research Centre, Belgium

For polymers and metals, additive manufacturing (AM) methods, i.e. layer-wise assembling of components from CAD data, are already well established and used for manifold applications. On the contrary, for ceramics, AM is still far from industrial maturity. Nevertheless it is gaining more and more importance, especially by the opportunities given in terms of designing extremely complex geometries and thanks to its tool-free shaping methodology. Processing of ceramics through additive manufacturing can be achieved by two main approaches: an indirect and a direct one. The indirect approach requires a post-thermal treatment to obtain a dense part. On the contrary, the direct approach allows to manufacture near net shape parts without post-treatments but highly suffers from the difficulty to get defect-free parts. In this lecture, some recent advances made towards obtaining ceramics parts using selective laser melting are reviewed. Emphasis is put on alumina and alumina/zirconia composites for which results are the most promising. It is shown that adding colloidal graphite to the ceramic powders improves significantly their processability with conventional LBM machines. The dramatic influence of layer thickness on the part density is demonstrated as well, pointing towards a more efficient route for spreading the powder than conventional wipers or rollers in powder-bed AM.

11:10 AM

(ICACC-S15-015-2018) 3D Printing of Ceramics: Disruptive innovation in materials and processes

R. Lenk*[1]

1. CeramTec GmbH, Germany

Digitalization offers new possibilities for product innovation due to new materials and processes which are available. The paper describes the history of additive manufacturing in the field of technical ceramics. Starting with last century a wide variety of concepts and processes was developed, whereas only few of them could be commerzialized in the past. Advantages and limitations of different manufacturing concepts are discussed. Both material properties and process capability are in focus of development. Deep material know how, technological reliability and an effective process control will be the guarantee for the long-term success in this new field of technology. In the paper recent results in material and process development based on 3D Printing from powder bed as well as from photosensitive suspensions are highlighted.

11:30 AM

(ICACC-S15-016-2018) Additive manufacturing of dense ceramics with Laser Induced Slip Casting (LIS)

J. Lüchtenborg*[1]; T. Mühler[2]; A. Zocca[1]; J. Guenster[1]

1. BAM Federal Institute for Materials Research and Testing, Ceramic Processing and Biomaterials, Germany
2. Clausthal University of Technology, Institute of Non-Metallic Materials, Germany

The possibility to produce dense monolithic ceramic parts with additive manufacturing is at the moment restricted to small parts with low wall thickness. Up to now, the additive manufacturing of voluminous ceramic parts is realized by powder bed based processes which, however, generate parts with residual porosity. Via infiltration these parts can be processed to dense parts like for example SiC but this is not possible for all ceramics like for example Si3N4. There is a lack of methods for the additive manufacturing of dense voluminous parts for most ceramics. We have developed a new additive manufacturing technology, the Laser Induced Slip casting (LIS), based on the layerwise deposition of slurries and their local drying by laser radiation. Laser Induced Slip casting generates ceramic green bodies which can be sintered to dense ceramic components like traditional formed ceramic powder compacts. We will introduce the LIS technology, green bodies and sintered parts will be shown and their microstructure and mechanical properties will be discussed.

S17: Advanced Ceramic Materials and Processing for Photonics and Energy

Photovoltaics

Room: Halifax A/B
Session Chairs: Mauro Epifani, CNR-IMM; Scott Misture, Alfred University

8:30 AM

(ICACC-S17-008-2018) Silver nanoaggregates and rare-earth-ions in glasses and glass-ceramics for solar cell applications (Invited)

F. Enrichi*[1]

1. Luleå University of Technology (Sweden) and Centro Studi e Ricerche E. Fermi (Italy), Sweden

The frequency-conversion properties of rare earth (RE^{3+}) ions may be used to enhance the efficiency of solar cells, converting poorly absorbed regions of the solar spectrum into the frequencies of maximum absorption of the cell. In this presentation, we report the study of rare-earth-doped downconverting glasses and

glass-ceramics prepared by sol-gel synthesis and dip-coating deposition. In particular, Tb-Yb codoped silica-hafnia films with different RE^{3+} concentrations were investigated, showing the possibility to obtain energy-transfer efficiencies close to the maximum theoretical limit. Moreover, Ag-exchanged Tb-Yb codoped silica-zirconia films were obtained by ion-exchange in a molten salt bath and subsequent annealing in air, to induce the migration and aggregation of the metal. The interaction between Ag nanoaggregates and RE^{3+} ions was studied and the sensitizing effect on spectral absorption widening and efficiency increasing is presented. Results of structural, compositional and optical characterization are given on the studied systems, providing evidence for UV-Vis to NIR downconversion and discussing perspectives for potential applications to increase the efficiency of Si-based PV solar cells.

9:00 AM

(ICACC-S17-009-2018) Chalcogen Polymers for Completely Solution-Processed Inorganic Photovoltaics (Invited)

C. Luscombe*[1]

1. University of Washington, Materials Science and Engineering, USA

Chalcopyrite materials such as CuInSxSe2-x (CISSe), the gallium alloy variant CuInxGa1-xSySe2-y (CIGSSe), and the earth-abundant kesterite material Cu2ZnSnSxSe4-x (CZTSSe) possess a range of properties that are ideally suited for thin-film photovoltaics (PV) applications. Although these materials are beginning to see some commercial success, they are manufactured using complicated and expensive techniques such as high temperature processing, vacuum deposition methods, and vapor-phase reactions. This work focuses on developing novel chalcogen polymers to synthesize nanoparticles and produce thin-films for printed photovoltaics applications. This new method provides a pathway towards using chalcogen copolymers to produce these materials via a completely solution-processed, low-temperature fabrication procedure. If successful, this technique would constitute the first viable means to produce low-bandgap chalcogenides without additional vapor-phase or high-temperature reactions.

9:30 AM

(ICACC-S17-010-2018) Semiconducting metal oxides: Engineering nanostructures for energy related applications (Invited)

I. Concina*[1]

1. Luleå Tekniska Universitet, Sweden

Modulation of physical, chemical and opto-electronic features in nanostructured materials can be achieved by proper material design, i.e. by tuning size range, size dispersion, shape and bulk or surface chemistry. In this frame, semiconducting metal oxides (MOx) are excellent candidates as a multi-functional and versatile platform, whose features are enhanced/modified by material aspect engineering. In this lecture we will present some strategies that may be applied to the fabrication of nanostructured semiconducting MOx to be exploited in solar energy conversion applications, such as solar cells, water splitting and photocatalysis. The following cases will be discussed: 1) electrodes for excitonic solar cells, made by hybrid structures composed by: i) graphene sheets and TiO_2 nanoparticles, iii) layered architectures ZnO@SnO_2; 2) enhanced photocatalytic activity by modulation of materials aspect (ZnO); 3) promoting the oxygen evolution reaction by engineering a hematite-based photoelectrode. Particular emphasis will be given on a materials by design approach, highlighting the relevant potential to provide access to efficient functional materials. Attention will also be paid to simple, vacuum-free and green synthetic strategies in the big picture of working to comply with the parameters of so-called green chemistry, which appear particularly relevant in the field of solar energy exploitation.

10:20 AM

(ICACC-S17-015-2018) Integration of Freestanding Two-dimensional Transition Metal Dichalcogenides (Invited)

H. Jeong[1]; A. Gokarna[1]; M. Hye[2]; S. Yun[2]; G. Han[2]; M. Jeong[2]; Y. Lee[2]; G. J. Lerondel*[1]

1. University of Technology of Troyes, Laboratoire de Nanotechnologie et Instrumentation Optique, Institut Charles Delaunay, CNRS UMR 6821, France
2. SungKyunKwan University, Department of Energy Science, Republic of Korea

Atomically thin semiconductors consisting of transition metals covalently bonded to chalcogens i.e. 2D transition metal dichalcogenides (TMDs), are the focus of extensive research due to their remarkable intrinsic properties such as light emission and absorption. Such properties can nevertheless strongly be altered once the atomically thin layer is deposited on a support. We report here on the integration of freestanding TMDs based on the new limited contact concept. Monolayer (1-L) MoS_2, WS_2, and WSe_2 as representative TMDs have been transferred on ZnO nanorods (NRs), used here as nanostructured substrates. The photoluminescence (PL) spectra of the 1-L TMDs on NRs is largely enhanced (x 100) as to compare with the one of TMDs on SiO_2. The strong increases in Raman and PL intensities, along with the characteristic peak shifts, confirm the absence of stress in the TMDs on NRs. In depth PL analysis also reveals that the ratio between the exciton and trion peak intensity is almost not modified after transfer confirming that charge transfer is here negligible. Furthermore, confocal PL and Raman microscopy reveal a fairly consistent distribution of PL and Raman intensities. These observations confirm that the physical contact between the 1L-TMD and the support is negligeable. Perspectives include efficient ultrathin optoelectronics with direct application in photovoltaics and solid state lighting.

10:50 AM

(ICACC-S17-012-2018) Halide/oxide perovskites for efficient hybrid optoelectronic devices (Invited)

R. Nechache*[1]

1. Ecole de technologie Superieure, Electrical Engineering, Canada

Since the discovery of the bulk photovoltaic effect in ferroelectrics, there has been a growing interest in perovskite (PE) materials for energy related applications, including PV and water splitting. In such materials, the spontaneous polarization-induced electric field promotes the required separation of photo-excited carriers and allows photovoltages higher than their bandgap, which lead to efficiencies that can exceed the maximum possible in a semiconductor p–n junction solar cells. Among these materials, Bi_2FeCrO_6 is highly promising because it exhibits a conversion efficiency of about 8.1% under 1 Sun in thin film form. Other perovskites can be hybrid, if the cation A is replaced with an organic radical. This is the case for halide PE compounds (CH_3NH_3PbX), with X=Br, Cl, I, found recently to possess excellent light absorption in the VIS-NIR spectrum. The use of these materials in solar cells had led to a rapid increased of the photovoltaic conversion efficiency in the last year exceeding 22 %. Our study deals with the major challenges related with the implementation of functional PE oxides in organometallic PE based optoelectronic devices. The optimization of the properties of such hybrid systems and the performance of their related devices will be also discussed.

11:20 AM

(ICACC-S17-013-2018) Mesoporous Germanium for High-Efficiency Photovoltaic Cells (Invited)

C. Valdivia*[1]; M. N. Beattie[3]; Y. A. Bioud[2]; D. G. Hobson[3]; A. Boucherif[2]; D. Drouin[2]; R. Ares[2]; K. Hinzer[1]

1. University of Ottawa, School of Electrical Engineering and Computer Science (EECS), Canada
2. Université de Sherbrooke, Institut interdisciplinaire d'innovation technologique (3IT), Canada
3. University of Ottawa, Dept. of Physics, Canada

Germanium wafers were nanostructured via electrochemical etching, synthesizing a high density of randomly nanostructured vertical columnar crystallites of up to several microns in height. We have demonstrated that the electrical resistivity of these mesoporous Ge layers can be widely tuned by thermal annealing, ranging over 30-1700 Ω•cm. As-prepared mesoporous Ge with 70% porosity and crystallites of 4-10 nm in size has a resistivity of ~1100 Ω•cm, which is 10^5 times larger than the substrate. While low temperature annealing (<400°C) resulted in further resistivity increases, higher temperatures induced a significant morphological transition resulting in interconnecting granular crystallites of increasing size. This transition was accompanied by a 100-fold decrease in resistivity due to the diminishing influence of surface states at the void-Ge interfaces. To account for these changes, we developed a computational model to simulate the carrier concentration and mobility of p-type mesoporous Ge, which has suggested that the electrical properties might be further tuned by up to 4 orders of magnitude. While this tunable resistivity could be advantageous for many applications, our present interest is in obtaining low-resistance layers for use as either (1) a virtual substrate to grow epitaxial layers with reduced lattice-matching constraints, and/or (2) a release layer to manufacture thin film multi-junction solar cells.

Honorary Sympoisum: Advancing Frontiers of Ceramics for Sustainable Society Development - International Symposium in Honor of Dr. Mrityunjay Singh

Advancing Frontiers of Ceramics II -Novel Processing 1

Room: Coquina Salon E

Session Chairs: Pavol Sajgalik, Institute of Inorganic Chemistry, Slovak Academy of Sciences; Hisayuki Suematsu, Nagaoka University of Technology

8:30 AM

(ICACC-HON-010-2018) Designing the processing of advanced ceramics and composites to yield the required properties (Invited)

J. Binner*[1]

1. University of Birmingham, School of Metallurgy & Materials, United Kingdom

Advanced ceramics and composites find widespread uses across a very wide range of industries, including aerospace, electronics, health, defence, energy and many others. Their global market is predicted to reach ~US$80B by 2022[1] as a result of constant developments driven by end-users, the manufacturing sector and researchers. Currently, however, when new materials and components are needed they are developed by an iterative process based largely on empirical research. This is slow, time consuming, uncertain and hence costly; hence there is a tremendous need to develop a much smarter approach. This paper will discuss the first steps towards achieving a highly ambitious vision to develop the required understanding of how the microstructures, processing and properties interact to the point whereby a very wide range of ceramics materials with the required performance can be designed, manufactured and used for a wide range of end applications. [1] The Global Advanced Ceramics Market (MCP 1001), Global Analysts Industry Inc, 2016. See http://www.strategyr.com/MarketResearch/Advanced_Ceramics_Market_Trends.asp.

9:00 AM

(ICACC-HON-011-2018) SPS Sintering of near net shape ceramics (Invited)

F. J. Cambier*[1]; S. Hocquet[1]; M. Demuynck[1]; V. Lardot[1]

1. Belgian Ceramic Resaerch Centre, R&D, Belgium

Recent developments in prototyping allows to obtain complex shape components. However, sintering remains the slow step of the manufacturing process, but SPS technology is used for the fast densification of powders, combining uniaxial pressure application and thermal treatment resulting from Joule effect generated by a high current flowing through graphite die & punches and possibly through the powder if it is electrically conductive. There is still a strong limitation regarding shape: only samples with simple geometry can be obtained, reducing the scope of applicability of SPS technology. Some attempts to modify die and punches geometries are today studied, but then a new set of graphite tools has to be machined for each sample shape, increasing time and costs of production. In this communication, we will show another method consisting of embedding the green part in a granular medium in order to reach a quasi isostatic effect around the sample, avoiding uniaxial deformations. Powder beds (graphite and SiC) were selected regarding chemical and thermal stability; adapted flowability; good compressibility behavior; re-use possibility. Alumina and WC-Co were chosen as tested materials. Optimizing the technique was done in 3 steps: sintering simple shapes while minimzing distortion; set up of a numerical model describing the granular media behavior under pressure; densification of green complex shape taking into account results of simulation.

9:30 AM

(ICACC-HON-012-2018) Electrically Conductive Ceramics – Processing and Properties (Invited)

P. Sajgalik*[1]

1. Institute of Inorganic Chemistry, Slovak Academy of Sciences, Ceramic Department, Slovakia

Si_3N_4 and Al_2O_3 ceramics are insulating materials, SiC is a semiconductor. All of this type of ceramics are widely used for the engineering applications because of their excellent mechanical and chemical properties. Hardness is one of those which belongs to the most important. This brings also a problem with final machining of these materials. Introduction of the electrically conductive elements without substantial decreasing of the other properties of ceramics is a way how to apply also the electrical discharging machining also for this class of materials. Present paper deals with the processing of three types of ceramics with increased electrical conductivity. NbTiC electrically conductive phase was used for SiC ceramics, carbon nano-fibres were used for Si_3N_4 ceramics and carbon nanotubes were used for Al_2O_3 ceramics. In all types the increased electrical conductivity was observed, 2700 S/m for SiC ceramics, 1300 S/m for Si_3N_4 ceramics and 1800 S/m for Al_2O_3 ceramics. The way of conductive phase introduction and its influence on the mechanical properties will be discussed. For the processing was used hot-pressing as well as rapid hot-pressing.

10:10 AM

(ICACC-HON-013-2018) Fabrication of Transparent Ceramics Via Novel Processing Methods (Invited)

H. Kim[1]; Y. Park*[1]

1. Korea Institute of Materials Science, Republic of Korea

Two novel methods for the fabrication of transparent ceramics are introduced. One is a facile hot pressing method with tantalum foil shielding which effectively prevents the ceramic samples from carbon contamination caused by the graphite mold for hot pressing. As-hot-pressed Y_2O_3 was already highly transparent without a post-annealing step or hot isostatic pressing. The sample shows a very fine microstructure with an average grain size of about 1 μm owing to the low sintering temperature of only 1600°C. The result indicates that it is possible to produce transparent Y_2O_3 ceramics with excellent optical transparency using simple hot-pressing method. The other is a microfluidization method which disintegrates nano-particle agglomerates in aqueous slurry by passing the material through a narrow channel with a high shear stress. The uniformity of a green body of $MgAl_2O_4$ could be improved by both a wet shaping via microfluidization of the slurry and following slip-casting process. Another crucial factor for enhancing the transparency of $MgAl_2O_4$ was the addition of a small amount of Ca additive, which suppress the grain growth by the segregation at grain boundaries.

10:30 AM

(ICACC-HON-014-2018) Ultrafast Laser for Materials Drilling and Cutting (Invited)

S. Jiang*[1]

1. AdValue Photonics Inc, USA

In this presentation, we report our latest developments of high peak power ultrafast fiber lasers based upon our innovative fiber laser technology. Laser drilling and cutting on ceramics and glasses will be presented.

10:50 AM

(ICACC-HON-015-2018) Smart powder processing of advanced materials for sustainable society (Invited)

M. Naito*[1]; T. Kozawa[1]; A. Kondo[1]

1. JWRI, Osaka University, Japan

Recently, various novel powder processing techniques were rapidly developed for advanced material production, especially in consideration of green and sustainable manufacturing. Smart powder processing stands for green and sustainable powder processing technique that creates advanced materials with minimal energy consumption and environmental impacts. Particle bonding technology is a typical smart powder processing technique to make advanced composites. It creates direct bonding between particles without any heat support or binders of any kind in the dry phase. The bonding is achieved through the enhanced particle surface activation induced by mechanical energy, in addition to the intrinsic high surface reactivity of nanoparticles. Using this feature, desired composite particles can be successfully fabricated. By making use of the particle bonding, nanoparticles can be directly synthesized from raw powder materials by one-pot processing without applying extra heat. Furthermore, both the synthesis of nanoparticles and their bonding with another kind of particles to make nanocomposite granules is also easily achieved. In this presentation, the applications of the smart powder processing for the cathodes of LIB and the phosphors for LED will be presented. Both are key materials for sustainable society.

11:10 AM

(ICACC-HON-016-2018) Sol-gel synthesis and electrical properties of $MgZr_4P_6O_{24}$ Composite Solid Electrolyte and its application for sensing Mg in molten Al (Invited)

M. Adamu[1]; G. Kale*[1]

1. University of Leeds, School of Chemical and Process Engineering, United Kingdom

A magnesium ion conducting $MgZr_4P_6O_{24}$ (MZP) ceramic material with potential application as solid electrolyte in high temperature electrochemical sensor for non-ferrous metal refining and alloying operations has been synthesised by sol-gel method. An insight into the calcination process of the dried gel was obtained using simultaneous TGA/DSC studies. Phase identification of the calcined material was studied using XRD. Impedance spectroscopy measurement on platinised sintered-MZP pellets were carried out in the frequency range 100 mHz – 32 MHz and in a temperature range of 30 - 800°C to determine the electrical properties of MZP. The ac and dc conductivity of MZP show Arrhenius behaviour with activation energies in the range $0.84 \leq E_a(eV) \leq 0.87$. SEM of the fractured MZP pellet sintered at 1300°C for 24h revealed a highly dense microstructure with low porosity which is in good agreement with the relative density of ~99%. EDS confirms Mg, Zr, P, O in appropriate atomic ratio to yield stoichiometric $MgZr_4P_6O_{24}$. TEM of MZP particles confirmed the crystallite size of ~50nm for gel calcined at 900°C. The nanopowders of MZP were pressed isostatically and sintered into high density one end closed tubes for the fabrication of solid state sensor for measuring Mg in Al at 963 K using biphasic $MgCr_2O_4$+Cr_2O_3 and $MgFe_2O_4$+Fe_2O_3 ceramic reference electrodes.

11:30 AM

(ICACC-HON-017-2018) Catalytic Combustion-type Carbon Monoxide Gas Sensor with Platinum-loaded Oxide Ion Conducting Solid Electrolyte Catalysts (Invited)

N. Imanaka*[1]

1. Osaka University, Applied Chemistry, Japan

One of the features for conventional catalytic combustion-type CO gas sensors is that the catalysts such as Pt loaded Al_2O_3 or Pd loaded Al_2O_3, can operate relatively elevated temperatures over 400° C for the complete carbon monoxide gas oxidation. Since other gases such as methane and volatile organic compounds (VOCs) also burn out at such operating temperatures, this type of sensors has a great disadvantage in selectivity. Recently, by using 10 wt% Pt loaded $Ce_{0.68}Zr_{0.17}Sn_{0.15}O_{2.0}$ solid as the catalyst, we have succeeded in developing a new sensor showing considerably moderate temperature operable catalytic combustion-type, operating even at 70°C, which is more than 300°C below in comparison with the conventional ones. In addition, by the combination of the superior thermoelectric material (aluminum nitride ceramics powder) as an intermediate heat transfer with the above Pt loaded $Ce_{0.68}Zr_{0.17}Sn_{0.15}O_{2.0}$ catalyst, a novel portable catalytic combustion-type carbon monoxide gas sensor was realized, having an excellent sensing performance that drastically reduces the CO sensing response time at the moderate operating temperature of 70°C. Also, platinum is one of representative precious but rare metals (critical metals). Therefore, Pt-free CeO_2–ZrO_2–SnO_2 solid solution catalyst is also examined.

11:50 AM

(ICACC-HON-018-2018) Synthesis of novel materials utilizing pulsed power technologies

H. Suematsu*[1]; T. Suzuki[2]; T. Nakayama[1]; K. Niihara[2]

1. Nagaoka University of Technology, Extreme Energy-Density Research Institute, Japan
2. Nagaoka University of Technology, Department of Nuclear System Safety Engineering, Japan

Pulsed power technologies which generate high power in short duration have been developed in the nuclear fusion research. These technologies include to make large current sources with large capacitors and low inductance circuits to shorten the pulse width. From material science point of view, such large current sources to input high energy trigger quick phase changes or novel chemical reactions to target materials. By using the advantage of pulsed power technologies, we have developed preparation methods of passivated metal and alloy nanosized powder by pulsed wired discharge and oxynitride hard coating by pulsed laser deposition. Titanium, zirconium and magnesium nanosized powders preserve the metallic phase in air at room temperature for more than a month. Thin films of Cr(N,O) exhibit 60% higher hardness than that of CrN. Other achievements of the pulsed power technologies are also shown in the presentation.

7th Global Young Investigator Forum

Novel Characterization Tools of Ceramics and Composites

Room: Coquina Salon G
Session Chair: Manoj Mahapatra, University of Alabama at Birmingham

8:30 AM

(ICACC-GYIF-010-2018) Phase development in sol-gel processing of nano phase ZrN and ZrC powders (Invited)

G. Westin[1]; S. Naim Katea*[1]

1. Uppsala University, Sweden

ZrN and ZrC have many extreme properties such as high melting point, hardness, durability, thermal and electric conductivity and are therefore of interest for use in hard cermets and matrixes for nuclear fuels such as; U, Pu and Am in the Pb cooled Gen IV nuclear reactors. The present carbothermal processes typically yield irregular micron sized and impure powders requiring very high sintering temperatures, even when using SPS, which is not suitable for production. Here the phase development in new sol-gel processes to nano-phase ZrN and ZrC based on sucrose-Zr-alkoxide precursors is described in detail from the precursors to the final powders and key parameters affecting the powder quality will be discussed. A number of experimental techniques were used including; TG-DT, DSC, XPS, XRD, IR- and Raman spectroscopy, SEM-EDS and HR-TEM-EDS/EELS. It was found that an extremely intimate mixing of $ZrO_x(OH)_y$ and graphenic carbon was obtained at 200°C, and that the Zr-particles were converted to ZrO_2 at 600°C with retained structure at 600°C. The carbothermal reactions started at 1200°C and were finished at 1500°C. The ZrC and ZrN were phase pure according to XRD, but TEM showed that the nano-sized particles had a 4-5nm thick amorphous or fine-crystalline shell. The details of this shell are discussed, as well as its formation.

9:00 AM

(ICACC-GYIF-012-2018) Analyzing Damage in SiC/SiC CMCs Using In Situ Synchrotron Techniques

A. Hilmas*[1]; A. Singhal[2]; Y. Zhou[2]; G. Henson[2]; Y. Gao[2]; K. M. Sevener[1]

1. University of Michigan, USA
2. GE GRC, USA

In-situ mechanical testing of a SiC/SiC composite was performed in Beamline 8.3.2 of the Advanced Light Source (ALS) at Lawrence Berkeley National Laboratory. Composite samples were loaded at room temperature and imaged at incremental loads through fracture. Both uniaxial and cross-ply laminates were tested. The tomography images were used to quantify damage accumulation such as fiber breaks and matrix cracking in the composites. Measurements of the various data were compared to current models.

9:20 AM

(ICACC-GYIF-013-2018) Compressive response of ice-templated ceramics: Effects of solids loading, particle size, and particle morphology

M. Banda*[1]; H. Kang[1]; S. Akurati[1]; D. Ghosh[1]

1. Old Dominion University, Mechanical and Aerospace Engineering, USA

Ice-templated ceramics are typically synthesized from aqueous suspension of fine, equiaxed particles. While the pore volume of scaffolds depends on solids loading of suspensions, freezing kinetics influence lamellar bridge density, pore size and structural morphology. In our recent studies, we have shown that variations of particle size and particle morphology along with freezing front velocity can result in unprecedented modifications of the length-scale features of ice-templated ceramics, and marked improvement of compressive mechanical response has been measured. In this presentation, first we will demonstrate the unique effects of solids loading, particle size, and platelet morphology on porosity, lamellar bridge density, pore size and aspect ratio, and pore morphology of ice-templated ceramics. Next, we will present the results of measurements of uniaxial compressive response of the resulting scaffolds. Finally, we will discuss the connections between the pore volume and length-scale features, and the macroscopic compressive response of ice-templated ceramics. Overall goal of this study is to significantly improve our current understanding of the structure-property relationships of ice-templated ceramics. Implication of the work will be realized in advancing design and development of bioinspired, mechanically robust lightweight materials.

Young Researchers Funding, Mobility and Networks

Room: Coquina Salon G
Session Chairs: Daniele Benetti, Institut National de la Recherche Scientifique; Giorgia Franchin, University of Padova; Manoj Mahapatra, University of Alabama at Birmingham

10:20 AM

(ICACC-GYIF-014-2018) Making the Faculty Leap: Adventure and Learning as a New Assistant Professor (Invited)

D. L. Poerschke*[1]

1. University of Minnesota, Chemical Engineering and Materials Science, USA

Faculty positions can be incredibly fulfilling by allowing the intellectual and personal freedom to nurture new ideas and carry out creative research in a collegial, stimulating environment, while providing gratification as students learn and succeed at their own projects. At the same time, the process of building a lab and research group while raising money and teaching new courses can be tremendously time consuming, and at times stressful and isolating. Based on the collective experiences navigating the academic job market

and starting as a first-year assistant professor working in the structural ceramics field, this talk will provide advice and insight to young researchers interested in transitioning from graduate school to other academic positions. In addition to discussing technical aspects related to academic positions, the talk will also cover broader themes related to mentoring, collaborating, and work-life balance.

S1: Mechanical Behavior and Performance of Ceramics & Composites

Complex Sections, Texture, Indentation and Fatigue

Room: Coquina Salon D

Session Chairs: Jonathan Mackey, NASA Glenn Research Center; Amjad Almansour, NASA Glenn Research Center

1:30 PM

(ICACC-S1-020-2018) Constraint-induced transformation reversal during cyclic loading of Ce-TZP ceramics: A dangerous fatigue mechanism

M. Saran[1]; B. Murray[1]; T. Scott[1]; R. I. Todd*[1]

1. University of Oxford, Department of Materials, United Kingdom

Zirconia ceramics such as Y-TZP and Ce-TZP are used in critical applications such as hip joint replacements and dental implants because of their high toughness. These applications involve cyclic loading and are therefore susceptible to fatigue failure. It is thought that the accumulation of damage such as microcracking associated with the tetragonal to monoclinic transformation plays a role in cyclic fatigue. This presentation gives evidence that reverse transformation during unloading also contributes. The temperature required to transform stress-induced monoclinic zirconia back to the tetragonal polytype when unloaded is investigated. The results show that the temperature required is lower the greater is the constraint of the surrounding material, and that for Ce-TZP, reverse transformation can occur at room temperature when the constraint is sufficiently high. Fatigue tests were conducted under R=0 and R=-1 loading conditions. The lifetime was significantly shorter in fully reversed loading than in R=0 testing. Raman microscopy demonstrated that the fracture surfaces in the region of initial crack growth contained significantly less monoclinic phase in cycled specimens than in specimens tested in monotonic loading. The results are consistent with the cyclic degradation of toughness by reverse transformation during unloading.

1:50 PM

(ICACC-S1-021-2018) Characterization of Dynamic Indentation in Gas-turbine-grade Silicon Nitride Ceramic

N. Kedir*[1]; C. D. Kirk[2]; Y. Nie[3]; N. Parab[4]; B. Claus[3]; T. Sun[4]; K. Fezzaa[4]; W. Chen[3]

1. Purdue University, Materials Engineering, USA
2. Purdue University, Mechanical Engineering, USA
3. Purdue University, Aerospace Engineering, USA
4. Argonne National Lab, USA

A study of the dynamic indentation response of AS800 Silicon Nitride Ceramic was performed through phase contrast imaging (PCI) capabilities rendered by a high-speed Synchrotron X-ray imaging setup. Hardened steel indenters with diameters ranging from 1.2 to 2.8 mm were used as part of a modified Kolsky bar setup to apply the dynamic compressive load at an indenter velocity of ~ 6 m/s. The PCI method showed the sequential fracture process of the ceramic and the deformation of the indenter in real-time. Combined Hertzian cracks and median cracks were observed to yield catastrophic fracture of the ceramic into several fragments. Analysis of the surface morphology of some recovered projectiles and specimens also showed distinct markers for locations of surface ring cracks. A close match was obtained between the measured ring crack diameter and critical contact diameter for cracking in the PCI observations.

2:10 PM

(ICACC-S1-022-2018) Evaluation of New Technique to Estimate Yield Stress in Brittle Materials via Spherical Indentation Testing

B. L. Hackett*[1]; A. Wereszczak[3]; G. M. Pharr[2]

1. University of Tennessee, USA
2. Texas A&M University, USA
3. Oak Ridge National Lab, USA

Instrumented indentation testing provides the means to measure many mechanical properties and characteristics of materials. One such mechanical property that can be ascertained from indentation testing is a material's yield stress, the stress corresponding to the onset of plastic deformation. In brittle materials such as ceramics and glasses, traditional testing methods fall short of precisely establishing the yield stress. A new technique to estimate the yield stress, evidenced through sensing and interpreting the initiation of a residual surface impression, of several brittle materials (borosilicate, soda-lime silicate, and bulk metallic glass) using spherical nanoindentation was developed. The test method and manner of interpreting the loading-unloading history associated with the yield-like response are described.

2:30 PM

(ICACC-S1-023-2018) Weibull Scaling Effects in Silicon Carbide Tubes Using Various Strength Testing Techniques

S. M. Chown*[1]

1. Saint-Gobain, USA

Silicon carbide roller tubes are an increasingly attractive solution for the design of higher efficiency, high temperature roller hearth kilns. A demand for throughput has driven these furnaces to require higher running capacities. Subsequently, this increased volume leads to a heavier load on the rollers, which requires stronger high temperature roller materials. Therefore designing these ceramic products, often based solely on manufacturer's reported "strength", is a critical task. When comparing different materials and grades, it is important to understand the information that is available. Specifically, reported values for generically defined strength vary widely and are potentially misinterpreted when used for engineering calculations and design including a proper factor of safety. This can be exacerbated if there is no designation of test methodology as the source of the information. In this paper, we discuss the impact of four point and three point flexural strength tests and how Weibull volume is used to scale to the effective strength of the material. Through Weibull volume, we observe the scalability between preferred ASTM C1161 results and those obtained when testing the more readily available sections of production components, given the typical failure mechanisms observed in rollers.

2:50 PM

(ICACC-S1-024-2018) Mechanical characterization and fractography of ceramics produced by additive manufacturing

M. Schwentenwein*[1]; T. Lube[2]; J. Schlacher[2]; G. Mitteramskogler[1]; W. Harrer[2]; R. Danzer[2]

1. Lithoz GmbH, Austria
2. Montanuniversitaet Leoben, Institut für Struktur- und Funktionskeramik, Austria

Additive manufacturing (AM) techniques increasingly become a relevant fabrication method in the field of high-performance ceramics. The increasing importance of these components also requires the availability of testing procedures that ensure the quality and homogeneity of parts made by AM. A specific aspect of the strength of additive manufactured components and specimens

is the building direction. The interface between adjacent layers may have properties that deviate from the properties of the layers themselves. In a bending bar as usually used for strength testing of ceramics the layers may be oriented parallel to each of the specimens faces. During flexural strength testing the applied stress then acts normal or parallel to the interfaces. This enables an investigation of properties in relation to the building direction. In this contribution, we present mechanical properties obtained on specimens from additive manufactured alumina components. Specimens tested in different orientations with respect to the building direction are investigated. The relation between applied stress direction, strength and building direction is presented. It is shown that proper choice of the materials as well as the printing and post-processing parameters are extremely important to realize a homogeneous microstructure in the 3D printed part and thus, ceramic components with isotropic material properties.

3:30 PM

(ICACC-S1-025-2018) Strength Evaluation of Interconnects via Cantilever Testing

A. Wereszczak*[1]; B. Chen[2]; O. Jadaan[3]; B. Oistad[1]; M. Modugno[4]; J. Sharp[5]; J. Salvador[6]

1. Oak Ridge National Laboratory, USA
2. University of Delaware, USA
3. University of Mount Union, USA
4. Alfred University, USA
5. Marlow Industries, USA
6. GM Global R&D, USA

Cantilever testing is an underutilized test method whose results and interpretations promote greater understanding of the tensile and shear failure responses of interconnects, metallizations, or bonded joints, such as those used in thermoelectric devices. The use and analysis of this method were pursued through the mechanical testing of interconnects that joined copper pillars or silicon pillars to substrates. Sintered-silver was chosen as the interconnect test medium because of its high electrical and thermal conductivities and high-temperature capability - attractive characteristics for a candidate interconnect in power electronic components and other devices. Deep beam theory was used to correct the estimations of the tensile and shear stresses. The experimental simplicity of cantilever testing, and the ability to analytically calculate tensile and shear stresses at failure, result in it being an attractive mechanical test method to evaluate the failure response of interconnects. This manuscript has been authored by UT-Battelle, LLC, under Contract No. DE-AC05-00OR22725 with the U.S. Department of Energy.

3:50 PM

(ICACC-S1-026-2018) Torsion tests on joined ceramics: How to deal with brittle or ductile joining materials?

L. Goglio*[1]; M. Ferraris[2]; M. Salvo[2]; V. Casalegno[2]; S. De La Pierre[2]

1. Politecnico di Torino, Mechanical and Aerospace Engineering Department, Italy
2. Politecnico di Torino, Applied Science and Technology Department, Italy

Results of an experimental investigation on brittle and non-brittle joining materials for ceramics tested in torsion will be presented and discussed. SiC hourglass shaped samples have been joined by several brittle and non-brittle adhesives. The hourglass shape has been designed to minimize stress concentration, avoid interface stress singularity and have shear stress only in the joined area. When the joining material is purely brittle, the shear strength is evaluated from the maximum of the torque/rotation curve. In the case of non purely brittle joining materials, the joined area must be gradually reduced to a ring-shaped area until size independent results are obtained and the maximum of the torque/rotation curve can be used to evaluate the shear strength. Modelling results help understanding this behavior and therefore the meaning of the measured strength, which can be identified more accurately.

4:10 PM

(ICACC-S1-027-2018) Toward Standardization of Strength Testing of the Sectored Flexure Ceramic Specimen

B. Oistad*[1]; A. Wereszczak[1]; B. Chen[1]; O. Jadaan[2]

1. Oak Ridge National Lab, USA
2. University of Mount Union, Department of Engineering, USA

Efforts are underway to ASTM-standardize the strength testing of the sectored flexure specimen that is harvested out of ceramic and glass tubes and cylinders. The specimen was developed specifically for circumstances in which flaws located at a tube's or cylinder's outer diameter are strength-limiters when subjected to uniaxial bending or axial tension. Examples of previously tested specimens are reviewed, and attention is given to more recent 4-point flexure testing of SiC and glass sectored specimens harvested out of 25.6 mm x 18.9 mm x 60.9 mm and 87.9 mm x 76.2 mm x 101.6 mm, respectively. The imposed stresses, and potential specimen design and their machining considerations are discussed. This manuscript has been authored by UT-Battelle, LLC, under Contract No. DE-AC05-00OR22725 with the U.S. Department of Energy.

4:30 PM

(ICACC-S1-028-2018) Ice-templated ceramics: Understanding uniaxial compressive behavior both in quasistatic and dynamic regimes of loading

D. Ghosh*[1]; M. Banda[1]; H. Kang[1]

1. Old Dominion University, Mechanical and Aerospace Engineering, USA

Due to lightweight, high specific stiffness and strength, and energy absorption capacity, ice-templated ceramics are promising candidate materials for nuclear warheads, armor systems, anti-mining, and impact protection. In these applications, materials are often subjected to mechanical forces within loading durations of tens of microseconds. Therefore, mechanical response of ice-templated ceramics measured in quasistatic regime may not truly represent their dynamic (high-strain rate) mechanical behavior. In this presentation, we will discuss our work on understanding the uniaxial compressive response of ice-templated sintered ceramics both in the quasistatic and dynamic regimes of strain rates. We utilized ice-templated alumina as a model system and employed a split-Hopkinson pressure bar (SHPB) to measure the uniaxial dynamic compressive response. In the processed ice-templated ceramics, porosity, lamellar bridge density, structural morphology, and other length-scale features were modified through the systematic variations of the intrinsic (solids loading of suspension, particle size and morphology) and extrinsic (freezing front velocity) variables. This study will help to decipher the effects of pore volume, pore morphology, and length-scale features on the mechanical response of ice-templated ceramics at two widely different strain rate regimes.

4:50 PM

(ICACC-S1-048-2018) Applicability of Rapid Prototyping for manufacturing of advanced ceramic nano-composites

D. B. Kata*[1]; P. Rutkowski[1]; J. Huebner[1]; J. Lis[1]

1. AGH University of Science and Technology, Faculty of Materials Science and Ceramics, Poland

The applicability of RP is shown based on the two nanocmposites: AlN-graphene and Inconel 625 – WC. The 30% of anisotropy in thermal conductivity, elastic properties and K_{Ic} was obtained in AlN-graphene nanocomposites due to RP technology. The structures were examined by X-ray diffraction, SEM and TEM methods. It is showed that graphen plays a crucial role in anisotropy and influences on microstructure appearance. The possibility of controlling anisotropy by graphen content and RP conditions was showed. Chemically and structurally homogenous Inconel 625 – WC nanocomposite coatings were prepared due to Rapid Prototyping technology. Because of chemical composition of Inconel 625 superalloy, secondary carbides of WC, W_2C, NbC, (NbW)C, $W_6C_{2.54}$ and

$(W,Cr,Ni)_{23}C_6$ were detected in the intergranular spaces by XRD analysis. They appeared in form of eutectic with typical fishbone-like structure in samples containing increased amount of WC - 30 wt %. High cooling rate during RP process was the reason of fine microstructure in produced material. TEM observation showed two types of precipitations: angular carbides and spherical oxides containing increased amount of Cr, Si and Ti. Finally, a fundamental interaction betwen laser beam and AlN-graphene or Inconel 625-WC was examined. The different aspects of the ceramic laser processing from fundamental mechanism up to engineering applicability is discussed.

S2: Advanced Ceramic Coatings for Structural, Environmental, and Functional Applications

Innovative Multifunctional Coatings

Room: St. John

Session Chair: Eugene Medvedovski, Consultant

1:30 PM

(ICACC-S2-021-2018) Coatings Based on Thermal Diffusion Technology for Molten Salt Corrosion Environment in Pulp and Paper Processing

E. Medvedovski*[1]; G. Leal Mendoza[1]; A. Mahdavi[2]; A. McDonald[2]

1. Endurance Technologies Inc., Canada
2. University of Alberta, Canada

Protective coatings obtained through the thermal diffusion technology have been tested in modeling high temperature corrosion conditions with molten salts. According to microstructural and phase analysis studies, the coatings with multilayered architectures based on borides, aluminides and chromides, with and without additional oxide layers, demonstrated successful performance over bare steels. The coating technology is applicable for large size steel components and tubing required for recovery boilers and other production units in pulp and paper processing.

1:50 PM

(ICACC-S2-022-2018) Erosion-Corrosion Studies of the Iron Boride Coatings for Protection of Tubing Components in Oil Production

E. Medvedovski*[1]; S. Hernandez[2]; R. Sarrafi-Nour[2]; H. Arabnejad[3]; A. Prescott[3]; S. A. Shirazi[3]

1. Endurance Technologies Inc., Canada
2. Chevron Energy, USA
3. The University of Tulsa, USA

Heavy oil production and oil sand processing require protection of steel components against sand erosion and erosion-corrosion. Boronized coatings on carbon steels obtained through thermal diffusion process and consisted of dual iron boride layers FeB and Fe_2B with a total thickness of 200-250 μm have been evaluated in the dry sand erosion, wet erosion and synergistic erosion-corrosion (CO_2-brine water) conditions at elevated temperature. Iron boride coatings demonstrated significantly higher resistance in erosion and erosion-corrosion conditions at 90 degrees impingement angle over carbon steel commonly used in industry. Their performance would be even greater in sliding erosion generally occurring in the field production situations. High performance of the FeB-Fe_2B coatings is defined by their high hardness, chemical inertness and diffusion related bonding with steels. The tubing and casing with inner or inner and outer protective iron boride coatings can be successfully employed in downhole oil production conditions.

2:10 PM

(ICACC-S2-023-2018) Influence of Carrier Gas Species on the Room Temperature Powder Aerosol Deposition Process

M. Schubert*[1]; R. Wang[1]; J. Kita[1]; R. Moos[1]

1. University of Bayreuth, Department for Functional Materials, Germany

The Aerosol Deposition Method is a novel ceramic coating technique. It allows manufacturing of dense ceramic films at room temperature directly from ceramic powders without any high temperature sintering steps and without expensive vacuum processes. Due to its deposition mechanism based on collision and densification of fine ceramic powder particles on a substrate, it is also called "Room Temperature Impact Consolidation". Ceramic particles are accelerated up to several hundred m/s and ejected on a target. There, they build dense ceramic layers in the range of 0.5 to 50 μm by fraction of the particles to crystallite sizes of about 20 nm and subsequent consolidation. The carrier gas is the driving force of this process and therefore affects the process. The speed of sound, the viscosity and other properties directly affect the deposition rate, the resulting film quality and functional properties. Previous studies showed that the mechanical stress of the films are reduced by 50% by using O_2 instead of N_2 as carrier gas. In this study, seven different gases (N_2, O_2, Ar, Ne, Kr, CO_2, SF_6) were used to deposit of Al_2O_3-films on three different substrates (stainless steel, glass, Al_2O_3) and the deposition rate was calculated. Furthermore, the mechanical film stress, the permittivity, and the optical transmission were measured. The results correlated with the physical properties of the gases.

2:30 PM

(ICACC-S2-024-2018) Annealing of conductive films formed at room temperature by powder Aerosol Deposition to improve their electrical properties

J. Exner*[1]; M. Schubert[1]; D. Hanft[1]; J. Kita[1]; R. Moos[1]

1. University of Bayreuth, Department of Functional Materials, Germany

Functional ceramic films play a key role in a large variety of applications, ranging from sensors and catalysts to components for energy conversion (SOFC) and storage (batteries). Increasing requirements for long-term stability and enhanced electrical properties also create a rising demand for improved coating techniques. Aerosol Deposition (AD) could meet these demands, since it combines several advantages over conventional ceramic coating techniques. The unique feature is the possibility to form dense, nanocrystalline ceramic films directly from the ceramic powder caused by the Room Temperature Impact Consolidation (RTIC) mechanism. Without the need for a heat treatment during or after deposition, mechanical film properties like hardness or wear resistance are comparable to sintered samples. However, electrical properties significantly deteriorate during RTIC, resulting in a strongly reduced conductivity or permittivity. In order to regain bulk electrical properties, a post deposition film treatment is necessary. This can be accomplished by thermal annealing, i.e. by a heat treatment far below typical sintering temperatures. The behavior of several ionic as well as electronic conducting AD films during annealing was investigated and linked to changes within the film morphology and crystal structure.

CMAS Degradation of T/EBC & Mitigation Strategies I

Room: St. John

Session Chair: Bryan Harder, NASA Glenn Research Center

3:30 PM

(ICACC-S2-026-2018) Determination of Crystallization Kinetic Parameters of CMAS with T/EBC Materials

J. Stokes*[1]; B. J. Harder[2]; V. L. Wiesner[2]; D. E. Wolfe[1]

1. Pennsylvania State University, Materials Science and Engineering, USA
2. NASA Glenn Research Center, Materials and Structures Division, USA

Thermal and environmental barrier coatings (T/EBCs) are used to insulate and protect superalloy and ceramic matrix composite (CMC) components from oxidation, corrosion, and detrimental interactions with molten calcium-magnesium-aluminum-silicates (CMAS) caused by ingestion of sand or volcanic ash in aircraft engines at temperatures above 1200°C. The present study investigates the crystallization and reaction kinetics of T/EBCs with various CMAS compositions as a function of time, temperature, and CaO/SiO_2 ratios in 4-component CMAS systems. T/EBC and CMAS powders were mixed in a 50:50 mol% ratio, from which pellets were fabricated by uniaxial pressing, followed by furnace heat treatments. Heat treatments were carried out from 1-50 hours at 1000°C, 1100°C, 1200°C, 1300°C and 1400°C, and the phase constitution and morphology of reaction products were characterized using XRD, SEM, and EDS.

3:50 PM

(ICACC-S2-027-2018) Molten sand resistance of plasma-sprayed blends of rare-earth oxides with yttria-stabilized zirconia

M. J. Walock*[1]; A. Nieto[1]; M. Graybeal[2]; W. R. Gamble[2]; B. Barnett[2]; A. Ghoshal[1]; M. Murugan[1]; M. S. Pepi[2]; J. Swab[2]

1. US Army Research Laboratory, Vehicle Technologies Directorate, USA
2. US Army Research Laboratory, Weapons and Materials Research Directorate, USA

Degraded environments that contain sand, dust, ash, soot, and/or salt can significantly affect the durability and performance of advanced gas turbine engines. Contaminant accumulation can reduce the efficacy of component cooling schemes and reduce air flow through the engine; sand-glazing can increase the thermal conductivity and reduce the strain-tolerance of thermal barrier coatings (TBCs). In addition, calcia-magnesia-alumina-silicate (CMAS), formed by the molten sand, can attack and degrade/destroy TBCs through chemical interactions. In this research, commercially-sourced rare-earth oxide and yttria-stabilized zirconia (YSZ) powders are mechanically blended and subsequently deposited onto 1-in diameter IN 718 discs by atmospheric plasma spray (APS). For comparison, standard YSZ samples have also been fabricated by APS. These discs are exposed to hot gases and molten sand in a button cell flame test rig and hot-particulate ingestion rig, under engine relevant conditions. The evolution of the standard and blended TBC microstructures, and their resistance to calcia-magnesia-alumino-silicate deposits, are investigated with x-ray diffraction, scanning acoustic microscopy, scanning electron microscopy with energy-dispersive spectroscopy and scanning Auger electron microscopy.

4:10 PM

(ICACC-S2-028-2018) Effect of Biofuel Impurities on the Hot Corrosion of Thermal Barrier Coatings

J. H. Ramirez Velasco*[1]; H. Kenttämaa[2]; G. Kilaz[3]; R. Trice[1]

1. Purdue University, Materials Engineering, USA
2. Purdue University, Chemistry, USA
3. Purdue University, Aviation Technology, USA

Biofuels are a renewable fuel source for use in gas turbines employed for energy generation or aviation applications. However, these alternative fuels may contain the individual constituents found in calcium-magnesium-aluminum silicates or CMAS as result of how the biomass is harvested. Thus, critical components in a gas turbine engine, including blades and vanes, burning biofuels are exposed to impurities that may hasten their failure through a variety of different corroding mechanisms. In this study, the individual constituents of CMAS were sprayed onto air plasma spray (APS) and electron beam physical vapor deposition (EB-PVD) thermal barrier coatings (TBCs) and subsequently exposed to heating and cooling cycles at temperatures similar to those of a gas turbine. In each case, microstructure investigations revealed delamination of the yttria-stabilized zirconia (YSZ) topcoat that can be linked to infiltration and the formation of different phases as a consequence of the impurites. Elemental map analysis revealed evidence of primarily CaO and SiO_2 phases in the microstructure of the YSZ presumably which likely contributed to delamination.

4:30 PM

(ICACC-S2-029-2018) Thermal, mechanical properties and CMAS-resistance of ordered rare earth hafnates (δ-$Re_4Hf_3O_{12}$ Re = Yb, Lu) EBC candidates

W. Hu*[1]; J. Zhang[1]; J. Wang[1]

1. Institution of Metal Research, Chinese Academy of Sciences, Shenyang National Laboratory for Materials Science, China

Silicon-based ceramic composites are proposed materials for future gas turbine hot section components with improved engine efficiency and fuel consumption by increasing operating temperature. However, the harsh environment brings requirements for thermal and environmental barrier coating (T/EBC) with excellent temperature capability, thermal stability and durability, as well as corrosion-resistance of molten deposits of calcium-magnesium-aluminosilicate (CMAS). In the present work, we focused on the ordered phase δ-$Re_4Hf_3O_{12}$ (Re = Yb, Lu) and evaluated their capabilities to be used as EBC coatings. The bulk sample has been fabricated by hot-pressed sintering method and their mechanical and thermal properties were systematically investigated. They exhibited good high temperature elastic modulus retention and moderately matching thermal expansion coefficients with the silicon-based ceramic. The interactions of the hafnates with CMAS at 1300 and 1500 °C were studied respectively. HfO_2-based fluorites and apatite silicates were the main reaction products. The hafnate with larger ionic rare-earth radius exhibited better CMAS corrosion resistance and potentially applied in EBC system based on its high temperature stability.

4:50 PM

(ICACC-S2-030-2018) Evaluation of yttrium silicides to form in-situ EBCs in melt-infiltrated SiC CMCs

R. A. Golden*[1]; E. J. Opila[1]

1. University of Virginia, Materials Science and Engineering, USA

Current silicon melt-infiltrated (SMI) ceramic matrix composites (CMCs) are limited by the melting temperature of silicon (1414°C) and the volatility of the thermally grown SiO_2 scale in high-temperature water vapor environments. Replacement of the melt-infiltrated (MI) silicon with a rare-earth silicide offers the potential to address both limitations of SMI CMCs. This study focuses on the ability of yttrium silicides to form yttrium silicates (phases with greater stability in high-temperature water vapor than SiO_2) in high-temperature oxidizing environments. Yttrium silicides with compositions of 41, 67 and 95 at% Si-Y were fabricated using arc melting. Specimens were oxidized in air at 1000°C and 1200°C for times up to 24 hours. Rapid formation of a nonprotective Y_2O_3 scale and rejected Si was observed. Additional minor oxide phases of Y-Si-O, Y_2SiO_5, $Y_2Si_2O_7$ and SiO_2 formed on and beneath the specimen surface. Characterization of the microstructural evolution with time and temperature helped elucidate the diffusion mechanisms that control oxide growth rates. Replacement of MI silicon with a MI yttrium silicide would significantly compromise the high

temperature performance of a CMC due to Y_2O_3 CTE mismatch with SiC, high oxygen permeability and the large volume change associated with its rapid subsurface formation.

S3: 15th International Symposium on Solid Oxide Fuel Cells (SOFC): Materials, Science and Technology

Mechanical Stability I

Room: Crystal
Session Chair: Dario Montinaro, SOFCpower SpA

1:30 PM

(ICACC-S3-017-2018) The Implications of Creep Deformation of Anodes on the Durability and Reliability of Solid-Oxide Fuel Cells (Invited)

E. Lara-Curzio*[1]; A. Marquez[1]; A. Flores Betancourt[2]; E. Cakmak[1]; M. Lance[1]; B. L. Armstrong[1]; T. Burgess[3]; J. R. Bultman[4]

1. Oak Ridge National Lab, Materials Science & Technology Division, USA
2. University of California, Berkeley, USA
3. University of Tennessee, Knoxville, USA
4. University of Alabama, Tuscaloosa, USA

The failure rate of many engineering systems, including solid-oxide fuel cells (SOFCs), can be described by the bathtub curve in which failures in the third regime of the curve are associated with time-dependent degradation mechanisms. In this presentation, the creep deformation experienced by SOFC components will be analysed from a structural degradation perspective and the effect of creep deformation on the microstructure and functionality of Ni-YSZ anode materials will be discussed. The redistribution of internal stresses that occurs when cell materials exhibit different resistance to creep deformation will also be reviewed and the implications of internal stress redistribution on the reliability of SOFCs will be discussed.

2:00 PM

(ICACC-S3-018-2018) Chemo-mechanical effects influencing the mechanical integrity of SOFCs (Invited)

H. L. Frandsen*[1]; C. Chatzichristodoulou[1]; B. Charlas[1]; R. Kiebach[1]; K. Kwok[1]; P. Norby[1]; P. Hendriksen[1]

1. Technical University of Denmark, Department of Energy Conversion and Storage, Denmark

After manufacturing, solid oxide fuel cells (SOFC) experiences high residual stresses. Creep will over time relax these stresses. Here accelerated creep during reduction of the Ni(O)-YSZ anode is studied (~×10^4 faster than normal creep). Relaxation of the protective compressive residual stresses in the electrolyte significantly influences the integrity of the SOFCs. Creep rates were measured by bending and uniaxial tension - to exclude effects from asymmetric reduction. High deformation rates were found at simultaneous loading and shift of atmosphere - compared to different reference cases. The hypothesis for this chemo-mechanical phenomenon is a significant softening of Ni(O) leading to a drop of elastic modulus (×10), fast primary creep of YSZ phase, and release of residual stresses in the micro-structure. The hypothesis was investigated by measuring the stresses through the reduction in-situ by X-ray diffraction – by determining both macro-strains in multi-layered SOFCs, and micro-strains in the composite Ni(O)-YSZ anodes. The stress variation over temperature, through cell layers and in the micro-structure could also be understood using macro-scale and 3D micro-structural models. Stresses were found to practically reduce to zero at higher reduction temperatures (700-800°C). This confirms the hypothesis for accelerated creep. The implication is that the reduction temperature should be chosen with care.

2:30 PM

(ICACC-S3-019-2018) Classifying Heterogeneity in Porous, Composite Electrode Microstructures

W. K. Epting*[1]; T. Hsu[2]; R. Mahbub[2]; P. Salvador[2]; P. Ohodnicki[1]; H. Abernathy[1]; G. Hackett[1]

1. National Energy Technology Laboratory, USA
2. Carnegie Mellon University, Materials Science and Engineering, USA

To understand and predict the performance and degradation of solid oxide fuel cells (SOFCs), it is critical to understanding the microstructure of their porous, composite, ceramic-based electrodes, particularly the interfaces between phases and the triple phase boundaries. Valuable insight can be gained by characterizing these microstructures using 3D tomographic techniques, such as focused ion beam scanning electron microscopy and x-ray computed tomography. Through such 3D characterization, we have identified different sources of microstructural heterogeneity that exist in different length scales. These can include wide particle size distributions, aggregation of particles, poor mixing, rare but regular inclusions such as large particles or pores, and gradients in composition. To better understand the length scale on which these sources of heterogeneity affect the description of the cell, we have generated synthetic microstructures with deliberate variation of particle size distribution and the simulation of poor mixing. By comparing the resulting synthetic microstructures to large volumes of experimentally characterized microstructures, we find that it is necessary to consider multiple sources of heterogeneity across multiple length scales to approximate the structure – and therefore the performance and degradation – of an actual electrode.

Mechanical Stability II / Novel Processing and Design

Room: Crystal
Session Chair: Ayhan Sarikaya, Saint-Gobain

3:10 PM

(ICACC-S3-020-2018) Modeling performance degradation due to grain coarsening effects in solid oxide fuel cells

J. H. Mason*[1]; W. K. Epting[1]; Y. Lei[1]; I. Celik[1]; S. Lee[1]; H. Abernathy[1]; G. Hackett[1]

1. National Energy Technology Laboratory, US Department of Energy, USA

An integrated model utilizing multi-physics, phase field modeling and microstructure analysis is applied to predict performance loss as a function of time via particle coarsening in SOFCs. The multi-physics model is calibrated to experimental data of a button cell. Semi-empirical relations are used to model microstructural property evolution as a function of temperature. Performance predictions are then made for the entire cell lifetime (40,000 hours) at various temperatures. Then, reconstructed microstructures from SOFC electrodes obtained from PFIB analysis are coarsened as a function of temperature by the phase field model. The microstructure analysis tool outputs an array of properties which are applied within the performance model incrementally. Performance change over time due to coarsening is predicted by multi-physics model based on these properties. This is repeated for synthetically produced microstructures with varying initial particle size distributions. Predictions from this high-fidelity integrated model can then be used to improve the semi-empirical functional relations to improve accuracy of long term predictions.

3:30 PM

(ICACC-S3-021-2018) Phase Field Modelling of Microstructural Changes in Ni/YSZ Solid Oxide Electrolysis Cells Electrodes

M. Trini*[1]; S. De Angelis[1]; P. S. Jørgensen[1]; A. Hauch[1]; M. Chen[1]; P. V. Hendriksen[1]

1. Technical University of Denmark, Energy Conversion and Storage, Denmark

Solid oxide cells (SOC) are electrochemical devices that can be operated both in in fuel cell mode (SOFC) and electrolysis mode (SOEC). Understanding microstructural changes in SOECs is fundamental to investigate the degradation processes occurring during operation. Modelling these changes may show an important tool to improve the technology. Focused-ion-beam scanning electron microscopy tomography was used to characterize Ni/YSZ cermets which represent the state of the art material for SOC fuel electrodes. Different locations in a cell tested as part of a 25 cells stack for 9000 hours have been characterized in detail through indicators of microstructural degradation such as particle size distribution, triple phase boundary (TPB) length and network pathways. The aged cell is compared to a fresh cell (a cell from the same production batch but not long term tested). The porosity of the non-aged sample was ~25% and the overall TPB length ~2.4 $\mu m/\mu m^3$ of which 83% belongs to a percolated network. In the aged cell, the percolated TPB-length was significantly reduced (to ~0.76 $\mu m/\mu m^3$). The extent of the loss was observed to depend on the location relative to the steam inlet. In addition, phase field modelling was performed to simulate the microstructural evolution and extract degradation parameters. The experimental findings will be discussed on the basis of the simulation results.

3:50 PM

(ICACC-S3-022-2018) High Performance and Stability Based on Tri-layer Structure SOFC and SOEC (Invited)

M. Han*[1]

1. Tsinghua University, State Key Laboratory of Power Systems, Department of Thermal Engineering, China

To realize a sustainable energy future, wind and solar power has got a remarkable development in China, but energy storage is needed because of their intermittent characteristics. Moreover, advanced fossil fuel conversion technologies are also urgently needed. Solid oxide electrochemical cell (SOC) is a highly promising alternative for both energy storage and fossil fuel conversion due to its high efficiency and low emission. Degradation resulting from the electrolyte-electrode interface is a issue in both fuel cell and electrolysis mode for traditional cell. Here, a tri-layer structure cell with co-sintering technology is developed to mitigate the degradation. $La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_{3-\delta}$ is infiltrated to oxygen electrode backbone of a tri-layer NiO-YSZ|YSZ|porous-YSZ substrate. The 10cm×10cm cell gives a power output of 60W@0.7V-800 ºC, and shows long-term stability. In CO2/H2O co-electrolysis mode, a current output of $0.55Acm^{-2}$@1.3V-750 ºC is obtained. It suggests that $Gd_{0.2}Ce_{0.8}O_{2-\delta}$ (GDC) co-infiltration can improve the oxygen electrode stability through limiting the growing-up of nanoparticles, however, decrease the cell performance. Whilst, Ag co-infiltration can improve both electrochemical performance and stability. In addition, methane can be send to the oxygen electrode to reduce the electricity consume in electrolysis mode, which is a promising syngas production technology in future.

4:20 PM

(ICACC-S3-023-2018) Internal Methane Reforming for Efficient, High Power Density Solid Oxide Stack

S. Roychoudhury*[1]; C. Junaedi[1]; S. Vilekar[1]; T. LaBreche[1]; R. Mastanduno[1]

1. Precision Combustion, Inc., USA

Methane as direct fuel for SOFC has been widely investigated. Direct Internal Reforming SOFC (DIR-SOFC) could improve efficiency by avoiding external reformer and heat exchanger losses by close-coupling reforming and electrochemical reactions at the anode. Prior studies concluded that DIR-SOFC, without reforming catalyst in the anode, is challenging due to either carbon deposition promoted by traditional Ni-YSZ anode or the poor electrochemical activity of atypical Ni-free anodes. On-going research is focused on close-coupling a reforming catalyst with the anode, where CH_4 is reformed to produce H_2 and CO and is followed by the electrochemical reaction. Internal at-anode CH_4 reforming has been discussed for facilitating CH_4-fed SOFC operation with minimal carbon formation and providing additional cooling of the SOFC. Conventional DIR-SOFC approaches use catalysts located prior to the intercellular region. These approaches add bulk, have limited thermal ramp capability, require complex flow distribution patterns, and do not effectively dispatch heat. These challenges are circumvented by a PCI innovation, which improves stack heat management and carbon avoidance during internal CH_4 reforming. It addresses challenges with DIR-SOFC designs, including temperature uniformity, carbon deposition, and degradation of electrochemical activity. Preliminary test results and potential benefits will be presented.

4:40 PM

(ICACC-S3-024-2018) Challenges of processing and operating metal supported fuel cells

F. Thaler*[1]; D. Udomsilp[1]; A. Opitz[2]; V. Rojek-Wöckner[3]; M. Bram[1]

1. Christian Doppler Laboratory for Interfaces in Metal-Supported Electrochemical Energy Converters, Forschungszentrum Juelich GmbH, IEK-1, Germany
2. Christian Doppler Laboratory for Interfaces in Metal-Supported Electrochemical Energy Converters, Vienna University of Technology, Institute of Chemical Technologies and Analytics, Austria
3. Christian Doppler Laboratory for Interfaces in Metal-Supported Electrochemical Energy Converters, Germany

Metal supported fuel cells (MSCs) are attractive for non-stationary energy applications like fuel cell generators or range extender for battery electric vehicles. Contrary to conventional anode (ASC) or electrolyte supported (ESC) solid oxide fuel cells, MSCs promise improved mechanical stability and less production costs. As close cooperation between Forschungszentrum Jülich, TU Wien, Plansee SE and AVL List GmbH, the Christian Doppler Laboratory for Interfaces in Metal-supported Electrochemical Energy Converters contributes to the development of the Plansee MSC concept. A specific focus lies on the optimized processing of the electrodes aiming on increased electrochemical performance and long-term stability. Recently, significant progress in cell performance was achieved by optimizing the sintering procedure of the cathode, enabling to introduce LSC as cathode material of the Plansee MSC. An adapted sintering route using an atmosphere with controlled oxygen partial pressure, optimized particle size and sintering aids enabled to improve the sintering behaviour of the cathode in moderate temperatures. In parallel, Ni/GDC anode with optimized microstructure was introduced. Both measures resulted in a significant increase of electrochemical performance. The presentation summarizes the latest results including a general discussion of factors which must be considered to achieve improved long-term stability as well.

5:00 PM

(ICACC-S3-025-2018) Development of Metal-Supported Proton-Conducting Solid Oxide Fuel Cells by Reactive Spray Deposition Technology

R. Ouimet*[1]; T. D. Myles[3]; L. Bonville[2]; R. Maric[1]

1. University of Connecticut, Department of Chemical and Biomolecular Engineering, USA
2. University of Connecticut, Center for Clean Energy Engineering, USA
3. Health eSense, Inc., USA

In order to reduce both the operating temperature and the cost of solid oxide fuel cells (SOFCs), new materials and processes are required. By utilizing proton-conducting materials rather than the traditional anion-conducting materials, it is possible to lower the operating temperature of the SOFC from 800-1000°C to 500-600°C while using multiple fuels. Once the operating temperature is lowered, further cost reductions can be made by replacing the expensive materials currently used in SOFCs with low cost metal supports. This requires new processes to properly deposit the dense electrolyte and porous electrode microstructures on the metal support while preventing degradation of that support. One of these new processes is the Reactive Spray Deposition Technology (RSDT). With RSDT, it is possible to deposit a dense yttrium, ytterbium-co-doped barium cerium zirconate ($BaZr_{0.4}Ce_{0.4}Y_{0.1}Yb_{0.1}O_3$) electrolyte which can operate effectively at temperatures about 500-600°C, as well as a porous nickel-electrolyte cermet (Ni-$BaZr_{0.4}Ce_{0.4}Y_{0.1}Yb_{0.1}O_3$) anode, and a porous lanthanum strontium cobalt ferrite ($La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_3$) cathode. By introducing cooling applications to the process, it is possible to deposit these materials using the RSDT while keeping the metal support at an acceptably low temperature to prevent degradation of the support material.

S4: Armor Ceramics - Challenges and New Developments

Materials Characterization III

Room: Coquina Salon F

Session Chair: Jerry LaSalvia, Army Research Laboratory

1:40 PM

(ICACC-S4-022-2018) Influence of Amorphization on Residual Stress Development in Boron Carbide via Quasi-static and Dynamic Vickers Indentation

M. Coovert*[1]; G. Parsard[1]; G. Subhash[1]

1. University of Florida, USA

Boron carbide ceramics are promising for use in structural and armor applications due to their low density and exceptional hardness. When subjected to high shear loads however, the material experiences a collapse of crystalline order known as amorphization, which is accompanied by a decrease in mechanical performance. Micro-Raman spectroscopy is commonly used to confirm the presence of amorphization in boron carbide, through the presence of high wavenumber peaks in the Raman spectrum. This study intends to provide a link between the presence of amorphization and residual stress in boron carbide using a Raman spectroscopic volume mapping technique in the regions surrounding quasi-static and dynamic Vickers indentations. By repeatedly scanning an indented surface and polishing away a small amount of material, the amorphization volume beneath the indent is mapped. Further, a relationship between stress state and the shifting of the nominal 1088 cm^{-1} characteristic peak is developed and utilized to map residual stresses in the indentation volume. This use of piezospectroscopy reveals compressive stresses in the elastic matrix, offering insight into possible volume expansion due to amorphization, which aligns well with transmission electron microscopy imaging of indented boron carbide.

2:00 PM

(ICACC-S4-023-2018) Comparison of Amorphization Behavior in Boron Carbide and Boron Suboxide

G. Subhash*[1]

1. University of Florida, Mechanical and Aerospace Engineering, USA

Among icosahedral boron-rich solids, boron carbide (B4C) has garnered most attention due to its wide applicability in body armor. In recent years another promising ceramic with similar structure, boron suboxide (B6O), has received increased attention due to its properties being similar to those of B4C (e.g., low density and high hardness). However, there are also major differences in atomic structure and microstructure. While B4C has three-atom chains (e.g., CBC) which connect to icosahedra, B6O has two oxygen atom chain. B4C has many carbon-containing icosahedra [e.g., $(B_{11}C_p)$] and B6O exclusively has the more stable boron icosahedra [i.e., (B_{12})]. The closeness in boron and carbon size allows polymorphism in B4C, whereas the large size difference between boron and oxygen atoms makes B6O immune to polymorphism. Most importantly, B6O is prone to extensive nanotwinning (twins separated at nanometer distance) whereas nanotwins have not been observed in B4C. These differences render the amorphization mechanism to be dramatically different in both materials. Indeed, Raman spectroscopy reveals new peaks beyond 1200 cm-1 in amorphized regions of B4C, whereas the Raman spectrum of virgin and deformed B6O show no such differences although amorphous bands appear to be the same. Using HR-TEM and DFT, a detailed discussion is made on possible differences in mechanisms of amorphization in these materials.

2:20 PM

(ICACC-S4-024-2018) Evaluation of carbon additive on silicon carbide- boron carbide composites sintered by spark plasma sintering method

Z. Ayguzer Yasar*[1]

1. Rutgers University, Material Science and Engineering, USA

SiC and B_4C have outstanding properties such as very high hardness, a high Young's modulus, high melting point and low density. Those properties make SiC and B_4C valuable hard material, especially in the defence industry. Starck Alpha-SiC, Starck B_4C and carbon lampblack were used as starting materials. Powders were mixed with ball milling in ethanol for 24 hours and sintered by spark plasma sintering under vacuum 1950°C for 5min. Later on, density was measured by Archimedes method, elastic properties were checked by using ultrasound analysis, microstructure and grain size were measured by FESEM, Hardness. The highest relative density was 99.9% and obtained with 10% SiC-1.5%C-88.5%B_4C.

2:40 PM

(ICACC-S4-025-2018) Synthesis and Characterization of Dense Aluminum Dodecaboride - Based Ceramics with Enhanced Properties

T. Prikhna*[1]; R. A. Haber[2]; V. Domnich[2]; P. Barvitskyi[1]; S. Dub[1]; M. Karpets[3]; V. Muratov[3]

1. Institute for Superhard Materials of the National Academy of Sciences of Ukraine, Ukraine
2. The State University of New Jersey, Department of Materials Science and Engineering, Rutgers, USA
3. Institute for Problems in Material Scienceof the National Academy of Sciences of Ukraine, Ukraine

Lightweight ceramic materials based on aluminum dodecaboride (AlB_{12}) hold great potential for a wide range of applications, such as protective armor or constructional ceramics for nuclear power plants. Interest in higher aluminum borides and aluminum dodecaboride in particular exists for a long time. However, these materials have not found widespread use because of the lack of industrial and semi-industrial technologies for their powder production. Here, we

present the results of the investigation of the structure and mechanical properties of ceramic materials hot pressed from the submicton AlB_{12} powder with and without the addition of carbon (C) and titanium carbide (TiC). Temperature, pressure and composition on the starting powder are optimized to obtain sintered bodies with desired characteristics. X-ray diffraction, scanning electron microscopy and Raman microspectroscopy are utilized to investigate phase composition, bonding properties and microstructure of the sintered ceramics. The results of the measurements of micro- and nanohardness, fracture toughness, bending and compressive strengths demonstrate great potential of these materials for armor applications.

Synthesis and Processing I

Room: Coquina Salon F

Session Chair: Kristopher Behler, U.S. Army Reseach Lab

3:20 PM

(ICACC-S4-026-2018) Routes to formation of silicon doped boron carbide powder (Invited)

C. Besnard[1]; A. Bhowmik[1]; L. J. Vandeperre[1]; F. Giuliani*[1]

1. Imperial College London, Department of Materials, United Kingdom

Boron Carbide is an extremely hard ceramic material with potential as a light weight ceramic armor. However, under shock-loading conditions or non-hydrostatic unloading a pressure-induced amorphization has been observed. These experiments under non-hydrostatic conditions in the diamond anvil cell showed that the amorphization occurs from relatively modest pressures, as low as 25 GPa. Boron Carbide's strength against impact and therefore its applications as a superhard material are limited by this pressure-induced amorphization. It has been shown that doping of boron carbide can suppress this transformation under static loading conditions, however previous processing routes have only yielded small quantities of material. In this work we discuss processing routes that could be suitable for scale up allowing meaningful quantities of powder to be produced. Through the processing steps the structure and chemistry of the powder was monitored by SEM, XRD, Raman spectroscopy and XPS.

3:50 PM

(ICACC-S4-027-2018) Si-doping of Boron Carbide via Arc Melting

Q. Yang*[1]; A. U. Khan[1]; C. Hwang[1]; V. Domnich[1]; R. A. Haber[1]

1. Rutgers University, Materials Science and Engineering, USA

Silicon doping is predicted to be an effective strategy to mitigate amorphization in boron carbide. This opens up an avenue to design next generation body armor against high-speed threats. However, to fabricate sizable Si-doped boron carbide is challenging. In this work, we directly doped Si into boron carbide by melting them into liquid phase, in the attempt to diffuse more Si into boron carbide matrix. In this work, $B_{4<x<11.5}C$ were arc melted with 5 at. % of silicon (in Ar, >3500°C) to study the solubility of Si in boron carbide. Multiple melts were performed to ensure the homogeneity of the samples. Microstructural details were revealed by scanning electron microscopy (SEM). X-ray diffraction (XRD) and Raman spectroscopy were used to semi-quantify the Si concentration.

4:10 PM

(ICACC-S4-028-2018) Effect of sintering parameters on densification and properties of B_4C-TiB_2 composites densified via Spark Plasma Sintering

A. M. Celik*[1]; C. Hwang[1]; R. A. Haber[1]

1. Rutgers University, Materials Science and Engineering, USA

Boron carbide is an important hard material for lightweight armor applications due to its extreme hardness, low theoretical density and high Young's modulus. Titanium diboride is another significant hard material and it exhibits ultra-high melting temperature, good chemical stability and high fracture toughness. To combine above-mentioned properties in a hard ceramic, B_4C-TiB_2 composites could be manufactured. Various ratios of B_4C and TiB_2 powders were ball milled in ethanol for 24 hours using SiC media. The precursors then were sintered using various parameters under inert atmosphere via Spark Plasma Sintering (SPS) method to fabricate dense samples. Chemical analysis, phase determination, microstructure investigation, and mechanical property measurements were performed to evaluate bulk ceramic samples. B_4C-TiB_2 composites with promising mechanical properties were manufactured and tested.

4:30 PM

(ICACC-S4-029-2018) Industrial applications of Direct Current Based Spark Plasma/Field Assisted Sintering; Large Ceramic and Composite Parts for Armor Based Applications

L. S. Walker*[1]

1. Thermal Technology, USA

Direct current sintering (field assisted/spark plasma sintering) has nearly become the standard powder consolidation technique for new materials in academia, however it has seen limited industrial applications. The acceptance of the technology has been slow to evolve due to limited access to large industrial scale systems for proof of concept work and little research published on parts beyond 50mm. This work focuses on samples in the 100-200mm range of material systems relevant to industry for armor and other advanced applications such as B_4C, SiC, transition metal borides/carbides and ceramic matrix composites. Processing details including challenges in the scale up process, system setup for various materials and part uniformity will be discussed. In addition work on characterizing these materials for their microstructure and mechanical properties will be shared to show the impact of processing on material properties.

4:50 PM

(ICACC-S4-030-2018) Production of Functionally Graded Silicon Carbide-Titanium Diboride-Aluminium Composites by Spark Plasma Sintering Technique

M. Taner*[1]; G. Arslan[1]

1. Anadolu University, Material Science and Engineering, Turkey

In this study, production of functionally graded silicon carbide-titanium diboride-aluminium composites by spark plasma sintering technique was investigated. Silicon carbide-titanium diboride-aluminium powder mixtures containing 70-90 weight % silicon carbide-titanium diboride and 10-30 weight % aluminium were sintered at 1600-1900C for 5 minutes by spark plasma sintering. SiC-TiB_2-Al composites were sintered in a single step by using the spark plasma sintering method. Furthermore, the produced SiC-TiB_2-Al composites were designed as functionally graded composite material (FGM) layers having different compositions and/or residual porosity levels. Results obtained show that produced composite FGM's have a microstructure characterized by a co-continuous ceramic-metal network. Furthermore, it was determined that the bulk density of the produced functionally graded composite layers was always above 99,5 %, the hardness gradually decreased from the front layer to the back layer, while the compressive strength decreased in just the opposite manner.

S6: Advanced Materials and Technologies for Direct Thermal Energy Conversion and Rechargeable Energy Storage

Sodium Battery I

Room: Tomoka A

Session Chair: Palani Balaya, National University of Singapore

1:30 PM

(ICACC-S6-017-2018) New structures and new compositions of electrochemically active vanadium-based phosphates for Na batteries (Invited)

J. Chotard*[1]; C. Masquelier[1]; E. Boivin[1]; V. Kovrugin[1]; F. Chen[1]; T. Broux[2]; L. Nguyen[2]; F. Lalère[1]; V. Seznec[1]; O. Mentré[3]; F. Fauth[4]; R. David[1]; L. Croguennec[2]

1. University de Picardie Jules Verne, LRCS, France
2. ICMCB-CNRS, France
3. UCCS, France
4. CELLS-ALBA Synchrotron, Spain

Recent electrochemical and structural investigations of vanadium-based Na-containing phosphate compounds revealed the perspective of this chemical system for an exploration of new materials for advanced Na-ion batteries. In order to get an in-depth understanding of this system, a serie of new phases was synthesized and characterized by single crystal and operando powder X-ray diffraction analyses using laboratory and synchrotron X-ray sources, combined with galvanostatic electrochemical tests, XAS, NMR and EIS spectroscopies. A particular focus will be made on $Na_3V_2(PO_4)_2F_3$ for which a lot of discrepancies are reported throughout the literature considering its structure and electrochemical properties. In fact, most of the compounds reported are slightly oxidized due to synthesis conditions resulting in a partial oxygen substitution for fluorine, and thus in mixed valence within the bi-octahedral unit $V_2O_8F_{3-x}O_x$. Also, we investigated the substitution of a part of vanadium by aluminum or iron in the crystal structures of the studied compounds. As a result of our work in this research field, we discovered and electrochemically characterized a novel mixed-valence $Na_{11}V^{3+}(V^{4+}O)(P_2O_7)_4$ compound, and two groups of new M^{3+}-substituted phases, namely, $Na_7V_{4-x}Al_x(P_2O_7)_4(PO_4)$ (x = 2 and 1) and NASICON-type $Na_3V_{2-x}M_x(PO_4)_3$ (x = 1 [M^{3+} = Al, Fe], 0.75 and 0.25 [M^{3+} = Al]).

2:00 PM

(ICACC-S6-018-2018) Efficient Binders for Rechargeable Li/Na Batteries

N. Yabuuchi*[1]; S. Tanaka[2]; T. Narutomi[3]; S. Suzuki[3]

1. Tokyo Denki University and Kyoto University, Japan
2. Tokyo Denki University, Japan
3. Denka Company Ltd., Japan

Rechargeable lithium batteries consist of two different lithium insertion materials; positive and negative electrodes with electrolyte solution, in which a lithium salt is dissolved in aprotic solvent. Since lithium containing oxides for positive electrode materials are supplied as powder forms, polymer binders are used to make sheets of composite electrodes coated on aluminum current collectors. Recently, the polymer binders are being key materials to increase energy density and to improve cyclability of lithium batteries. In this study, polyacrylonitrile (PAN) possessing oxidation resistant properties is targeted as a base polymer, and is copolymerized by graft polymerization with poly(vinyl alcohol) (PVA) possessing high adhesive strength through strong hydrogen bonds of hydroxyl functional groups. Both polymers are known to be used as binder for positive electrode materials. The PAN-grafted PVA (PAN-g PVA) is a branched copolymer, and is used for a high-voltage spinel oxide for rechargeable Li batteries and oxide-based negative electrodes for rechargeable Na batteries. From these results, possibility of the branched copolymer as a non-fluorine binder is discussed for advanced rechargeable Li/Na batteries.

2:20 PM

(ICACC-S6-019-2018) $Na_2Mn_3O_7$: A suitable electrode material for Na-ion batteries?

E. Adamczyk*[1]; V. Pralong[1]

1. CNRS CRISMAT, France

Manganese based oxide materials are promising cathodes for sodium ion batteries due to their high energy density, low-cost and low-toxicity. Focusing on layered-type structures, the system Na–Mn–O is extremely rich. These materials are interesting because they show weak interlayer interactions with free space allowing sodium diffusion. The phase $Na_2Mn_3O_7$, synthesized by a conventional solid-state method, is a lamellar phase with [$Mn_3O_7^{2-}$] layers, built up with edge sharing MnO_6 octahedra, separated by NaO_6 and NaO_5 polyhedra. The charge discharge profiles of this material have been performed by a galvanostatic cycling at C/20 (1 Na^+ in 10h) in the potential window 1.5–3.0 V versus Na^+/Na. Starting from $Na_2Mn_3O_7$, a reversible capacity of 2 Na/f.u. (160mAh/g) is obtained through a plateau at 2.1 V with a low polarization of 100 mV. The PITT curve reveals a bell shape type response on the reversible phenomena illustrating a biphasic activity. Thus, the electrochemical process allows a reduced phase $Na_4Mn_3O_7$ to be obtained, which cans intercalate/de-intercalate two sodium per f.u. reversibly. The ex situ XRD patterns obtained for this reduced phase suggest a large atomic rearrangement with the formation of a new structure with the composition $Na_4Mn_3O_7$. The mechanism of insertion as well as the structures of the as-prepared and reduced phase will be discussed in this presentation.

2:40 PM

(ICACC-S6-020-2018) 100 Ah Sodium Nickel Chloride Cells for Efficient Energy Storage

M. Schulz*[1]; R. Weidl[1]; M. Hofacker[1]; B. Schüßler[1]; L. Kiesel[1]; M. Stelter[2]

1. Fraunhofer IKTS, System Integration and Technology Transfer, Germany
2. Fraunhofer IKTS, Germany

Sodium nickel chloride batteries represent a highly efficient and intrinsic safe energy storage technology. To break through the economical restrictions from expensive production efforts and sophisticated cell design a robust and simplified large cell was developed. The complete production chain from raw material to cell assembling was addressed by the developments. The conventional manufacturing of the ceramic solid electrolytes by isostatic pressing was transferred to highly productive cold plastic extrusion. A well suited water based feedstock for extrusion was developed. Rheological properties of the plastic masses were optimized by varying of organic binders, moisture contents and process parameter variation. The end capping of the electrolyte tubes was integrated in the extrusion process by a particular die design. Tubes with a diameter of 60 mm, a wall thickness of 2 mm and a length of about 600 mm were extruded successfully. The thin walled tubes were dried and sintered at 1600 °C. Gas tight electrolytes with densities higher than 97 % of the theoretical value and a ß” content of 94 % were reached. A cell for a nominal capacity of 100 Ah cells was designed under the objective of a maximized robustness and a minimized number of components. FEM simulations supported the constructive interpretation of the cell lid. First cells were assembled successfully and tested by charging and discharging experiments.

Sodium Battery II / Materials Characterization

Room: Tomoka A
Session Chair: Fei Chen, Wuhan University of Technology

3:20 PM

(ICACC-S6-021-2018) One Dimensional Nanomaterials for Emerging Energy Storage (Invited)

L. Mai*[1]

1. Wuhan University of Technology, State Key Laboratory of Advanced Technology for Materials Synthesis and Processing, China

One-dimensional nanomaterials can offer large surface area, facile strain relaxation upon cycling and efficient electron transport pathway to achieve high electrochemical performance. Hence, nanowires have attracted increasing interest in energy related fields. We designed the single nanowire electrochemical device for in situ probing the direct relationship between electrical transport, structure, and electrochemical properties of the single nanowire electrode to understand intrinsic reason of capacity fading. As the battery was charged and discharged repeatedly, lithium was progressively incorporated into the electrode, causing it to lose its crystalline structure and weakening its conductivity. Then, we identified the exciting electrochemical properties (including high electric conductivity, small volume change and self-preserving effect) and superior sodium storage performance of alkaline earth metal vanadates through preparing CaV_4O_9 nanowires. We also constructed a new-type carbon coated $K_{0.7}Fe_{0.5}Mn_{0.5}O_2$ interconnected nanowires through a simply electrospinning method. The interconnected nanowires exhibit a discharge capacity of 101 mAh g^{-1} after 60 cycles, when measured as a cathode for K-ion batteries. Our work presented here can inspire new thought in constructing novel one-dimensional structures and accelerate the development of energy storage applications.

3:50 PM

(ICACC-S6-022-2018) In Situ study of electrochemical processes at electrode/electrolyte interfaces (Invited)

T. Masuda*[1]

1. National Institute for Materials Science (NIMS), Research Center for Advanced Measurement and Characterization, Japan

In energy conversion devices such as fuel cells, batteries and photocatalysts, various important processes take place at electrode/electrolyte interfaces. Understanding of the interfacial processes is essential to design highly efficient and durable electrode materials. To date, various in situ techniques to investigate those interfacial processes under electrochemical potential control have been developed. In this talk, in situ x-ray absorption fine structure (XAFS) and atomic force microscopy (AFM) studies on fuel cell interfaces and development of an in situ electrochemical x-ray photoelectron spectroscopy (XPS) apparatus for the solid/liquid interfaces will be presented.

4:20 PM

(ICACC-S6-023-2018) Analysing operando spectroscopy data in battery studies: A chemometric approach (Invited)

L. Stievano*[1]

1. Université de Montpellier, ICGM - UMR 5253, France

Recently, a growing number of operando analyses have helped unraveling the electrochemical mechanism of lithium and post-lithium battery materials. The corresponding experiments usually lead to large amounts of data, requiring long and time consuming analyses. An alternative and innovating approach is using chemometric tools such as Principal Component Analysis (PCA) and Multivariate Curve Resolution (MCR). PCA is used to discover the minimal particular structures in multivariate spectral datasets. In the case of operando experiments, it allows determining the number of independent components contributing to the whole dataset collected during electrochemical cycling. The number of principal components can then be used as the basis for MCR analysis, which permits the stepwise reconstruction of the "real" spectral components. A detailed description of MCR is given by Tauler et al. who also proposed it for the analysis of in situ spectroscopic data, whereas its intrinsic limits are discussed by Ruckebush et al. In this presentation, we will show how such approach can be effectively applied to different techniques, such as Mössbauer and X-ray absorption spectroscopy, or transmission X-ray microscopy, for the comprehension of the electrochemical mechanisms in batteries.

S7: 12th International Symposium on Functional Nanomaterials and Thin Films for Sustainable Energy Harvesting, Environmental, and Health Applications

Synthesis, Functionalization and Assembly of 1D, 2D and 3D Nanostructures I

Room: Coquina Salon C
Session Chair: Scott Barnett, Northwestern Univ

1:30 PM

(ICACC-S7-017-2018) Science and Engineering of Nanodiamond Particles (Invited)

O. Shenderova*[1]; N. Nunn[1]; M. Torelli[1]; G. McGuire[1]

1. Adámas Nanotechnologies, USA

Despite that nanodiamond (ND) particles were discovered more than 50 years ago and were mass produced in the early 80s, for a long time they were in the shadow of their more famous sp^2 carbon cousins. Recent major breakthroughs including production of individual 4-5nm particles, NDs containing color centres exhibiting stable luminescence and advances in ND surface engineering have brought ND particles to the forefront of materials research. It is important to be aware, though, that the method of synthesis of NDs influence their structure (morphology, size, state of doping elements) and, therefore, other physical properties which ultimately determine their use in niche applications. Nanometer size particles are produced by detonation of carbon-containing explosives (called detonation nanodiamonds) or by grinding of microdiamond powders produced by phase transformation of carbon precursors under high pressure and high temperature (HPHT). While the former are a source of single-digit NDs, the latter are the basis for production of NDs containing color centers. A goal of this talk is to provide a general background on the synthesis and processing of ND particles, and to outline current and perspective applications of ND in areas which have broad societal impact such as polymer nanocomposites, nanolubricants and the biomedical field.

2:00 PM

(ICACC-S7-018-2018) Integrating energy storage and light harvesting: a melanin photocapacitor (Invited)

C. Santato*[1]

1. Ecole Polytechnque de Montreal, Canada

Eumelanin is a black-brown biopigment found in the human body, in other mammals, reptiles, amphibians and fishes as well as in invertebrates, such as cuttlefish and insects. Eumelanin extracted from the ink sac of cuttlefish is commonly called Sepia melanin. The heterogeneous eumelanin macromolecules are constituted by the building blocks 5,6-dihydroxyindole (DHI) and 5,6-dihydroxyindole-2-carboxylic acid (DHICA). The building blocks can polymerize at different positions. Different redox states of the building blocks (hydroquinone, semiquinone and quinone) coexist in eumelanin. Here we report on the cyclic voltammetric behavior, in acidic electrolyte buffers, of chemically controlled eumelanins

obtained from the solid-state polymerization of the DHI and DHICA building blocks as well as of natural eumelanin, Sepia. Eumelanin samples were fabricated on carbon paper electrodes and studied in electrolytes including monovalent (NH_4^+, Na^+, K^+) and divalent (Cu^{2+} Fe^{2+}) cations. Considering the optical properties of eumelanin, we decided to contribute to the development of biologically derived organic solar photocapacitors, integrating the energy storage and solar energy conversion functions. Exposing the eumelanin electrodes to simulated solar light clearly increased the charge stored by the melanin electrodes.

2:20 PM

(ICACC-S7-019-2018) Novel carbon-based catalyst for clean energy applications, specifically hydrogen generation (Invited)

D. Chua*[1]

1. National University of Singapore, Materials Science & Engineering, Singapore

Electrocatalysts are critical to increase reaction rates and control selectivity in many electrochemical reaction. Rational design and synthesis of material with controlled structures and morphologies from nanoscale to microscale are of utmost importance in order to achieve optimal performances. Today's catalyst goes beyond traditional metals such as Pt and Fe and specifically in the area of hydrogen generation (or hydrogen evolution reaction), we shall like to report on a new generation of 0D, 1D and 2D nanocomposites in the chalcogenide and phosphide family which shows very good promise. As a part of this electrochemical system strongly depends on the catalyst support. We further show that the formation of hybrid core-shell nanostructures utilizing carbon materials as a base template effectively enhance the performance and extend the range of applications. Other than HER, these applications include PEM fuel cells and Li batteries. For example, MoS2 coated graphene/carbon nanotubes formed excellent catalytical activity in HER and PEM fuel cell applications while MoS2 on carbon nanotubes are highly effective electron emitters.

2:40 PM

(ICACC-S7-020-2018) Effect of Reactive Additives on Polysiloxane Derived SiOC Porous Ceramics (Invited)

K. Lu*[1]; D. Erb[1]

1. Virginia Tech, USA

Silicon oxycarbide (SiOC) is a class of polymer-derived ceramics that enables the formation of a homogeneous structure at the molecular level starting from polymer precursors. Porous SiOC is a promising material for many applications such as electrodes in lithium ion batteries, catalyst supports, thermal barriers, gas separation membranes, and lightweight components. Because of the flexibility of molecular level composition and microstructure designs, the systems can be made porous with high specific surface areas by changing the precursor compositions and the ceramization conditions. In this study, in-situ conversion of tetraethyl orthosilicate (TEOS) into SiO_2 and its incorporation into silicon oxycarbide (SiOC) ceramics during the polysiloxane ceramization are investigated. The effect of the TEOS-derived SiO_2 on the phase development within the SiOC matrix at pyrolysis temperatures between 1100°C and 1400°C is studied using X-ray diffraction and nitrogen adsorption after etching the resulting SiOC with hydrofluoric acid. The SiOC with 10 wt% TEOS within the crosslinked polymer creates the highest specific surface area of ~2100 m^2/g. The ability to drastically alter the specific surface area of the ceramics through the use of pyrolysis atmosphere and the addition of organic additives presents a promising processing route for porous SiOC ceramics.

Functional Nanostructures for Energy Conversion and Storage and Catalysis II

Room: Coquina Salon C

Session Chairs: Olga Shenderova, Adámas Nanotechnologies; Clara Santato, Ecole Polytechnque de Montreal

3:20 PM

(ICACC-S7-021-2018) Green chemistry strategies for sustainable functional materials (Invited)

I. Concina*[1]

1. Luleå Tekniska Universitet, Sweden

Accessing sustainable energy applications through the use of nanomaterials calls for the development of strategies complying with the principles of green chemistry and enhancing functionality while at the same time reducing the costs for material and device production. This lecture focuses on vacuum free, cheap and scalable strategies delivering i) ZnO single-crystal nanostructures, ii) reduced graphene oxide@ZnO composites and iii) plasmonic metal nanoparticles@ZnO architecture to be applied as electrodes in solar energy converting devices and photocatalysts for water remediation. Emphasis will be given to the management of critical material properties such as light management and charge transport through the tuning of material aspect. The potential held by ZnO to act as a multifunctional platform for energy and environmental applications will be also discussed. Finally, the limits we are currently facing in these fields as materials scientists will be as well highlighted.

3:50 PM

(ICACC-S7-022-2018) Electrochemical Conversion of CO_2 In Solid Oxide Cells Utilizing Nano-Scale Electrodes (Invited)

S. Barnett*[1]

1. Northwestern Univ, USA

Solid oxide cells provide a highly efficient means for carrying out high-temperature electrolysis of CO_2 and CO_2/H_2 mixtures. Applications include renewable fuel production, electricity storage, CO production, and in situ resource utilization on Mars. This talk will focus on grid-sale storage of electrical energy, which is becoming an increasingly important problem as the utilization of intermittent renewable electricity sources increases. This talk describes reversible solid oxide cells (ReSOCs) that electrolyze H_2O-CO_2-rich mixtures to a CH_4-rich fuel. In order to reach cell performance targets at the desired operating temperature, nanoparticle-based electrodes are being utilized. The ReSOC fuel electrode catalyst material is nano-scale Ni, formed either by wet impregnation or exsolution of Ni from a perovskite oxide. The highest-performance oxygen electrodes contain nano-scale perovskite oxide catalysts such as $(Sm,Sr)CoO_3$. The long-term stability of the nano-scale electrodes at ReSOC operating temperature is discussed.

4:20 PM

(ICACC-S7-023-2018) Perovskite Solar Cells with Structure Engineering (Invited)

Q. Dai*[1]

1. Jackson State University, Physics, USA

Considering recent energy demand, it is obvious that the development of renewable energy sources and high efficient devices are indispensable. Solar cells are believed to be very promising to replace fossil energy to solve the energy crisis. Perovskite solar cells (PSCs) is very promising to dominate solar cell market due to their high efficiency. However, it is still challenging to prepare PSCs with high efficiency and high stability, for outdoor applications. Structure engineering including interface layer manipulation and nanoparticle incorporation are used to improve the stability and efficiency of PSCs. Moisture stability of the devices are improved by

hydrophobic layer induced into PSCs. UV light stability is enhanced by light converter, which can convert UV light into visible light. The converted light will be used by devices to achieve a higher efficiency. Metallic nanostructures are introduced into devices to increase the optical path length of the incident light leading to improved light harvesting and increased device performance. PSC efficiency was improved to 18% by structure engineering, which is very promising for practical applications. Solar energy storage effect is also obtained by integrating afterglow fluorescence nanomaterials into PSC devices to realize solar cells working in dark conditions.

4:40 PM

(ICACC-S7-024-2018) Synthesis and Characterization of Ni/GDC Nanostructured Cermet Catalysts for Hydrogen Production

A. Caravaca[1]; S. Picart[1]; B. Arab-Chapelet[1]; P. Vernoux[2]; T. Delahaye*[1]

1. CEA, DMRC, France
2. IRCE Lyon, France

In this study we developed novel materials based on Ni supported on $Ce_{0.8}Gd_{0.2}O_{2-d}$ (GDC), to be used in the H_2 production technology. GDC as catalyst carrier is known to limit the coke formation in H_2 production processes. However, previous studies dealing with the catalytic reforming of methane over Ni/GDC materials (Ni loading ~10 %) show important structural limitations, such as the high particle size of the Ni particles (~ 30 nm), and their low specific surface area (~20-40 m^2/g). In this work we developed novel Ni/GDC cermet (ceramic-metal) materials by the Weak Acid Resin (WAR) method. This synthesis procedure allows to prepare Ni/GDC materials in one step. In addition, in order to enhance the surface area of these materials, Ni was partially dissolved. The whole procedure led to catalysts with high metal loadings (≥ 10 %), small Ni nanoparticles (< 10 nm), and high surface areas (> 70 m^2/g), exhibiting therefore promising properties in view of their further utilization for the H_2 production technology.

5:00 PM

(ICACC-S7-025-2018) Au@Cd1-xZnxSe Yolk@Shell Nanocrystals with Tunable Compositions for Photocatalytic Hydrogen Production

J. Wu*[1]

1. National Chiao Tung University, Taiwan

The yolk@shell nanocrystals with void space offer large surface area and homogeneous environment for reactant molecules, both of which are favorable for photocatalytic reactions. Here we demonstrated the use of Au@Cd1-xZnxSe yolk@shell nanocrystals for practical photocatalytic hydrogen production. The samples were prepared by conducting anion exchange reaction on Au@Cu2Se yolk@shell nanocrystals which were obtained by performing sulfidation treatment on the initial Au@Cu2O core@shell nanocrystals. The superiority of Au@Cd1-xZnxSe was attributed to the pronounced charge separation caused by the Au core as well as the abundant active sites endowed by the yolk@shell structures. The varied composition of Cd1-xZnxSe further endows tunable band structure, extending solar spectrum harvesting to promote carrier generation.

S8: 12th International Symposium on Advanced Processing and Manufacturing Technologies for Structural and Multifunctional Materials and Systems (APMT12)

Unique Processing I

Room: Coquina Salon A
Session Chairs: Tohru Suzuki, National Institute for Materials Science; Satoshi Tanaka, Nagaoka University of Technology

1:30 PM

(ICACC-S8-015-2018) Electrospinning as manufacturing technology for bioactive calcium phosphate layer deposition from biogenic raw materials (Invited)

K. Balazsi*[1]; C. Balazsi[1]

1. Centre for Energy Research HAS, Hungary

There are more than 400 000 hip joint operations made every year in the word and there are some 25 000 000 people who have either a partial or a total hip replacement. It has been estimated that the aged people population has increased tremendously in recent past and there will be seven times increase. Whit time, the wear and risk of the implant loosening increases so that after 10 years 10-20% of the implants have to be renewed. The materials used for artificial implants by default are TiAl6V4, Ti alloy and ceramics. Despite application of different biocompatible materials with properties of native joints has not been found to date. Biomaterials used for implant should possess some important properties in order to long-term usage in the body without rejection and should be made with properties as excelent biocompatibility, superior corrosion resistance and non-toxic. Ceramic structures at the nanometre range have been proven to have improved properties and characteristics that differ from their bulk, allowing for opportunities in novel technological applications. Ceramic layers with some advantages, including a very high surface-to-volume and aspect ratio can be processed by cheap and quick method; electrospinning. This presentation reviews results on the electrospinning of various bioceramic lyaers their potential applications in the biomedical field.

2:00 PM

(ICACC-S8-016-2018) Mechanical properties of alumina with crystalline orientation controlled by a magnetic field

T. S. Suzuki*[1]; T. Uchikoshi[1]; B. Kim[1]; K. Morita[1]; Y. Sakka[1]

1. National Institute for Materials Science (NIMS), Japan

Tailored microstructure in materials is important to improve their mechanical and functional properties. Crystallographic orientation is one of the effective way in ceramics as well as metals. We already reported that crystalline orientation can be controlled by a magnetic field even in diamagnetic ceramics. Ceramics particles with asymmetric crystal lattice structure show anisometric magnetic susceptibility. When the ceramics particles dispersed in the solvent are located in the strong magnetic field, the particles were rotated to an angle minimizing the system energy by a magnetic torque generated from the interaction between the magnetic anisotropy and the applied magnetic field. After consolidation of alumina slurry in a magnetic field, the compact bodies are sintered by electric furnace or SPS without a magnetic field. The c-axis of alumina was aligned uniaxially parallel to the magnetic field. The bending strength of textured alumina was higher than that of random alumina at room temperature and high temperature. High temperature deformation of alumina depends on the orientation direction and enhanced to the direction at 45-degree angle to the crystallographic orientation.

2:20 PM

(ICACC-S8-017-2018) Magnetic freeze casting inspired by nature

J. McKittrick*[1]; M. B. Frank[3]; M. Porter[2]; M. Meyers[1]

1. UC San Diego, USA
2. Clemson University, Mechanical Engineering, USA
3. nanoComposix, USA

Magnetic field aligned freeze casting is a novel method to fabricate porous, anisotropic ceramic scaffolds with a hierarchy of architectural alignment in multiple directions. A weak rotating magnetic field applied normal to the ice growth direction in a uniaxial freezing apparatus allowed the manipulation of magnetic nanoparticles to create different pore structures and channels with long-range order in directions parallel and perpendicular to the freezing direction. Porous scaffolds consisting of different host ceramics as particles or platelets (hydroxyapatite (HA), ZrO_2, Al_2O_3, or TiO_2) mixed with varying concentrations (0–9 wt%) of Fe_3O_4 nanoparticles were fabricated by freeze casting under no magnetic field and a static magnetic field of 0.12 T. The HA, ZrO_2, and Al_2O_3 scaffolds showed biphasic material properties with separate Fe_3O_4-rich and Fe_3O_4-poor regions. The TiO_2 scaffolds showed homogeneous distributions of Fe_3O_4 throughout the macrostructures, which resulted in aligned pore channels parallel to the magnetic field, normal to the ice growth direction. In the magnetic field direction, the compressive strength and stiffness of the scaffolds containing was doubled. The enhanced mechanical performance of the field aligned scaffolds are the result of the long-range microstructural order in multiple directions—(1) the magnetic field direction and (2) the ice growth direction.

2:40 PM

(ICACC-S8-018-2018) Grain-oriented polycrystalline transparent alumina ceramics prepared by colloidal processing

S. Tanaka*[1]

1. Nagaoka University of Technology, Materials Science and Technology, Japan

Grain-oriented alumina ceramics with high transparency was successfully fabricated through colloidal processing in a high magnetic field and sintering with hot isostatic pressing. Until now, many efforts have been taken place to improve its optical property. However, the transparency is limited by light scattering at the grain boundaries due to the birefringence of alumina crystals. The objective of this study was to fabricate highly transparent grain-oriented alumina ceramics through processing in a magnetic field with subsequent hot isostatic pressing (HIP). Very fine α-alumina powders were used as raw materials. An alumina slurry with a high solid concentration of 45 vol.% was prepared in an aqueous system. As dispersant, Tiron reagent with a concentration with respect to alumina was used. The slurry viscosity was optimized using a rheometer. A plastic vessel (ϕ:25 mm) containing slurry was placed in magnetic field of 10 T, which was induced by a superconducting magnet. C-axis of each particle in the slurry oriented to parallel to the magnetic field. The dried specimens were vacuum-sintered at 1350 °C for 1 h and followed by the final HIP process at 1300 °C to arrest grain growth. Result shows the in-line transparency of grain-oriented alumina specimens 78% at wave length 650 nm. Their in-line transmission was 84.5% to that of sapphire single crystal.

3:20 PM

(ICACC-S8-019-2018) The Effect of Tape Casting Parameters on TGG Texture Alignment

R. L. Walton*[1]; M. D. Vaudin[2]; A. Hofer[3]; E. R. Kupp[1]; G. L. Messing[1]

1. Pennsylvania State University, Materials Science and Engineering, USA
2. National Institute of Standards and Technology, USA
3. MontanUniversityet Leoben, Austria

Textured ceramics for use in structural and electronic applications can be evaluated by texture fraction and degree of alignment. To date there has been almost no research on texture alignment during texturing processes. This paper reports how tape casting parameters and rheology influence the alignment quality for a TGG textured alumina model system. Using rocking curves to analyze alignment, we show that viscosity and the slope of the velocity gradient are the primary factors affecting template alignment. The results are explained with a Couette flow model and torque on the template particle. The highest quality texture is measured when the torque on the template particles is not severe enough to cause over-rotation of the particle, but the torque applied is enough to orient the particle. A higher viscosity slurry will increase this torque limit, as it will be more difficult to rotate a particle in a higher viscosity slurry.

3:40 PM

(ICACC-S8-020-2018) Robocasting of solid state sintered SiC

J. Teo*[1]; L. J. Vandeperre[1]; E. Saiz[1]

1. Imperial College, Materials, United Kingdom

Additive manufacturing of ceramics is gaining substantial interest especially for complex parts and small production volumes. Robocasting is one of such additive manufacturing processes for ceramics and consists of the continuous extrusion of filaments of ceramic paste. This is followed by drying and sintering. While in the past robocasting has been used extensively for porous scaffolds, it is now increasingly being investigated for the production of complex shaped, dense, parts too. In this paper, the robocasting of solid state sintered silicon carbide will be described. Previous work on robocasting of silicon carbide used oxide additives resulting in liquid phase sintering. This reduces the resistance to creep for high temperature use and therefore in this work a robocasting slurry was developed for solid state sintered SiC using 5 wt% B4C and 2 wt% carbon as sintering additives. Aqueous slurries containing in between 38 and 45 vol% solids and in between 18 and 25 wt% Puronic F127 were prepared and used in printing tests to optimise the slurry. The Robocasted SiC parts were then pressureless sintered to full density at temperatures up to 2200 °C. The hardness, elastic modulus and flexural strength of the samples are reported.

4:00 PM

(ICACC-S8-021-2018) Low temperature synthesis and electrochemical characterization of $LiMn_2O_4$ prepared by a polymeric steric entrapment precursor route

D. Ribero*[1]; K. Tseng[1]; W. Luo[1]; S. J. Dillon[1]; W. M. Kriven[1]

1. University of Illinois, Materials Science and Engineering, USA

A nano/microscale and pure crystalline $LiMn_2O_4$ (LMO), was synthesized in a low temperature single step, using the polymeric steric entrapment method, from precursor chemicals of $LiNO_3$ and $Mn(NO_3)_2 \bullet 4H_2O$ stoichiometrically dissolved in distilled water. An 80% hydrolyzed polyvinyl alcohol (–[CH_2–CHOH]-n or PVA) was used as the organic carrier for the precursors, which served as entrapper of the metal ions, fuel for the exothermic reactions and reducing agent for the intermetallic cation (Mn). The influence on the physical/chemical/electrochemical characteristics of the powders by processing variables such as drying procedures, amount of polymer (cation:organic ratio), characteristics of the exothermic reaction (C/N ratio), calcination/crystallization atmosphere and temperature, were investigated. The powders synthesized at 300°C (nano-sized), 500°C, 700°C (micron-sized) exhibited specific surface areas of 14.6 m^2/g, 12.3 m^2/g and 6.4 m^2/g, respectively. The specific capacities evaluated at C/10 for the 300°C, 500°C and 700°C powders were 93.3 mAh/g, 110 mAh/g 131.8 mAh/g, respectively. In all cases the powders produced in this work performed similar or even better than the commercial LMO) powders measured under the same conditions.

4:20 PM

(ICACC-S8-022-2018) Low-temperature synthesis of micron-sized $LiNi_{0.5}Mn_{1.5}O_4$ particles by solid-state reaction in water vapor

D. Hirobe*[1]; T. Kozawa[1]; M. Naito[1]

1. Osaka University, Joining and Welding Research Institute, Japan

$LiNi_{0.5}Mn_{1.5}O_4$ is one of the promising high-voltage cathode materials for high-energy lithium-ion batteries (LIBs). However, traditional organic electrolytes suffer from oxidative decomposition owing to the high working voltage of $LiNi_{0.5}Mn_{1.5}O_4$. Hence, the $LiNi_{0.5}Mn_{1.5}O_4$ cathode with a small particle size, which has a large surface area, causes a fall in cycle performance of LIBs. Although the micron-sized $LiNi_{0.5}Mn_{1.5}O_4$ powder can be produced by a solid-state reaction, this method requires high calcination temperatures. Moreover this high-temperatures heating leads to the formation of secondary phases by partialy oxygen release from $LiNi_{0.5}Mn_{1.5}O_4$. Here, we propose the solid-state reaction in water vapor for producing micron-sized $LiNi_{0.5}Mn_{1.5}O_4$ powders at a lower temperature than in air. In a water vapor atmosphere, the formation of $LiNi_{0.5}Mn_{1.5}O_4$ particles and the subsequent particle growth were promoted at low temperatures. Better electrochemical performances of LIBs were exhibited for the $LiNi_{0.5}Mn_{1.5}O_4$ cathode obtained in water vapor than that in air.

4:40 PM

(ICACC-S8-023-2018) Study of the tape casting-calendaring process for fabricating ceramic substrate

S. Wang*[1]; H. Lan[1]; W. Wang[1]; Y. Huang[1]

1. Shantou University, Department of Mechatronics Engineering, China

Due to the advantages of high thermal conductivity, good insulation, compatible thermal expansion coefficient with the chip, ceramic substrate has been paid great attention in the recent years. The forming process of ceramic has a great influence on the quality and performance of ceramic substrate. Based on the application of ceramic substrate for electric package, a new technology combined tape casting and calendaring process for preparing ceramic substrate was proposed in this paper. Micro and nano multi-scale ceramic powders were mixed as raw material. The ceramic green tape with micro-nano hierarchical structure was prepared by tape casting plus calendaring process. Finally, the ceramic substrates were sintered under 1573 to 1873K. The microstructure and properties of the ceramic substrates prepared by tape casting-calendaring process were studied. Experimental results showed that the Al_2O_3 ceramic substrate sintered at 1873K with no additives exhibits good performance with relative density of 96.9%, z-axis shrinkage rate of 11.65%, dielectric constant of 9.5, thermal conductivity of 23.8W/(MK).

S12: Advanced MAX/mxene Phases and UHTC Materials for Extreme and High Temperature Environment

Different Perspectives on Designing of MXenes

Room: Tomoka B

Session Chair: Surojit Gupta, University of North Dakota

1:30 PM

(ICACC-S12-021-2018) MXene: What we know and do not know about their surface terminations (Invited)

M. Barsoum*[1]

1. Drexel University, Materials Science and Engineering, USA

The 2D early transition metal carbides known as MXenes - obtained by etching the A-layers from the MAX phases - were discovered in 2011 and have generated substantial interest in the scientific community because of their potential in an ever expanding host of applications. Unlike hydrophobic graphene, MXenes are hydrophilic and behave as "conductive clays" a hitherto unknown combination. Along the same lines, because the density of states at the Fermi level is substantial and the electric conductivity, for the most part, metal-like, MXenes can be considered as 2D metals that are surface terminated by OH and similar groups. Said otherwise, they posses a metallic core with a hydroxide shell. The surface terminations that are typically a combination of O, OH, and/or F. It is for this reason that the proper chemical description is $M_{n+1}X_nT_x$, where T represents a surface termination. Quantifying and understanding the latter is non-trivial, but key to many applications and scientific understanding. Using careful XPS analysis, ion exchange experiments and others, it is possible to quantify the surface terminations and show that for the most part the sum of the negative charges corresponding to the surface terminations depend on MXene chemistry. The ramifications of these insights on ion exchange capabilities, delamination, energy storage, and interlayer spacings are important and will be discussed.

2:00 PM

(ICACC-S12-022-2018) Prediction and synthesis of a family of MAX phases and MXenes with in-plane chemical ordering (Invited)

J. Rosen*[1]

1. Department of Physics, Chemistry and Biology, Sweden

The exploration of two-dimensional solids is an active area of materials discovery. A more recent addition to the 2D world are MXenes, obtained by selective etching of the A-layer from parent MAX phases (where M is a transition metal, A is an A-group element, X is C and/ or N). Recently, a new type of atomic laminated phases coined i-MAX was reported, in which the M-atoms in $(M1_{2/3}M2_{1/3})_2AlC$ are chemically ordered. The first phase discovered was $(Mo_{2/3}Sc_{1/3})_2AlC$, and we here show that this was a first example of a potentially large family of stable phases, exemplified through theoretically predicted and experimentally verified $(V_{2/3}Zr_{1/3})_2AlC$, $(Mo_{2/3}Y_{1/3})_2AlC$, and $(Cr_{2/3}Sc_{1/3})_2AlC$. Using i-MAX for MXene synthesis allows a new route for tailoring the MXene structure and composition: By employing different etching protocols, we can i) remove only the Al atoms, and obtain a MXene with in-plane elemental order, or ii) remove Al and one of the M-elements, and obtain a MXene with ordered vacancies. We show that the difference in the MXene structure and composition have a drastic influence on, e.g., transport properties and potential for energy storage applications. One example is $Mo_{1.33}C$ with vacancies, which exhibits a 65% higher volumetric capacitance than its counterpart, Mo_2C, without vacancies. This materials design on atomic level expands the property tuning potential of MXenes.

2:30 PM

(ICACC-S12-023-2018) MXenes as Host Materials for Ions (Invited)

M. Naguib*[1]

1. Tulane University, Physics and Engineering Physics, USA

MXenes are two-dimensional transition metal carbides and carbonitirdies with composition of $M_{n+1}X_nT_z$ where M is an early transition metal (e.g. Ti, V, Mo, Nb) and X is either carbon or nitrogen, T_z stands for a mixture of surface terminations (e.g. O, OH, F, Cl), and n can be 1, 2, or 3. They exhibit an excellent electrical conductivity, excellent capability to host various ions in-between the layers and redox active sites on the surface. Thus, they can be used as electrode materials for various ion batteries (e.g. Li, Na, K) and Pseudocapacitors with an excellent cyclability to handle high cycling rate. For example, Mo_2C showed a reversible capacity of 570 mAh/g at a specific current of 400 mA/g when tested as anode material in Li-ion batteries (LIBs). At very high specific currents such as 5 A/g and 10 A/g, reversible capacities of 250 mAh/g and

75 mAh/g, respectively, were found to be stable over 1000 cycles. In this presentation I will focus on the performance of MXenes as electrode material hosting ions for batteries and the effect of ions intercalation on the structure and behavior of MXenes.

Novel Synthesis Paradigm and Unique Properties of Mxenes-I

Room: Tomoka B
Session Chairs: Michael Naguib, Oak Ridge National Laboratory; Konstantina Lambrinou, SCK-CEN

3:20 PM

(ICACC-S12-024-2018) Ordered quaternary MAX phases and their 2D ordered double-transition metal carbide MXenes (Invited)

B. Anasori*[1]; Y. Gogotsi[1]

1. Drexel University, Materials Science and Engineering, USA

In 2014, a new chapter was added to the story of MAX phases, when ordered quaternary MAX phases were discovered, in which two transition metals occupy separate atomic layers in the $M_{n+1}X_n$ of MAX phases (n = 2, 3). For example, when Mo and Ti elements are mixed to make M_3AlC_2, an ordered phase is formed, in which a layer of Ti atoms is sandwiched between two layers of Mo atoms, instead of a random mix of Ti and Mo in the three M layers of the M_3C_2. In three years, more than thirty ordered quaternary MAX phases of $M'_2M''AlC_2$ and $M'_2M''_2AlC_3$ have been predicted and about 10 of them have already been synthesized. All these phases have only aluminum as their A element, which implies several more ordered MAX phases are waiting to be explored and synthesized. By selective etching of the aluminum layers, two-dimensional (2D) double-transition metal carbides (MXenes) have been made, such as Mo_2TiC_2, Mo_2ScC_2, Cr_2TiC_2, and $Mo_2Ti_2C_3$, significantly expanding the family of 2D carbides. By controlling the atomic layer ordering in MXenes, we can further tune their electronic, optical, electrochemical, and magnetic properties. In this talk, we present the structure, and stability of ordered quaternary MAX phases followed by the properties and applications of their ordered double-M MXenes.

3:50 PM

(ICACC-S12-025-2018) Thermal Transport Properties of Functionalized MXene monolayers (Invited)

D. Cakir*[1]; S. Sarikurt[2]; C. Sevik[3]; M. Keceli[4]

1. University of North Dakota, Physics and Astrophysics, USA
2. Dokuz Eylul University, Turkey
3. Anadolu University, Turkey
4. Argonne National Lab, USA

MXenes are the newest class of 2D materials, and they offer a great potential in thermoelectric. In this study, we investigated the lattice thermal transport properties of two different functionalization models (model 1 and model 2) of M2CO2 (M=Ti, Zr, Hf, Sc) MXenes using Density Functional Theory and the Phonon Boltzmann Transport Theory. Due to the coupling among different phonon modes, Peierls-Boltzmann transport equation is always solved under some approximations, namely single mode relaxation time approximation and iterative solution. We used both approximations to calculate thermal transport properties of MXenes. We found that thermal conductivity strongly depends on the structural model of MXene and type of metal atom. For instance, thermal conductivity of model 2 structure of Ti2CO2 is about three times larger than that of model 1. By changing absorption site of functional groups, we can significantly tune thermal conductivity. In other words, the surface chemistry tunability of MXenes monolayers gives us an opportunity to modify thermal properties of this class of 2D materials in a controllable manner, which is absent in other 2D crystals. Model 2 configuration of Ti2CO2 has the lowest thermal conductivity as compared to the same configurations of Zr2CO2 and Hf2CO2. Our calculation suggested that Ti2CO2 would be a good candidate for next-generation thermoelectric applications.

4:20 PM

(ICACC-S12-026-2018) Thermodynamic Studies of MAX and MXene Phases (Invited)

G. Sharma*[1]; M. Naguib[2]; E. Muthuswamy[1]; D. Wu[3]; Y. Gogotsi[4]; A. Navrotsky[1]

1. University of California Davis, Peter A. Rock Thermochemistry Laboratory and NEAT-ORU, USA
2. Oak Ridge National Laboratory, Materials Science and Technology Division, USA
3. Washington State University, Pullman, The Gene and Linda Voiland School of Chemical Engineering and Bioengineering, USA
4. Drexel University Philadelphia, Department of Materials Science and Engineering, USA

Recently, MXenes have garnered significant attention because they offer unique combination of metallic conductivity in the sheets, ion exchange and hydration between the layers, and show attractive electrochemical properties. Here, we report enthalpies of formation from the elements measured using high temperature oxide melt solution calorimetry for a layered MAX phase, and the corresponding clay-like MXene. The thermodynamic stability of the MXene is assessed by calculating the enthalpy of reaction in aqueous solution. Our results show that, the surface terminations and cations chemisorbed on the surface and in the interlayers play a major role in the thermodynamic stabilization of MXene. This work also investigates the energetics of alkali ion exchange in clay-like MXenes using aqueous solution immersion calorimetry which suggests that K^+ ions interact more strongly with anions present in the interlayers than Na^+ and Li^+ ions. Water vapor adsorption calorimetry indicates very weak interaction of water with the MXene while immersion calorimetry suggests weakly hydrophilic nature of MXene surface.

4:50 PM

(ICACC-S12-027-2018) Chemical Origin of Termination-Functionalized MXenes: $Ti_3C_2T_2$ as a Case Study

T. Hu*[1]; Z. Li[1]; M. Hu[1]; J. Wang[1]; Q. Hu[1]; Q. Li[2]; X. Wang[1]

1. Institute of Metal Research, Chinese Academy of Sciences, China
2. Yantai University, The Laboratory of Theoretical and Computational Chemistry, School of Chemistry and Chemical Engineering, China

MXenes represents a burgeoning family of two-dimensional (2D) functional materials with a variety of applications that highly rely on termination-mediated surface functionalization, but the understanding of termination is limited. Here, we take $Ti_3C_2T_2$ (T = O, F, OH and H) as an example of MXenes, to demonstrate how termination stabilizes the Ti_3C_2 monolayer matrix through saturating the nonbonding valence electrons of surface Ti atom by the low-energy orbitals of the termination. This is achieved by orbitally resolved density of states analysis through simply yet efficiently manipulating the internal coordination of octahedral crystal field to match exactly the Cartesian coordination. Highly degenerate 3d orbitals of surface Ti split in such a way that exhibits pseudogaps whose widths predict a stability order: $Ti_3C_2O_2 > Ti_3C_2F_2 > Ti_3C_2(OH)_2 > Ti_3C_2H_2 > Ti_3C_2$, consistent well with Bader charge analysis, thermodynamic calculations and experimental results. This new criterion could get implications in the general context of ubiquitous termination phenomenon of MXenes and other relevant termination-functionalized 2D materials.

S13: Advanced Ceramics and Composites for Nuclear Fission and Fusion Energy

Novel Ceramics for Nuclear Energy

Room: Coquina Salon H
Session Chairs: Takaaki Koyanagi, Oak Ridge National Laboratory; Sosuke Kondo, Kyoto University

1:30 PM

(ICACC-S13-019-2018) ATF material development of SiC with enhanced safety LWR core (Invited)

K. Kakiuchi*[1]; M. Akimoto[1]; S. Suyama[1]; M. Ukai[1]; H. Heki[1]; A. Kawaguchi[2]; T. Takagi[2]; Y. Sato[3]; Y. Taniguchi[3]; T. Goto[4]; Y. Kagawa[5]; S. Yamashita[6]

1. Toshiba Corporation, Japan
2. IBIDEN Co., LTD., Japan
3. Nuclear Fuel Industries Ltd., Japan
4. IMR Tohoku University, Japan
5. Tokyo University of Technology, Japan
6. Japan Atomic Energy Agency, Japan

Toshiba has been conducting ATF development, involving Japanese industries and national universities since 2008. We identified that a silicon carbide (SiC) ceramic will be the most promising candidate for ATF material based on the comprehensive feasibility studies. The development is accelerated by the lessons of the Fukushima Daiichi nuclear power plant accident. We and the project partners are making efforts to develop SiC/SiC composite for ATF material. During the period (Phase-1) from FY2012 to FY2015, feasibility studies on material design and fabrication were performed. In the period (Phase-2) from FY2016, the irradiation in the test reactor is under consideration. The commercial applications of the SiC/SiC composite core components in nuclear power plants during the 2020s will be planed. This presentation will be included the principal results and the current issues through a comprehensive feasibility study of SiC core application for LWR.

2:00 PM

(ICACC-S13-020-2018) Radiation damage behavior in a multiphase ceramic (YSZ, Al_2O_3 and $MgAl_2O_4$) irradiated with 946 MeV Au ions

K. Ohtaki*[1]; K. K. Karandikar[2]; O. Graeve[2]; C. Trautmann[3]; M. Tomut[3]; M. Patel[4]; M. Mecartney[1]

1. University of California, Irvine, Chemical Engineering and Materials Science, USA
2. University of California, San Diego, Department of Mechanical and Aerospace Engineering, USA
3. GSI Helmholtzzentrum für Schwerionenforschung GmbH, Secretariat of Materials Research Department, Germany
4. University of Liverpool, Department of Mechanical, Materials and Aerospace Engineering, United Kingdom

This study focuses on engineering a composite with high radiation damage tolerance without relying on intrinsic material properties. Radiation damage tolerance is reported to be improved in polycrystalline materials with nano-sized grains because grain boundaries act as sinks for defects. Moreover, disorder at the grain boundaries in multiphase systems is expected to increase the radiation damage tolerance. An equivolume 3-phase ceramic material of cubic 8 mol% yttria stabilized zirconia (YSZ), α-Al_2O_3 and $MgAl_2O_4$ with nano-sized grains (~260 nm) and single phase nanocrystalline Al_2O_3 and $MgAl_2O_4$ (~260 nm) were prepared by spark plasma sintering and then irradiated with 926 MeV Au ions with fluence up to $2x10^{12}$ ions/cm^2 at RT. The nanocrystalline grain size and the disordered interfaces between dissimilar phases are hypothesized to enhance the radiation damage tolerance of the 3-phase material. Single phase samples of Al_2O_3 and $MgAl_2O_4$ started chipping and cracking as the fluence increased whereas 3-phase sample remained stable. XRD shows that single phase samples have a higher degree of lattice expansion for Al_2O_3 and spinel than the 3-phase samples suggesting a more efficient defect annihilation in the 3-phase sample. In this study the correlation between the microstructure evolution and the mechanical behaviors of the irradiated samples will also be reported.

2:20 PM

(ICACC-S13-021-2018) High dose neutron irradiation response of nuclear grade SiC/SiC composites

Y. Katoh*[1]; T. Koyanagi[1]; T. Nozawa[2]; L. Snead[3]

1. Oak Ridge National Laboratory, USA
2. QST, Japan
3. Stony Brook University, USA

SiC composites are promising materials for nuclear energy systems. However, irradiation tolerance to high neutron doses in a wide temperature range needs to be understood to address the design limits for current generation materials. In particular, the Generation III SiC fibers and the pyrocarbon interphase are known to undergo significant degradation in extreme radiatoin environments. In the present work, neutron irradiation was conducted in the High Flux Isotope Reactor. The irradiation dose ranged up to ~100 dpa in the irradiation temperature range ~300 to ~800°C. Hi-Nicalon Type-S or Tyranno-SA3 fiber reinforced chemical-vapor-infiltration (CVI) SiC matrix composites with pyrocarbon interphase were tested. The post-irradiation evaluation by four point flexural tests revealed mechanical property changes for both fiber composites with more severe degradation at lower irradiation temperatures. Detailes microstructural examinations by scanning and transmission electron microscopy led to findings including the partial debonding at fiber/matrix interfaces and modificaiton of secondary carbon phase distributions in the fibers. Details of the experimental results and the underlying mechanisms are discussed. This research was sponsored by the US DOE under contracts DE-AC05-00OR22725 with UT-Battelle, LLC. The use of HFIR was sponsored by the Office of Basic Energy Sciences, U.S. Department of Energy.

2:40 PM

(ICACC-S13-022-2018) Swelling and Creep of SiC Irradiated to 0.1 dpa at 300°C

K. Terrani*[1]; T. Koyanagi[1]; T. Karlsen[2]; Y. Katoh[1]

1. Oak Ridge National Lab, USA
2. Institute for Energy Technology, Norway

High purity chemical vapor deposition (CVD) SiC tensile bars were irradiated in the Halden reactor in Norway. A host of in-pile instrumentation were utilized that enable strict control of temperature and continuous in situ measurement of specimen length change. Two specimens were irradiated with and without applied stress that allowed for measurement of swelling and irradiation creep, respectively. The specimens were extracted from the reactor after a dose of ~0.1 dpa and underwent detailed post-irradiation examination.

3:20 PM

(ICACC-S13-023-2018) Post-irradiation examination of SiC tubes neutron irradiated under a radial high heat flux (Invited)

T. Koyanagi*[1]; Y. Katoh[1]; G. Singh[1]; C. Petrie[1]; C. Deck[2]; K. Terrani[1]

1. Oak Ridge National Laboratory, USA
2. General Atomics, USA

Silicon carbide (SiC) composite is considered among leading candidate materials for accident tolerant fuel cladding in light water reactors (LWRs). An important task in the development is the establishment of a thermomechanical analysis capability to predict the in-pile stress state of the cladding, thus, to assess the probability of cladding failure. This study provides critical experimental data needed for validation of the thermomechanical model. SiC monolith and composite tubes were neutron-irradiated under a radial high heat flux (~0.7MW/m^2) in the HFIR to ~2dpa to produce a

simulated in-pile stress state under normal operation of LWR; the through-thickness temperature gradient under irradiation results in gradient of swelling, which causes a significant stress buildup. Irradiation temperature distribution within the tube was determined by analysis of Raman spectra and dimensional changes upon annealing. Residual stress due to the swelling gradient was investigated by a slit ring method. In addition, potential irradiation-induced cracking was examined by x-ray tomography. These experimental results will be presented together with the stress state calculated by the thermomechanical model to discuss the model validation. This work was sponsored by the U.S. DOE, Office of Nuclear Energy, for the Advanced Fuel Campaign under contact DE-AC05-00OR22725 with ORNL managed by UT-Battelle, LLC.

3:50 PM

(ICACC-S13-024-2018) Swelling and Wigner Energy Release in Neutron Irradiated Silicon Carbide (Invited)

L. Snead*[2]; Y. Katoh[1]; K. Terrani[1]; T. Koyanagi[1]

1. ORNL, USA
2. MIT, USA

Swelling and energy release in graphite due to neutron-induced simple defect production led the way into the current field of irradiation materials science. Since this original work into what later became known as Wigner Energy, or the spontaneous energy release excess of a material's specific heat, essentially no other ceramic material has been shown to exhibit the same behavior as graphite. In this work the swelling and approach to amorphization of single crystal cubic and hexagonal SiC is presented and compared with its related Wigner Energy to 700°C. Samples studied were irradiated in the High Flux Isotope Reactor at the Oak Ridge National Laboratory near water coolant temperature of ~60-80°C. As will be shown, this Wigner energy release for the crystalline material scales directly with swelling approaching 1400 J/gm prior to amorphization. In the as-amorphized condition the energy released over a similar temperature range, independent of the dose for amorphization, is approximately 600 J/gm. These results and related discussion will be supported with analysis of sample microstructure as determined trough TEM and X-ray diffraction analysis.

4:20 PM

(ICACC-S13-025-2018) Radiation effects on SiC/SiC composites for advanced accident tolerant fuel cladding tubes

S. Agarwal*[1]; W. J. Weber[1]

1. University of Tennessee, Material Science and Engineering, USA

Following the beyond-design-basis (BDB) accidents at Fukushima, Japan, the need to develop advanced accident tolerant fuel cladding using innovative materials such as SiC/SiC composites has been recognised. The fabrication of SiC/SiC composites-based cladding poses several challenges, one of them is to understand the radiation induced swelling and cracking. In this work, the effect of ion irradiation in SiC/SiC composites, coupled with state-of-art characterization techniques like transmission electron microscopy experiments (done on the fiber-interface-matrix), Raman spectroscopy (to understand the degradation of carbon layer on the fiber-interface-matrix and other bonding changes) and optical profilometry (to measure step height changes) is presented to understand the overall radiation induced microstructure evolution.

4:40 PM

(ICACC-S13-026-2018) Irradiation Testing of a SiC/SiC Channel Box and SiC Joints in the High Flux Isotope Reactor

C. Petrie*[1]; C. Deck[2]; K. Terrani[3]; Y. Katoh[4]

1. Oak Ridge National Lab, Reactor and Nuclear Systems Division, USA
2. General Atomics, USA
3. Oak Ridge National Lab, Fusion and Materials for Nuclear Systems Division, USA
4. Oak Ridge National Lab, Materials Science and Technology Division, USA

SiC fiber-reinforced SiC matrix (SiC/SiC) composite materials are being considered for nuclear fuel cladding and core structural components due to their high temperature strength, radiation tolerance, and oxidation resistance. For fuel cladding applications, these composites must be sealed with a proper joining technique and remain hermetic over the fuel lifetime. An irradiation capsule is being designed under the Nuclear Science User Facilities Program within the US Department of Energy Office of Nuclear Energy (DOE-NE) to test SiC joint end plugs at light water reactor temperatures (300-350°C) during irradiation in the High Flux Isotope Reactor (HFIR). SiC/SiC composites are also being considered to improve the accident tolerance of other core structural materials such as a channel box in a boiling water reactor (BWR). An unresolved technical feasibility issue for a SiC-based BWR channel box is whether a strong radial neutron flux gradient will induce significant bowing in the channel box due to differential swelling. To investigate this issue, the Advanced Fuels Campaign (DOE-NE) is funding irradiation testing of a SiC/SiC channel box in the core reflector of the HFIR where significant radial fast neutron flux gradients are present. This presentation will discuss the capsule design and analysis efforts, the irradiation test matrix, and plans for post-irradiation examination.

S14: Crystalline Materials for Electrical, Optical and Medical Applications

Optical Material I

Room: Tomoka C

Session Chairs: Victoria Blair, US Army Research Laboratory; Luiz Jacobsohn, Clemson University

1:30 PM

(ICACC-S14-016-2018) Luminescence of activators in glass depending on the preparation process (Invited)

H. Masai*[1]

1. National Institute of Advanced Industrial Science and Technology (AIST), Department of Materials and Chemistry, Japan

Since oxide glass is a metastable solid state material obtained from the supercooled liquid state, the glass structure and physical properties of the glass depend on the preparation process even though the nominal chemical composition are fixed. In the case of phosphor application with doped activators, the local coordination states in glass will be important for the advancement of glass science from both scientific and industrial perspectives. Recently, we reported on the photoluminescence (PL) properties of xSnO-(25-x)SrO-75B_2O_3 glass; a stoichiometric chemical composition of a Sn-doped SrB_6O_{10} crystal. Sn^{2+} belongs to ns^2-type cations ($n \geq 4$), whose emission is strongly affected by the local coordination state. Therefore, we expect that the local coordination state of the ns^2-type emission centre, which can be estimated from the PL and radioluminescence. Here, the relationship between the structural ordering of the Sn-doped SrO-B_2O_3 glass and the luminescent properties of

the Sn^{2+} centre is examined. We have confirmed densification of the glass network was occurred by the heat capacity, ^{11}B MAS NMR, and elastic modulus. The densification of matrix induces an aggregation of the Sn^{2+} centre, and changes the PL and PL excitation spectra. The results suggest that it is important to tailoring the local coordination state of activators during the solidification process.

2:00 PM

(ICACC-S14-017-2018) Sol-gel synthesis and down-conversion photoluminescence properties of Tb^{3+}/Yb^{3+} co-doped ZrO_2-SiO_2 nano-crystallized glasses

M. Isogai*[1]; T. Hayakawa[1]; J. Duclere[2]; P. Thomas[2]

1. Nagoya Institute of Technology, Japan
2. Limoges University, France

Recently, the development of wavelength conversion materials for application to solar cells, light-emitting diodes (LEDs), and displays have been drawn interest widely. Above all, down-conversion (DC) materials which convert visible light to near-infrared light are a promising tool to enhance the efficiency of crystalline silicon solar cells. In this study, the wave conversion properties in Tb^{3+}/Yb^{3+} co-doped ZrO_2-SiO_2 nano-crystallized glasses are investigated. ZrO_2 has many excellent properties such as wide band gap (larger than 5eV) and low phonon energy (less than 650 cm^{-1}). The materials were synthesized by a sol-gel method, followed by heat-treatment at different temperatures (900°C, 1000°C, 1100°C) and characterized using Tb^{3+} luminescence decay curves. As a result, it was found that the energy transfer efficiency (ETE) from Tb^{3+} to Yb^{3+} increased with rising heat-treatment temperatures. In addition, a reduction treatment in $5\%H_2/95\%N_2$ was applied to the sample heated at 1100°C because DC intensity decreased with increasing heat-treatment temperature. Consequently, the reduction treatment at 1100 °C for 1 h was found to be effective for increasing DC intensity.

2:20 PM

(ICACC-S14-018-2018) Balancing microstructure and spectroscopic behavior of nanocomposites for mid-infrared solid-state lasers (Invited)

V. L. Blair*[1]; N. Ku[3]; Z. D. Fleischman[2]

1. US Army Research Laboratory, Weapons and Materials Research Directorate, USA
2. US Army Research Laboratory, Sensors and Electronic Devices Directorate, USA
3. US Army Research Laboratory, ORISE, USA

Several challenges exist for mid-infrared laser materials, including (1) the need to dissipate heat generated in lasing (2) the common issue of luminescence quenching by multi-phonon relaxation, and (3) the trade-off between high thermal conductivity and small maximum phonon energy. These challenges are being addressed by synthesizing a ceramic nanocomposite in which multiple phases (MgO and Er:Y_2O_3) will be incorporated into the same structure. To obtain a transparent part, several processing strategies must be employed, such as colloidal mixing and processing to enable particle homogeneity. A fast sintering cycle and timeline are also crucial for preventing massive grain growth. In this presentation, the goal is to tread the fine line between the ideal microstructure and ideal spectroscopic behavior in fabricating enhanced transparent materials. In our case, the transmission often trades off with the fluorescence intensity.

3:10 PM

(ICACC-S14-019-2018) Silicon content in transparent YAG ceramics, analysis of the production process (Invited)

J. Hostasa*[1]; R. M. Gaume[2]; A. Piancastelli[1]; S. J. Pandey[3]; M. Martinez[4]; M. Baudelet[4]; T. Epicier[5]; V. Biasini[1]; L. Esposito[1]

1. National Research Council of Italy, ISTEC CNR, Institute of Science and Technology for Ceramics, Italy
2. CREOL – The College of Optics and Photonics, University of Central Florida, USA
3. University of Central Florida, Physics Department, USA
4. University of Central Florida, National Center for Forensic Science, USA
5. INSA of Lyon, F-69621 Villeurbanne Cedex, MATEIS, France

Transparent ceramics are usually prepared with the use of a sintering aid, in the case of YAG mostly silicon in the form of SiO_2 or TEOS. However, the presence of Si may lead to the formation of secondary phases or to the deterioration of optical and laser properties due to the presence of color centers. It is therefore important to know and control its content in the sintered material. In this work we analyze the quantity and distribution of silicon in transparent YAG ceramics produced by reaction sintering of a mixture of oxide powders under high vacuum. Silicon is added in the mixture as TEOS or nanometric SiO_2 powder. Laser-induced breakdown spectroscopy (LIBS) was used to quantify and compare the silicon content after the various steps of the production process (i.e. in the as-shaped samples, after sintering and after air annealing). A strong decrease of Si concentration was observed after the vacuum sintering. TEM/EDX showed an increase of silicon content at grain boundaries after air annealing steps at elevated temperatures.

3:40 PM

(ICACC-S14-020-2018) Effects of Polishing Tool Characteristics on the Material Removal Mechanisms of Polycrystalline YAG Ceramics

D. M. Ross*[1]; H. Yamaguchi[1]; J. Long[1]; M. Parker[1]

1. University of Florida, Mechanical and Aerospace Engineering, USA

Transparent polycrystalline yttrium aluminum garnet (YAG) ceramic host materials have garnered an increased level of interest for high-power laser applications due to their ability to be manufactured in large sizes and doped in substantial concentrations. However, surface characteristics have a direct effect on the lasing ability of these materials, and a lack of a fundamental understanding of the polishing mechanisms of polycrystalline ceramics remains a challenge to their utilization. The focus of this paper is to more deeply develop this understanding through investigating the effects of polishing tool characteristics and abrasive behavior on the grain structure of YAG ceramics during fine polishing. Using Magnetic Field-Assisted Finishing (MAF), sub-nanometer scale polishing of 0.25 % Nd-doped polycrystalline YAG ceramics was performed using a variety of polishing tool types. It was demonstrated that the form in which abrasives are applied to the surface during the finishing process plays a significant role in the material removal mechanisms associated with the localized grain structure. This can relate to grain dislodgment, uneven material removal between individual grains, and increased material removal at grain boundaries.

4:00 PM

(ICACC-S14-021-2018) Production of sinterable YAG Powder for the applications as laser host materials

J. Sharma*[1]; H. Singh[1]

1. PEC University of Technology, India

Till recently the most widely used commercial technique, Czochralski method and Bridgman techniques at nealy 2300°C, produces single crystals from the melt. These processes last for weeks, leading to inculcation of impurities and stresses in the crystal. Synthesis of Polycrystalline Transparent Ceramics (PTCs) is new spearheading technology producing powders through nano

synthesis routes are capable to counter the challenges of purity, size, chemical stability, optical coherency and transparency. PTCs produced are capable to be designed for their chemical coherency, phase uniformity and morphological requirements for sintering. Present work deals with the production of Yttrium Aluminium Garnate (YAG) powder through Co-precipitation and Urea precipitation method. Ammonium hydrogen carbonate (AHC) and urea are used as precipitants in both processes respectively and calcined in range of 1200-1400ºC. The reaction mechanism has been investigated using different techniques such as XRD, FESEM, FTIR and DLS. Crystallite size are below 45nm with near spherical shape. Agglomeration is lesser in Urea precipitation method. FTIR and XRD confirms the formation of YAG powder and purity of phases produced. Urea method produces partilcle size of 105nm. Resulted yield is in range of 81-83%. The powders produced are sinterable to produce the transparent ceramic for various civil and defense applications

4:20 PM

(ICACC-S14-022-2018) Causes for the lower performance of polycrystalline ceramic scintillators: The case study of LuAG:Ce (Invited)

L. G. Jacobsohn*[1]; A. A. Trofimov[1]

1. Clemson University, Materials Science and Engineering, USA

The scintillation performance of polycrystalline ceramic scintillators is commonly inferior to single crystals. A comparative investigation between $Lu_3Al_5O_{12}$:Ce (LuAG:Ce) transparent polycrystalline ceramic and single crystal scintillators was executed in terms of their microstructure, optical, luminescent and scintillating properties to gain insight into the causes for the lower scintillation performance of polycrystalline ceramics. The polycrystalline ceramic was fabricated by high temperature vacuum sintering and the single crystal was grown by the Czochralski method with ~0.06 and ~0.04 at.% Ce, respectively. The polycrystalline ceramic presented lower optical transmission, higher self-optical absorption, and a higher degree of structural disorder. Radioluminescence (RL) and photoluminescence measurements indicated the presence of defect bands, including F^+-type centers. Thermoluminescence measurements as a function of the irradiation time combined with glow curve deconvolution revealed the presence of 6 glow peaks in the single crystal and 4 glow peaks in the polycrystalline ceramic, all with first-order kinetics. Equations of detailed balance elucidaded the effect of trap characteristics on the probability of capture of electrons from the conduction band. This material is based upon work supported by the National Science Foundation under Grant No. 1653016.

S15: Additive Manufacturing and 3-D Printing Technologies

Direct Writing Technologies I

Room: Coquina Salon B

Session Chair: Paolo Colombo, University of Padova

1:30 PM

(ICACC-S15-017-2018) Innovative Electric Motor Designs Enabled by Additive Manufacturing (Invited)

M. C. Halbig*[1]

1. NASA Glenn Research Center, USA

New manufacturing methods are needed which enable innovative electric motor designs that have much higher power densities and/or efficiencies compared to the current state-of-the-art. Compared to conventional fabrication methods for electric motors, additive manufacturing offers the potential to radically change motor designs so that they have compact designs, complex geometries, multi-material components, innovative cooling, and optimally designed and manufactured components. Optimized stator design concepts are being pursed in which advanced conductive 3-phase coils are fabricated using direct printing of optimized silver pastes and dielectrics along with the integration of magnets from binder jetting. An overview of other additive manufacturing activities will be provided for the fabrication of additional motor components such as a rotor backing plate, a housing, and a copper wire embedded stator. The performance benefits from optimized materials and new component design will be emphasized.

2:00 PM

(ICACC-S15-018-2018) 2D colloids of graphene oxide for materials manufacturing

E. Garcia-Tunon*[1]; E. Feilden[2]; E. D'Elia[2]; E. Saiz[2]

1. University of Liverpool, Materials Innovation Factory & School of Engineering, United Kingdom
2. Imperial College, Materials, United Kingdom

Wet processing is behind most of the traditional and advanced shaping techniques. Among the latter, Additive Manufacturing has attracted much attention from industry, academia, media and society in the last decade, promising new designs for bespoke applications at the click of a button. However one of the main challenges to make this a reality is the limited range of materials available for Additive Manufacturing. On the other hand a large amount of resources have been invested in graphene research. A field rapidly evolving, spreading across multidisciplinary areas and leading to the development of new basic science and practical applications. Connecting 2D materials and Additive Manufacturing research leads to promising applications in materials manufacturing. Graphene oxide (GO) colloids have the potential of making other materials behave like clay; enabling shaping to create complex objects. GO plays multiple roles: as dispersant, rheology modifier and binder; likely becoming the most versatile existing processing additive. This talk will illustrate the unique behaviour of GO colloids of graphene oxide in water and their role in ceramic processing. They have the ability to tune the viscoelastic response of suspensions containing particles with different chemistries, shapes and sizes; enabling the manufacturing of complex 3D structures made of oxide and non-oxide ceramic particles and platelets.

2:20 PM

(ICACC-S15-019-2018) Direct Writing and Characterization of Silver Pastes with Advanced Carbon Additions for Electric Motor Applications

A. Salem*[3]; J. Zhou[4]; V. L. Wiesner[1]; M. C. Halbig[1]; M. Singh[2]

1. NASA Glenn Research Center, USA
2. Ohio Aerospace Institute, USA
3. Washington University in St. Louis, USA
4. Case Western Reserve University, USA

In recent years, commercially available conductive silver pastes have significantly improved and ultimately increased the number of potential applications for printed electronics. Silver has proven to be particularly promising due to its relatively low cost and high conductivity. Commercially available silver pastes were printed and characterized for their electrical conductivity and thermal stability. Resistivity measurements were conducted using the 4-probe (Kelvin) method. A wide variety of carbon black powders (CB) and carbon nanostructures (CNS) were added in silver pastes to improve the electrical conductivity while maintaining the printability. In theory, the CNS and CB should improve conductivity by creating an electrically conducting network at low weight percentages. To maintain the printability of modified pastes, Dimethylformamide (DMF) and Brij 30 were added to ensure the dispersion of carbon additions. Effects of carbon fillers on electrical and thermal properties are discussed along with the effect of curing conditions and microstructure.

2:40 PM

(ICACC-S15-020-2018) Graphene Modified Silver Pastes for Additive Manufacturing of Electric Motors

J. Zhou*[2]; A. Salem[3]; M. C. Halbig[1]; M. Singh[4]

1. NASA Glenn Research Center, USA
2. Case Western Reserve University, USA
3. Washington University in St. Louis, USA
4. Ohio Aerospace Institute, USA

Additive manufacturing technologies are expected to improve wire packing and compactness enabling new electric motor designs with higher power densities and/or efficiency. This study focuses on the effect of graphene on the resistivity of silver paste. High conductive carbon-based additives such as graphene can potentially yield resistivity values equal to or better than that of copper wire. Two types of graphene, single-layered and multi-layered sheets of graphene, with varying weight percentages were added to a commercial silver paste. Electrical conductivity of traces made with graphene modified pastes were measured with four point probe method and show the influence of the amount and type of graphene. SEM analysis of the traces was carried out to see the distribution and packing of secondary phases in silver matrix. By attaining lower resistivity values for traces printed with these pastes and utilizing additive manufacturing methods, new motor designs can be achieved.

Direct Writing Technologies II

Room: Coquina Salon B

Session Chair: Elizabeth Kupp, The Pennsylvania State University

3:20 PM

(ICACC-S15-021-2018) Direct ink-writing of ceramic matrix composite structures

G. Franchin*[1]; P. Colombo[1]; L. Wahl[1]; H. S. Maden[1]

1. University of Padova, Industrial Engineering, Italy

Direct ink-writing (DIW) process was employed as a novel approach for the production of ceramic matrix composites (CMCs) using a system based on a preceramic polymer and short fibers. The optimization of the ink comprising of a polysiloxane and chopped carbon fibers involved the use of different additives (SiC powder, colloidal silica) and the minimization of the amount of (non-aqueous) solvent content. Rheological characterization was performed in order to characterize and validate the inks suitable for being extruded through fine (~400 μm diameter) nozzles and the fabrication of suspended struts. The formation of a gel structure, essential for the production of highly porous lattices with unsupported features, was confirmed. DIW was followed by pyrolysis in inert atmosphere (nitrogen), during which the polysiloxane converted into an amorphous SiOC phase. This could result in extensive cracking of the matrix, due to the decomposition of the polymer and to hindered shrinkage. The role of the carbon fibers and the addition of fillers (SiC powder) in enhancing or preventing such phenomenon will be discussed. The results of mechanical tests on samples fabricated with different inks and geometries will also be presented. The incorporation of fibers to a preceramic polymer opens the possibility of generating complex ceramic shapes with enhanced mechanical properties.

3:40 PM

(ICACC-S15-022-2018) 3D-Printing of hierarchical porous ceramic materials for catalysis

J. Lefevere*[1]; L. Protasova[1]; S. Mullens[1]; V. Meynen[2]

1. VITO, Sustainable Materials Management, Belgium
2. University of Antwerp, Belgium

A more sustainable chemical industry and the efficient use of depleting resources are a hot topic in both research and society. In order to improve the commonly used packed beds of catalyst pellets, a structured catalyst can be used. Macroporous structured support materials coated with a layer of catalyst or directly printed catalyst can result in better mass- and heat transfer properties in combination with a lower pressure drop. It is clear that the architecture of the support has a major influence on these properties. Therefore 3D-printing technology can be a good tool in rapid prototyping of new architectures as it allows an enormous degree of freedom. In this work the use of 3D-printing for the manufacturing of new catalytic materials was investigated. The goal of this work was to synthesise 3D printed catalyst materials and map the possible advantages and drawbacks of the use for catalysis or sorption. Either by using an optimized coating method of an inert support or direct printing of the zeolite an active catalyst in the desired shape could be manufactured. The impact of binders and architecture on the physico-chemical and catalytic properties of these materials was investigated and discussed. The most important advantage of the use of this technique is that the architecture as well as the composition of the structure can be optimized in function of the application.

4:00 PM

(ICACC-S15-023-2018) Complex Shaped Boron Carbides

R. Lu*[1]; S. Chandrasekaran[1]; W. L. Du Frane[1]; M. A. Worsley[1]; J. D. Kuntz[1]

1. Lawrence Livermore National Laboratory, Materials Science Division, USA

Complex shaped boron carbide (B_4C/C) geometries at near-full densities are achieved using various additive manufacturing techniques. Negative additive manufacturing is explored which involves 3D printing of sacrificial molds that are used for casting negative copies. This technique incorporates gelcasting using resorcinol-formaldehyde polymer and is favorable at the industrial scale due to the ease and speed of production for large complex shaped parts. By optimizing B_4C particle size and sintering conditions, 97-98% theoretical max density is achieved. Direct Ink Write is another additive manufacturing technique that has also been explored to create complex shaped boron carbides. Direct Ink Write involves the extrusion of a thixotropic ceramic slurry from a nozzle in 3-dimensional space. Print resolution with struts as low as 250um is achievable when using slurries with a mean B_4C particle size of 0.54um. Rheology of the boron carbide slurry has been optimized to print lattices and other structures. This work was performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344.

4:20 PM

(ICACC-S15-024-2018) Direct Ink Writing of Boehmite: From Microstructural Control to Mechanical Properties

A. M'Barki*[1]; A. Stevenson[1]; L. Bocquet[2]

1. Saint-Gobain CREE, LSFC, France
2. Ecole Normale Supérieure, France

Direct Ink Writing is an additive manufacturing technique based on continuous layer-by-layer filament deposition. Mostly used to print porous structures, DIW of dense and strong ceramic objects remains an open challenge. However, the advantage of DIW resides in its ability to print multimaterial objects, offering the possibility to combine complex shaping to precise microstructural and functional control, from bioinspired materials, to novel composite structures. Our work focuses on using boehmite gels for DIW, an Al_2O_3 precursor, as a ceramic matrix to obtain different microstructures. Very small changes to the gels composition lead to completely different microstructures and hence, functional properties. By combining the microstructural versatility of boehmite gels with an understanding of rheology, we are able to print micro and macrocomposites with enhanced mechanical properties. We take advantage of the flow behaviour inside DIW nozzles to align alumina platelets during printing. This provides the printed object with increased fracture toughness in the desired direction, with the

ability to deviate the fracture propagation perpendicularly to the printing direction. A single object can thus be precisely designed, alternating between dense, strong layers, and directionally tough, fracture deviating layers, to combine the complexity of the shape with the tailoring of mechanical behaviour

Direct Writing Technologies III

Room: Coquina Salon B
Session Chair: Michael Halbig, NASA Glenn Research Center

4:40 PM

(ICACC-S15-025-2018) Direct-Writing of Flexible Barium Titanate/PDMS 3D Photonic Crystals with Mechanically-Tunable Terahertz Properties

P. Zhu[1]; W. Yang[1]; R. Wang[1]; S. Gao[2]; B. Li[2]; Q. Li*[1]

1. Institute of Metal Research, Chinese Academy of Sciences, Shenyang National Laboratory for Materials Science, China
2. Graduate School at Shenzhen, Tsinghua University, Division of Energy and Environment, China

Mechanically flexible 3D terahertz photonic crystals (3D-TPCs) are created by the direct-writing technology with a composite ink system composed of polydimethylsiloxane (PDMS) and barium titanate ($BaTiO_3$) nanoparticles. The direct-writing technology allows an easy creation of complex 3D structures with designed geometry, while the refractive indices of the composite ink can be modulated by varying the content of $BaTiO_3$ nanoparticles. Thus, 3D-TPCs with different terahertz properties are obtained by the direct-writing technology. More interestingly, these 3D-TPCs demonstrate a unique tunable terahertz property under external force field due to their mechanical flexibility from the PDMS matrix of the composite ink. Thus, their terahertz property is responsive to external force fields reversibly, which can find novel applications in terahertz technology and other relative technological applications.

5:00 PM

(ICACC-S15-026-2018) Direct ink writing of cementitious materials

J. P. Youngblood*[1]; M. Moini[2]; J. Olek[2]; P. Zavattieri[2]

1. Purdue University, School of Materials Engineering, USA
2. Purdue University, School of Civil Engineering, USA

Direct Ink Writing (DIW) or Robocasting is an Additive Layer Manufacturing (ALM) technique that is "3D" printing method where liquid "inks" are extruded from a nozzle and solidified to prepare precisely architectured solid shapes. This method has been used for ceramic structures, typically by using colloidal inks to prepare near net shape ceramic parts. Here, we will detail efforts at DIW of cementitious materials, as well as elucidating design criteria for the "inks". Materials, uses and issues related to their manufacture will be presented, as well as discussion of the design flexibility that DIW allows with cement.

5:20 PM

(ICACC-S15-035-2018) Direct write and 3d printing of pre-ceramics polymers: Materials, approaches and applications

J. M. Lavin*[1]; D. M. Keicher[1]; L. R. Evans[1]; L. Tsui[1]; S. S. Mani[1]

1. Sandia National Laboratories, USA

The evolution of additive manufacturing in ceramics has lead to an increasingly diverse and flexible means of printed ceramics. Given the relatively recent advances in printers, materials and tooling the opportunities are vast for the exploration of printed ceramics. To that end, we at Sandia National Labs are invested in exploring innovative approaches and materials to gain a unique understanding in the printing of (highly loaded) pre-ceramic polymer based pastes and resins. Here, we will present ongoing work investigating stereolithographic (SLA) and direct write extrusion printing of highly loaded alumina polymers, post processing and the incorporation of printed parts into components.

S17: Advanced Ceramic Materials and Processing for Photonics and Energy

Multifunctional I

Room: Halifax A/B
Session Chairs: Rafik Naccache, Concordia University; Alessandro Martucci, University of Padova

1:30 PM

(ICACC-S17-014-2018) Transparent and Flexlble Tin Oxide Electrolyte-Gated Transistors (Invited)

I. Valitova[1]; A. Subramanian[1]; I. Ruggeri[2]; F. Soavi[2]; C. Santato[3]; F. Cicoira*[1]

1. Polytechnique Montreal, Chemical Engineering, Canada
2. Università di Bologna, Chemistry, Italy
3. Polytechnique Montreal, Engineering Physics, Canada

Metal oxide semiconductors, such as ZnO, SnO, In_2O_3 and indium gallium zinc oxide are of great interest for large area and high-performance electronic devices, because of their transparency, ease of process, chemical stability and high n-type mobility. SnO_2 already found applications in sensing, photovoltaic, optoelectronic devices and batteries. We fabricated, both on rigid and flexible substrates, electrolyte-gated SnO_2 transistors making use of high surface area activated carbon, as a gate electrode, and the ionic liquid (IL) 1-Butyl-3-methyl imidazolium bis(trifluoromethylsulfonyl)imide [EMIM][TFSI], as the gating media. Thin films of SnO_2 have been deposited by sol gel and water based synthesis techniques and were photolithographically patterned as transistor channel materials. We investigated bottom-contact top-gated transistor where we varied the area of the SnO_2 layer in contact with electrolyte and the area of overlap of source/drain electrodes with the gate. Using electrical measurements and cyclic voltammetry we demonstrated that the performance of electrolyte-gated SnO_2 transistors can be tuned via patterning of the metal oxide layer. Patterned SnO_2 transistors work in depletion mode, while unpatterned ones work in enhancement mode. These simple device architectures working at low voltages are promising for low cost, flexible, transparent large area electronics.

2:00 PM

(ICACC-S17-011-2018) Photoluminescent Properties and Hyperspectral Imaging of Eu^{3+} Complexes (Invited)

D. Errulat[1]; M. Murugesu[1]; E. Hemmer*[1]

1. University of Ottawa, Chemistry and Biomolecular Sciences, Canada

Seeking more efficient light management in solar devices, photon conversion, such as down-shifting, is an attractive approach. In down-shifting, an ultraviolet (UV) photon that is otherwise lost for solar energy conversion is converted into a visible photon that then becomes available for the solar cell absorber. Trivalent europium (Eu^{3+}) complexes are well-known for their bright luminescence and photostability. Under UV excitation (typically 280 to 450 nm), the most dominant emission is centered at 612 nm due to the $^5D_0 \rightarrow {}^7F_2$ electronic f-f transition of Eu^{3+}, which makes them potential candidates for solar applications. Aside from Eu^{3+}, trivalent terbium (Tb^{3+}) can be used in down-shifting complexes. Thus, homodinuclear Eu^{3+} and Tb^{3+} as well as heterodinuclear Eu^{3+}-Tb^{3+} complexes were synthesized and their photoluminescent properties were analyzed. The $^5D_0 \rightarrow {}^7F_2$ Eu^{3+} transition is of special interest from a fundamental point of view since being hypersensitive to the chemical environment of the Eu^{3+} centers. It was found that the Eu^{3+}-Tb^{3+} complex crystallizes in three different crystal morphologies (polymorphs) that were characterized by hyperspectral imaging. Each of them shows distinct

characteristic spectral features, namely splitting of the $^5D_0 \rightarrow {}^7F_2$ transition into J sub-levels, providing evidence for a different chemical environment of the Eu^{3+} centers in the three crystal types.

2:30 PM

(ICACC-S17-016-2018) Ceramic Materials for Energy Applications (Invited)

D. Xue*[1]

1. Changchun Institute of Applied Chemistry, Chinese Academy of Sciences, China

Recent studies on mesoscale ceramics were introduced in this talk, from both theoretical and experimental aspects. Toward energy applications some mesoscale ceramics like single crystals, microcrystals, nanomaterials, and colloids were fabricated via various processing strategies, such as single crystal Czochraski growth processes, hydrothermal and solvothermal processes, combustion process and colloidal forming processes. With the help of the chemical bonding theory of single crystal growth, bulk single crystals can be more efficiently grown from melts. Performance dependent ceramics processing toward electrochemical energy storage was also reported.

3:20 PM

(ICACC-S17-017-2018) Carbon-based composite materials with applications in Supercapacitors and fuel cells (Invited)

D. Chua*[1]

1. National University of Singapore, Materials Science & Engineering, Singapore

Carbon materials have attracted much attention due to their unique properties, ranging from low dimensional effects, good structural integrity, high electrical and thermal conductivity, and chemical stability. Increasingly, carbon-based materials have progressed from thin films to the nanoscale dimensioned carbon nanotubes and graphene. In this aspect, one area of interest lies in whether carbon is useful as a catalyst support with direct applications in clean energy, specifically supercapacitors and fuel cells. In this talk, we will show that first that we can engineer various 1D and 2D carbon-based materials. We will further show and compare the fuel cell properties when other 2D materials are integrated with the carbon-based materials as catalyst support. A series of in-situ tests are also performed which includes accelerated degradation test and electrochemical impedance spectroscopy to validate the effectiveness and robustness of these materials. We will mention briefly other applications for these carbon based materials.

3:50 PM

(ICACC-S17-018-2018) Metal-organic Frameworks at the Biointerface (Invited)

C. J. Doonan*[1]

1. The Univeristy of Adelaide, Chemistry, Australia

Many living organisms are capable of producing inorganic materials of precisely controlled structure and morphology. This ubiquitous process is termed biomineralization and is observed in nature from the macroscale (e.g., formation of exoskeletons) down to the nanoscale (e.g., mineral storage and transportation in proteins). Extensive research efforts have pursued replicating this chemistry with the overarching aims of synthesizing new materials of unprecedented physical properties and understanding the complex mechanisms that occur at the biological–inorganic interface. Metal-organic Framework (MOFs) materials are a new class of materials that are well known for their ultra-high surface areas and gas storage and separation properties. However, recent studies have focused on the synthesis of MOF biocomposites for application to applied research fields including protection and delivery of biopharmaceuticals, biosensing, biocatalysis, biobanking, and cell and virus manipulation. This talk will canvass how MOF biocomposites can be synthesized via a process termed biomimetic mineralisation.

4:20 PM

(ICACC-S17-019-2018) Charge transfers and ionic diffusion at amorphous-crystal interface and related electrochemistry of TiO_2

H. Choi*[1]

1. Virtual Lab Inc., Republic of Korea

Amorphous phases of oxides are frequently observed as intermediates of crystalizations or are intentionally prepared with thermal processing for engineering purposes. The existence of amorphous phases in nanocrystal oxides is known to strongly affect the electrochemical properties of oxides. Recently, partial amorphization of TiO_2 has been reported to enrmously improves various functionality, such as photocatalysis and battery performances. However, the physical origins of improved photocatalytic activity and battery performances have not been clearly found so far. This study systematically investigated the diffusivity of ionic conductivity and electronic structures of amorphous-crystal interfaces of TiO_2 using density functional theory (DFT) calculations and experiments.

4:40 PM

(ICACC-S17-020-2018) Electrochemical Study of MgAl and MgAlTi Hydrotalcite

E. d. Magdaluyo*[1]; R. Bonifacio[1]; G. Magayanes[1]

1. University of the Philippines, Dept of Mining, Metallurgical and Materials Engineering, Philippines

The electrochemical behavior of MgAl and MgAlTi layered double hydroxides was investigated in electrolytic media of 0.1 M NaOH and 0.1 M NaOH/0.05 M NaCl. Voltammetric and amperometric studies were performed to identify the oxidation and reduction processes and the effect of Mg^{2+}, Al^{3+} and Ti^{4+} cations in the total reactivity of the hydrotalcite material. Electrocatalytic behavior of water oxidation by the two hydrotalcites shows better performance for the MgAlTi compared to MgAl. This is apparently due to the presence of Ti^{4+} as promoter ion. Chloride adsorption at the surface of the electrode affects the current obtained by using sodium-chloride containing electrolyte, with larger currents resulting from the use of MgAl hydrotalcite. The reduction processes are also observed in the cations within the networks of both hydrotalcites.

Honorary Sympoisum: Advancing Frontiers of Ceramics for Sustainable Society Development - International Symposium in Honor of Dr. Mrityunjay Singh

Advancing Frontiers of Ceramics III -Composite Materials 1

Room: Coquina Salon E
Session Chairs: Csaba Balazsi, HAS Centre for Energy Research; Zhengyi Fu, Wuhan University of Technology

1:30 PM

(ICACC-HON-019-2018) Advances in Ceramic Composites for Fission and Fusion Energy Applications (Invited)

S. J. Zinkle*[1]; Y. Katoh[2]

1. University of Tennessee, USA
2. Oak Ridge National Lab, USA

Ceramic composites are candidate high performance materials for multiple challenging applications in fission and fusion energy systems, due to their favorable mechanical, thermal and neutron interaction properties. The ability to tune some key mechanical and physical properties in ceramic composites is key for their successful

performance in these extreme operating environments. Potential applications of advanced ceramic composites in existing light water reactors include SiC/SiC fuel cladding that may provide enhanced accident tolerance, as well as novel composite fuels that could provide higher burnup capability, lower fuel operating temperatures, and/or improved accident tolerance. Ceramic composites are also being considered for several structural applications in proposed Generation IV fission reactor concepts. Potential fusion energy applications include SiC/SiC composites for the structural material in the blanket region surrounding the plasma, ultrahigh temperature ceramic composites for plasma facing components, high temperature SiC/SiC flow channel inserts to enable high thermodynamic performance in PbLi dual coolant concepts, and potential novel architectures in Li-bearing ceramic composites in the fusion blanket region to generate tritium fuel for the fusion reaction. The design advantages and R&D challenges associated with use of ceramic composites in these applications will be summarized.

2:00 PM

(ICACC-HON-020-2018) Designing Ceramic Composites for use at High Temperatures: Current Trends and Future Prospects (Invited)

R. N. Singh*[1]

1. Oklahoma State University, School of Materials Science and Engineering, USA

Applications of ceramic composites require an understanding of the interrelationship among processing, fiber-matrix interface and mechanical properties both at room and elevated temperatures. In addition, these applications require an understanding of the crack propagation and fracture behaviors in CMCs for assessing their performance, life prediction and for designing superior CMCs with even higher temperature capability. A novel in situ technique based on video imaging is used to directly observe and measure crack growth and fracture behaviors in CMCs between 25°-1400°C under monotonic and fatigue loads. The results of the crack growth thus obtained are used to analyze fracture resistance behavior, and theoretical analyses are done to develop models to predict the crack growth and fracture resistance behaviors. Bridging stress functions are also obtained from the analytical models over a range of temperatures, which are then used in designing CMC with superior elevated temperature mechanical properties. The models developed are used to identify the roles of the fiber, matrix, and interface properties in obtaining superior properties at high temperatures. These results on SiC_f-reinforced CMCs will be presented and discussed along with the prospects of using CMCs in current and future systems.

2:30 PM

(ICACC-HON-021-2018) "Tough Behavior" of Short Carbon Fiber-dispersed SiC Matrix Composites Fabricated by Melt Infiltration Process (Invited)

Y. Kagawa*[1]; Y. Atsumi[1]; Y. Arai[1]; H. Hatta[1]

1. Tokyo University of Technology, The Center for Ceramic Matrix Composites, Japan

Short carbon fiber-dispersed SiC matrix composites have been developed for advanced car brake rotors. Fracture toughness of the composites are usually very low ~4-5 $MPam^{1/2}$. However, the composites behave like a tough ceramics. This presentation examines the cracking behavior of short carbon fiber-dispersed SiC matrix composite fabricated by melt infiltration process. Two different cracks, initial thermal cracks and long cracks initiated under mechanical loading play important role on the tough behavior. The initial cracks helps release of residual stress and they are no influence on the long crack growth. Long crack growth is controlled by the interaction process between crack in SiC/Si phases and short carbon fiber-C/SiC minicomposite phase. Some of the implications associated with the micro fracture model are discussed.

3:20 PM

(ICACC-HON-072-2018) Advanced Environmental Barrier Coating and SA Tyrannohex SiC Composites Integration for Improved Thermomechanical and Environmental Durability (Invited)

D. Zhu*[1]; M. C. Halbig[1]; M. Singh[1]

1. NASA Glenn Research, Materials and Structures Division, USA

The 2700°F environmental barrier coating (EBC) systems, particularly, the Rare Earth – Hf – Si coatings, have significant improved the temperature capability and environmental stability of SiC/SiC Ceramic Matrix Composite (CMC) Systems. We have specifically developed the EBC systems, integrating the EBC to the high temperature SA Tyrannohex SiC fiber composites, for comprehensive performance and durability evaluations for turbine engine airfoil component applications. The fundamental mechanical properties, environmental stability and cyclic durability of the composites were investigated. The paper will emphasize the high pressure combustion rig recession, thermal stress resistance and thermomechanical Low Cycle Fatigue of the environmental barrier coated composites in simulated engine combustion water vapor, thermal gradients, and mechanical loading conditions. We have also investigated high-heat-flux and fatigue degradation mechanisms, determined the upper limits of operating temperatures for the coated composite systems. Recent progress has also been made by using the self-healing rare earth-silicon based EBCs, thus enhancing the SA composite hexagonal fiber columns bonding for improved thermomechanical and environmental durability. More advanced EBC- composite systems based on the new EBC-Fiber Interphases will also be discussed.

3:40 PM

(ICACC-HON-022-2018) Properties of SiC/SiC composites with BN interphase (Invited)

S. Dong*[1]; J. Hu[1]; X. Zhang[1]

1. Shanghai Institute of Ceramics, Chinese Academy of Sciences, China

Continuous SiC fiber reinforced SiC ceramic matrix composites (SiC/SiC) have attracted great attention due to their excellent properties and huge potential application in hot structures of aeroengine. As an essential constituent element of SiC/SiC composites, the interface plays an important role in the material's mechanical properties and oxidation resistance. In order to improve the anti-oxidation property, BN interphase was prepared on the SiC fiber surface by chemical vapor deposition method. The effects of processing parameters on the growth rate, microstructure and crystallinity of BN were investigated. The growth mechanism was also discussed. SiC/SiC composites were fabricated by chemical vapor infiltration and reactive melt infiltration combined method. The mechanical properties, oxidation resistance and thermal stability of SiC/SiC composites were tested, and the relationship between BN prepared under different condition and the properties of SiC/SiC composites was studied. The materials show good oxidation resistance and thermal stability. The mechanical properties almost did not decrease after oxidized for 50h at 700 DC. The composites still had 80% strength retention after heat treatment for 300h at 1200 DC.

4:00 PM

(ICACC-HON-023-2018) Performance of New Catalyst Carriers Made from Conducting Ceramics for PEM Fuel Cell (Invited)

Z. Fu*[1]

1. Wuhan University of Technology, State Key Lab of Advanced Technology for Materials Synthesis and Processing, China

For the proton exchange membrane fuel cell (PEMFC), catalyst with low cost and excellent catalytic properties is important in application. Traditionally, the Pt catalyst particles are homogeneously dispersed on carbon, which might be corroded in acid liquids in PEMFC. People were thinking about to replace carbon with conducting ceramics. The performance of conducting ceramic

particles in acid liquid needs to be studied. In this paper, the variations of conducting ceramic particles (TiB_2, ZrB_2) in HF and H_2O_2 were studied. Conducting ceramic particles will be oxidized in different extent in the environment of HF and H_2O_2. However, the oxidization can be minimized through appropriate process. Conducting ceramic particles as catalyst carriers with two different structures were analyzed. The catalysts prepared with Pt on Nafion stabilized TiB_2 particles displays more stable than that of a commercial Pt/C catalyst, which is likely attributed to the stability of the TiB_2 support and the stabilization effect from Nafion.

4:20 PM

(ICACC-HON-024-2018) Silicon-based ceramic nanocomposites for environmental applications (Invited)

Z. Yu*[1]

1. Xiamen University, College of Materials, China

Silicon-based ceramics and nanocomposites modified with transition metals are effectively synthesized via the single-source-precursor approach. The resultant ceramics possess advanced properties including adsorption capacity and catalytic activity towards organic dyes and electromagnetic (EM) properties and have to be considered as potential candidate materials for environmental applications. Depending on the transition metal, in-situ formed carbon nanophases as well as (multinary) ceramic phases are generated. The introduction of metal such as Fe and Ni endows the final ceramic nanocomposites magnetic properties and nanoporous characteristics with excellent adsorption capacity, catalytic activity and convenient recycling ability for removal of organic dyes. The introduction of Hf and/or Ta results in the in-situ formation of HfyTa1-yCxN1-x-carbon core-shell microstructure together with graphene-like carbon dispersed within the Si-based matrix. Due to the enhanced dielectric properties, Hf and/or Ta containing Si-based ceramic nanocomposites possess outstanding EM performance and versatile designability ranging from EM absorbing to shielding behavior, which shows substantial progress beyond the state of the art. The relationship between the obtained nano/microstructure of the synthesized Si-based ceramics and their property features will be highlighted.

4:40 PM

(ICACC-HON-025-2018) Structure and properties of Si_3N_4/graphene nanocomposites (Invited)

C. Balazsi*[1]; K. Balazsi[1]

1. HAS Centre for Energy Research, Hungary

Advanced ceramic materials have proved their superior wear resistance as well as mechanical and chemical properties in a wide range of industrial applications. Today there are standard materials for components and tools that are exposed to severe tribological, thermal or corrosive conditions. The main aim of this work is to develop novel, highly efficient tribological systems on the basis of ceramic/graphene nanocomposites as well as to prove their superior quality and to demonstrate their suitability for technical applications e.g. for slide bearings and face seals in aqueous media. Current research in the field of ceramic nanocomposites shows that is possible to make ceramic materials with improved mechanical and tribological properties by incorporating graphene into the Si_3N_4 structure. Multilayered graphene (MLG) was prepared by attritor milling at 10 hours intensive milling of few micrometer sized graphite powders. Si_3N_4/MLG nanocomposites were prepared by attritor milling and sintered by hot pressing (HP). The Si_3N_4 ceramics were produced with 1wt%, 3wt%, 5wt% and 10wt% content of MLG. The tribological behavior of composites in different environments was investigated and showed the decreasing character of wear at increased MLG content. This new approach is very promising, since ceramic microstructures can be designed with high toughness and provide improved wear resistance at low friction.

5:00 PM

(ICACC-HON-026-2018) Heat Resistant Liquid-Phase Sintered Silicon Carbide Ceramics (Invited)

Y. Kim*[1]; T. Nishimura[2]

1. University of Seoul, Dept. of Materials Science & Engineering, Republic of Korea
2. National Institute for Materials Science (NIMS), Japan

Microstructure and high temperature strength of three different liquid-phase sintered SiC ceramics with a small amount of additives were characterized: SiC ceramic sintered with 1 wt% AlN-Lu_2O_3 (SCALu); SiC ceramic sintered with 1 vol% Y_2O_3-Sc_2O_3 (SCYSc); and SiC ceramic sintered with 2000 ppm Y_2O_3 (SCY2). Observation of the ceramics using HRTEM showed the followings: the SCALu exhibited a lack of amorphous films in both SiC/SiC boundaries and junction areas; the SCYSc exhibited both clean and crystallized SiC/SiC boundaries, as well as clean SiC/junction phase boundaries; and the SCY2 exhibited a clean SiC/SiC boundaries without an intergranular glassy phase and an amorphous Y-Si-O-C-N junction phase with nanocrystalline Y-containing phase embedded. The SCALu and SCY2 maintained 100% of their room temperature (RT) strength (630 MPa and 550 MPa, respectively) up to 1600°C and 1700°C, respectively. Degradation at temperatures above those temperatures was due to softening of the grain boundary phase, as evidenced by the nonlinear behavior of load-displacement curves. The SCYSc maintained 93% of its RT strength (536 MPa) up to 1600°C, and showed rapid degradation at temperatures above 1700°C. The present results suggest that LPS-SiC could be more heat resistant than solid state sintered SiC ceramics by judicious selection of sintering additive composition with a minimal amount of additive content.

7th Global Young Investigator Forum

Novel Ceramic Processing Methods and Synthesis Routes

Room: Coquina Salon G

Session Chair: Daniele Benetti, Institut National de la Recherche Scientifique

1:30 PM

(ICACC-GYIF-015-2018) Towards the colonization of Mars by means of in-situ resource utilization: Slip cast ceramics from Martian soil simulant (Invited)

D. Karl*[1]; F. Kamutzki[1]; A. Zocca[2]; O. Goerke[1]; J. Guenster[2]; A. Gurlo[1]

1. Technical University of Berlin, Chair of Advanced Ceramic Materials, Germany
2. Federal Institute for Materials Research and Testing (BAM), Ceramic Processing and Biomaterials, Germany

Planet Mars is the most suited candidate for human colonisation as it has similar size, close proximity and similar weather conditions to Earth. In the course of human exploration and subsequent colonisation of our neighbouring planet mankind will need to build a variety of things on site. The only viable way to produce on Mars will be to use in-situ resource utilization (ISRU). ISRU is the practice of on-site collection, processing, storing and use of native materials encountered in the course of human or robotic space exploration, replacing materials that otherwise would have to be transported from earth. On Mars the most readily available resources are surface minerals, called regolith. Regolith's chemical makeup makes conceivable the extraction of two classes of materials - ceramics (including glass) and metals. Focusing on ceramic materials, we wish to present the first slip/slurry based processing route to manufacture ceramics on Mars, demonstrating that by applying exclusively Martian resources from fabrication to finished product, a route similar to traditional ceramics can be established for the future colonization of Mars.

Furthermore, we will explore the possibility to use water-based slurries in conjunction with additive manufacturing technologies like layer-wise slurry deposition (LSD) to gain remotely controlled production capabilities on Mars.

2:00 PM

(ICACC-GYIF-016-2018) Preceramic Polymer Routes to Advanced Ceramics (Invited)

E. Ionescu*[1]

1. Technical University Darmstadt, Materials Science, Germany

Polymer-derived ceramics (PDCs) have been addressed in the last decades and were shown to possess intriguing properties which make them excellent candidates as structural and (multi)functional materials. PDCs can be synthesized via polymer-to-ceramic conversion of suitable single-source precursors, leading in a first step to amorphous single-phase materials, which subsequently undergo phase separation and crystallization processes to furnish bi- or multi-phase ceramic nanocomposites. In the present talk, the conversion of the single-source precursors into PDC, which are of amorphous nature, as well as subsequent phase separation and crystallization processes occurring at high temperatures will be addressed in detail. Special emphasis will be set on describing the intimate relationship between the molecular architecture of the single-source precursors and the phase composition / microstructural features of the resulting PDCs. Preparative concepts for the knowledge-based design of PDCs with tailored phase compositions and property profiles as well as selected energy-related applications will be highlighted and discussed.

2:30 PM

(ICACC-GYIF-017-2018) Investigation of porous alumina derived from a slurry including aluminum powder and polysiloxane

K. Kita*[1]; N. Kondo[1]

1. AIST, National Institute of Advanced Industrial Science and Technology, Structual Materials Research Institute, Japan

Porous alumina is one of promising materials for light structure material, substrate, gas separator, filter, insulator etc. To obtain porous alumina, we tried sintering porous alumina by using a mixture containing alumina powder and aluminum powder coated by a polysiloxane. Due to transformation of the polysiloxane into aluminum silicate or mullite, porous alumina could be obtained by using the mixture after sintering at 800 degree C or over. Moreover, the density and porosity of porous alumina were almost fixed. In this study, we tried sintering porous alumina by using slurry including alumina powders with various diameters for adjusting the density and porosity of the porous alumina.

3:10 PM

(ICACC-GYIF-018-2018) Novel Low-temperature Process for the Synthesis of Perovskite-type Oxide Fine Powders (Invited)

Y. Yamaguchi*[1]; H. Shimada[1]; H. Sumi[1]; T. Yamaguchi[1]; K. Hamamoto[1]; K. Nomura[1]; Y. Fujishiro[1]

1. National Institute of Advanced Industrial Science and Technology (AIST), Japan

Perovskite oxides are widely used as the functional ceramic materials. To obtain the high function, many kinds of perovskite oxides were prepared by some solution methods. In this study, a novel large-quantity synthesis method of perovskite fine powder was developed. Some perovskite oxides (ABO_3) were successfully prepared by only leaving the starting material powder mixture at around 25 °C. This starting material included an alkaline earth hydroxide ($A(OH)_2$) and a hydrous metal oxide gel ($BO_2{\cdot}nH_2O$). The synthesis reaction occurred based on an acid-base reaction between the hydroxide and the oxide gel. For example, strontium titanate ($SrTiO_3$) could be prepared at 25 °C by leaving the powder mixture including strontium hydroxide octahydrate ($Sr(OH)_2{\cdot}8H_2O$) and titania hydrous gel ($TiO_2{\cdot}nH_2O$) for 10 days. In spite of the room-temperature synthesis, this $SrTiO_3$ had a quite high crystallinity, which was comparable to that prepared by a solid-state synthesis at 1400 °C. The particle size of $SrTiO_3$ was approximately 80 nm. In this synthesis, the hydrated water in oxide gel played an important role to promote the present synthesis reaction. In addition, it was found that the tolerance factor of perovskite oxide was also important for the formation of perovskite structure. When the tolerance factor was larger than 1, perovskite oxides could be synthesized at around 25 °C.

3:40 PM

(ICACC-GYIF-019-2018) Complex colloidal approaches for direct ink writing of ceramic suspensions (Invited)

E. Garcia-Tunon*[1]

1. University of Liverpool, Materials Innovation Factory & School of Engineering, United Kingdom

As new technologies in key areas such as energy and medicine develop, the demand for state-of-the art fabrication to create complex multifunctional structures and devices also grows. Additive Manufacturing (AM) promises a revolution on how things are made, bringing freedom to create completely new designs and more efficient devices. But making this a reality, demands a major effort in material development, since the commercial applications in AM are mostly limited to a number of metals and polymers. Aiming to address current challenges in the field, we have developed different water-based formulation for robocasting; an AM technique also known as direct ink writing and 3D printing. The formulations are designed following three strategies based on: 'responsive' building blocks (using pH responsive surfactants); hydrogels (thermo-responsive copolymers); and 2D colloids as rheology modifiers. The three approaches are flexible, clean and robust. They provide 3D-printable pastes with controlled viscoelasticity for materials with different chemistries and shapes: from oxide and non-oxide ceramics and metals to polymers and even steel. This enables printing complex 3D structures with controlled architecture at multiple scale lengths for health and energy applications. This talk will illustrate these approaches, the optimum rheological behaviour for the printing process and the properties of the final objects.

4:10 PM

(ICACC-GYIF-020-2018) Young modulus and electrical conductivity of GNP/3YTZP composites prepared by PLS

C. Lopez Pernia*[1]; R. Poyato Galán[2]; Á. Gallardo López[1]; A. Morales Rodríguez[1]

1. Universidad de Sevilla, Física de la Materia Condensada, Spain
2. Instituto de Ciencia de Materiales de Sevilla, Spain

Multilayered graphene is a potential nano-scale reinforcement for ceramics, since it can improve the mechanical properties and make the ceramics electrically conductive. These facts make graphene-ceramic composites very interesting for some applications like electro-discharge machining. These advanced composites are typically prepared by wet powder processing followed by a sintering technique which usually involves uniaxial pressure, such as spark plasma sintering or hot pressing. However, these sintering techniques require expensive equipments and produce highly anisotropic materials. Besides, there is a lack of studies about the properties of graphene ceramic matrix composites prepared by conventional pressureless sintering (PLS). This work focuses on the properties of composites of 3YTZP with inclusions of graphene nanoplatelets (GNP) sintered by PLS in a wide temperature range. The elastic modulus of the composites is measured by the impulse excitation technique. The electrical conductivity is studied for two orientations of the composites: parallel and perpendicular to the pellet axis, to detect anisotropy in this property. The effects of the

powder processing, the GNP content and the sintering temperature on the Young modulus and electrical conductivity of the composites are analysed and discussed.

4:30 PM

(ICACC-GYIF-021-2018) Investigation of the diffusion path for the nitridation of $TiSi_2$ powder with nickel addition and nitride ceramic synthesis

L. Nouvian*[1]

1. LCTS - CNRS, France

The nitridation mechanism of a $TiSi_2$ solid with nickel addition was examined. First, a $TiSi_2$/Ni interface was obtained by Spark Plasma Sintering (SPS) and isothermally treated at 1080°C for durations between 1 and 4 hours under normal pressure and continuous flow of pure nitrogen or argon. The diffusion paths were determined with the help of the ternary diagram of the system Ti-Si-Ni and the chemical quantification of the phases formed during the treatments. During the treatment, a eutectic liquid, NiSi, is formed and permits to enhance the reaction of the system. Knowing that, the influence of parameters such as temperature, duration and granulometry was examined for the nitridation of a powder containing 10mol.% of Ni and 90mol.% of $TiSi_2$. The conversion rates estimated thanks to the mass variations during the treatment showed that the reaction is complete for temperatures between 1060°C and 1100°C, and durations between 3 and 5 hours. The phases formed during the nitridation were TiN, Si_3N_4 and $Ti_4Ni_4Si_7$ or NiSi depending on the duration and the temperature. It showed that for experimental conversion rate reaching 82%, the NiSi phase is formed whereas under 82% it is the ternary component $Ti_4Ni_4Si_7$ which exists.

Poster Session A

Room: Ocean Center Arena

5:00 PM

(ICACC-S2-P002-2018) YOF Coatings Prepared by Suspension Plasma Spray

S. Lee*[1]; Y. Oh[1]

1. Korea Institute of Ceramic Engineering and Technology (KICET), Republic of Korea

We have fabricated YOF coatings using suspension plasma spray (SPS) technique. The YOF coating has been studied as a promising protective material against Fluorine-base plasma. In this study, YOF was in-situ synthesized during SPS coating using Y_2O_3 and YF_3 powders as raw materials by controlling the plasma spray condition. In addition, YOF material was syntheised through solid state reactions and then it was used for SPS.

(ICACC-S2-P003-2018) High Temperature Insulating Properties of Aerosol Deposited Alumina Films

M. Schubert*[1]; N. Leupold[1]; J. Kita[1]; R. Moos[1]

1. University of Bayreuth, Department for Functional Materials, Germany

Due to its very low electrical conductivity, alumina is a widely used material for high insulating films. As alumina is typically sintered above 1200 °C, low melting substrates like polymers or low-melting metals are excluded for applications. The Aerosol Deposition Method (ADM) is a novel ceramic coating technique. It allows manufacturing of dense ceramic films at room temperature directly from ceramic powders without any high temperature sintering steps. Thereby, it is possible to produce high insulating alumina films on nearly all types of substrates. As known from literature, the functional properties of aerosol deposited films directly after deposition are not completely identical with bulk samples. Nevertheless, annealing at temperatures around 500 °C can improve these properties. Therefore, it is necessary to know the electrical resistivity of aerosol deposited alumina films after deposition and after annealing. In this study, alumina films were deposited by ADM on a three-electrode setup with guard ring and the electrical conductivity was measured between 400 °C and 900 °C by direct current measurements according to ASTM D257 or IEC 60093. The measured conductivities correlate well with literature values and can be improved by annealing at 900 °C. The processed films show an even higher resistivity than conventional alumina substrates.

(ICACC-S2-P004-2018) Effect of difference in thickness of coating and type of substrate on aerosol deposited mullite coating under heat exposure

T. Shibuya*[1]; A. Iuchi[1]; T. Kayama[1]; M. Hasegawa[1]

1. Yokohama National University, Japan

Environmental barrier coatings (EBCs) are essential for SiC fiber reinforced SiC ceramics matrix composites (SiC/SiC CMCs) to protect from heat and combustion atmosphere which has oxygen and water vapor in next generation turbine engines. Typical EBCs are composed of several ceramics layers formed by air plasma spraying (APS). However, the APS coatings is porous and has amorphous phases. Due to the shrinkage of the coatings by sintering and crystallization at high temperature, vertical cracks initiate at EBCs. This promotes substrate oxidation and leads the coating spallation. In order to prevent it, dense coatings are required from the as-deposited state. Aerosol deposition (AD) is known to fabricate a dense ceramic coating directly on a substrate at room temperature. In this study, mullite which is one of the component materials for EBCs is deposited on two kinds of substrates: silicon and SiAlON. Effect of difference in thickness of layer and type of substrates on AD mullite coating under heat exposure in an air at 1573 K is investigated. In the mullite coating on the silicon substrate, aluminum and oxygen mutually diffused during the heat exposure. The mullite decomposed at the vicinity of the interface between mullite and silicon. On the other hand, decomposition of mullite delayed on the SiAlON substrates in same heat exposure condition.

(ICACC-S2-P005-2018) Evaluation of Ti_2AlC as Environmental Barrier Coating for Ti Alloys

Y. Chen*[1]; A. Pilchak[2]; R. John[2]; I. Karaman[1]; M. Radovic[1]

1. Texas A&M University, Materials Science & Engineering, USA
2. Air Force Research Laboratory, USA

Ti_2AlC MAX phase has promising applications at elevated temperature due to its excellent oxidation resistance. A thin layer of Ti_2AlC was is successfully diffusion bonded onto Ti alloy Ti6242 at 800 °C by Pulsed Electric Current Sintering (PECS). Interfacial phases and their evolution after long exposure to high temperatures for different times were characterized using scanning electron microscopy and electron backscatter diffraction analysis. It was found out that as a result of diffusion of Al from Ti_2AlC to Ti alloy, β phase in Ti alloy transforms to α phase, and then intermetallic TiAl and $TiAl_2$ starts to form at the interface when Al amount is sufficiently high. The interfacial structure is confirmed to be Ti_2AlC/$TiAl_2$/TiAl/α-Ti alloy. Nano-indentation tests showed a smooth change of the elastic moduli thorough the interface between Ti_2AlC and Ti alloy. Oxidation tests carried out at 800 °C under static air for 100 h showed that Ti_2AlC can be used as protective coating to prevent severe oxidation of Ti6242 alloy. Oxidation of Ti6242 samples with different thickness of Ti_2AlC protective layers was also studied and discussed in more details.

(ICACC-S2-P006-2018) Texture Development of Aerosol Deposited Alumina Coating for Advanced Environmental Barrier Coatings

K. Kimura*[1]; M. Komuro[1]; M. Hasegawa[1]; M. Tanaka[2]; S. Kitaoka[2]; Y. Kagawa[3]

1. Yokohama National University, Solid State Materials and Engineering, Japan
2. Japan Fine Ceramics Center, Japan
3. Tokyo University of Science, Japan

An advanced environmental barrier coatings (EBCs) composed of a multilayer of alumina and $Y_2Ti_2O_7$ which reflect thermal energy from radiation and prevent oxidation of the substrate of SiC fiber reinforced SiC ceramic matrix composites. During the high temperature service, structure and microstructure of EBCs are required to be stable. Thus, EBCs are necessary to have a dense ceramic coating from the as-deposit state. Aerosol deposition (AD) is known as a ceramic coating process which can form dense ceramic coatings at room temperature by impacting fine ceramics powders on the substrate. In this study, texture development of alumina coating by different AD process conditions such as the distance between the nozzle and substrate, the angle of the flow direction from the nozzle to the substrate plane, and gas flow rate were evaluated. Dense and crystalline coatings were formed without chemical composition change between coatings and raw powders. Fiber texture where the (0001) plane of alumina is tilted about 15° from the coating plane was formed in case that the flow direction is 90°. When the flow direction is 60°, (0001) plane is tilted about 25° from the coating plane. Further, it is informed that (0001) plane is declined about 40° from the coating plane. The difference in formed texture will be discussed based on the change in slip system and deformation mode of the particle.

(ICACC-S2-P007-2018) Oxidation Study of Ultra High Temperature Ceramic Coatings Based on HfSiCN

D. Sacksteder[1]; D. Waters[1]; D. Zhu*[1]

1. NASA Glenn Research, Materials and Structures Division, USA

High temperature fiber-reinforced ceramic matrix composites (CMCs) are important for aerospace applications because of their low density, high strength, and significantly higher temperature capabilities compared to conventional metallic systems. The use of the SiCf/SiC and Cf/SiC CMCs allows the design of lighter-weight, more fuel efficient aircraft engines and also more advanced spacecraft airframe thermal protection systems However, CMCs have to be protected with advanced environmental barrier coatings, when they are incorporated into components for the harsh environments such as in aircraft engine or spacecraft applications. In this study, high temperature oxidation kinetics of an advanced HfSiCN coating on Cf/SiC CMC substrates were investigated at 1300, 1400, and 1500°C by using thermogravimetric analysis (TGA). The coating oxidation reaction parabolic rate constant and activation energy were estimated from the experimental results. The oxidation reaction studies showed that the coatings formed the most stable, predominant HfSiO4-HfO2 scales at 1400°C. A peroxidation test at 1400°C then followed by subsequent oxidation tests at various temperatures also showed more adherent and slower scale growth because of reduced the initial transient oxidation stage and increased HfSiO4-HfO2 content in the scales formed on the HfSiCN coatings.

(ICACC-S3-P009-2018) Cation Interdiffusion between Lanthanum Strontium Manganite and Yttria-Stabilized Zirconia

Y. Yu*[1]; J. Liu[1]; Y. Jee[1]; P. Ohodnicki[1]; H. Abernathy[1]; T. Kalapos[1]; G. Hackett[1]

1. National Energy Technology Laboratory, USA

Lanthanum Strontium Manganite (LSM) and Yttria-Stabilized Zirconia (YSZ) have been chosen and widely used as cathode and electrolyte materials for solid oxide fuel cells (SOFCs) due to their various conductive, mechanical, and catalytic properties. However, during operation, the gradual change in the composition bordering two phase boundaries produces a corresponding degradation in cell performance. To minimize the SOFC degradation, this change between the two phases must be understood. This study focuses on quantification of the interdiffusion of cations between LSM and YSZ at different temperatures, oxygen partial pressures and polarization conditions. In this study, cation interdiffusion behaviors across LSM/YSZ interfaces have been studied and quantified by transmission electron microscope (TEM)-based energy dispersive X-ray spectroscopy (EDS). Porous electrode samples as well as thin film LSM on single crystal YSZ model systems were used in this study. These samples have been subjected to testing under various temperatures, atmospheres and amounts of polarization prior to investigation using TEM. The effects of such testing conditions on cation interdiffusion profiles will be discussed.

(ICACC-S3-P010-2018) Relationship between LSM/CeO_2 nanocomposite composition and cathode properties

H. Sakuma*[1]; S. Suda[1]; J. Wiff[2]

1. Shizuoka University, Japan
2. FCO Power Inc., Japan

Co-sintering of SOFC cells is indispensable for fabricating high-efficient compact SOFC stacks. $La_{1-x}Sr_xMnO_3$ (LSM) shows preferable low-reactivity with fluorite electrolytes during sintering at high temperatures, but lower cathode properties than LSC or LCSF because of poor oxygen ion conductivity. We then prepared nanocomposite of a-site deficient LSM and CeO_2 to improve oxygen diffusion of LSM. Adequate glycine (GL) and Pechini(PE) processes made nano-scale dense LSM/CeO_2 composites. Oxygen diffusion coefficient (D) and surface reaction constant (k) were estimated by electrical conductivity relaxation (ECR) method. The LSM/CeO_2 nanocomposite that was composed of 10 mol% CeO_2 much improved D value as compared to LSM. However, LSM composition of the nanocomposite was much altered by easy La diffusion from LSM to CeO_2. The amount of diffusion depended on composite fabrication processes. PE process showed relative large diffusion, but solid-state process showed no diffusion. The La diffusion to CeO_2 makes La doped CeO_2 (LDC) and it increases oxygen ion conductivity of CeO_2, but diminishing La of LSM would much decrease cathode properties. More than 25% La of LSM was moved from LSM to CeO_2 when the nanocomposite was prepared by GL or PE method. The composition optimization by considering La diffusion depending on preparation processes was essential to make most use of LSM/CeO_2 nanocomposite cathode.

(ICACC-S3-P011-2018) Preparation and Characterization of $BaY_{0.2}Ce_{0.7}Zr_{0.1}O_{3-\delta}$ Ceramic Powder by Glycine Nitrate Combustion (GNC) Process for Proton Solid Oxide Fuel Cell

W. Kao[1]; T. Lin*[1]; M. Liao[1]; H. Kuo[1]; C. Yeh[1]; Y. Chen[1]; R. Lee[1]

1. Institute of Nuclear Energy Research, Nuclear Fuels and Materials Division, Taiwan

Proton conducting SOFC owns higher ionic conductivity than that of the conventional SOFC with oxygen ion conducting electrolyte. $BaY_{0.2}Ce_{0.7}Zr_{0.1}O_{3-\delta}$ (BYCZ) is a promising candidate as electrolyte for a proton SOFC (P-SOFC). In this work, the ceramic powder of BYCZ was synthesized by glycine nitrate combustion (GNC) process. Characterizations of the powders were executed by X-ray diffractometer (XRD), field-emission scanning electron microscopy (FE-SEM), and Brunauer-Emmett-Teller (BET) analyses. The results reveal that the single phase of perovskite structure of BYCZ powder occurs at calcination temperature of 1550 °C and the average value of the thermal expansion coefficient (TEC) is 12.6×10^{-6} K^{-1} at the temperature range of room temperature to 700 °C. In addition, the relative density of BYCZ powder can be achieved near 100 % at 1550 °C. The result indicates that the crystal phase of BYCZ is cubic perovskite structure at the calcination temperature of 1550 °C.

Further application on the BYCZ-based P-SOFC cell performance evaluation will be conducted.

(ICACC-S3-P012-2018) Ionic Conductivity and Phase Stabilization in Zirconia-Scandia-Europia

J. P. Souza[1]; E. N. Muccillo*[1]

1. Energy and Nuclear Research Institute, Brazil

Zirconia-8 mol% yttria is widely used as electrolyte in solid oxide fuel cells, due to its high ionic conductivity and good mechanical and chemical properties. Nevertheless, scandia-stabilized zirconia is recognized as exhibiting the highest ionic conductivity among zirconia-based solid electrolytes. Stabilization of the high symmetry and high ionic conductivity cubic phase in this system is non-trivial. Introduction of a second additive (or dopant) is a usual strategy to overcome that constraint. In this work, europium oxide has been added to 10 mol% scandia-stabilized zirconia (10ScSZ) aiming full stabilization of the cubic structure at room temperature. The influence of europia addition (up to 1.25 mol%) on phase stabilization and ionic conductivity of 10ScSZ was investigated by differential scanning calorimetry and impedance spectroscopy. The higher is the fraction of europia into solid solution the lower is the temperature of the endothermic event due to phase transition. The cubic phase is fully stabilized at room temperature for 1 mol% europia addition. The grain and total ionic conductivity show typical Arrhenius behavior. The magnitude of the total conductivity increases with increasing europia addition.

(ICACC-S3-P013-2018) Influence of Calcium Addition on the Electrical Conductivity of Samarium Doped Ceria

S. L. Reis*[1]; E. N. Muccillo[1]

1. Institute of Nuclear Energy Research, Brazil

Ceria-based ceramics have been proposed to be used as solid electrolyte in solid oxide fuel cells operating at intermediate (500-750°C) temperatures, due to their high ionic conductivity. Samarium ion is recognized as one of the most reliable dopants for cerium oxide because of its lower association energy with oxygen vacancies. One challenge posed to ceria-based solid electrolytes is how to improve sinterability, avoiding high temperatures to attain good densification. Thermal treatments at high temperatures may reduce Ce^{4+} to Ce^{3+}, favoring electron transport and generation of micropores in the sintered electrolyte. Introduction of a second additive is a usual approach to overcome these constraints. In this work calcium ion was chosen as the second additive. $Sm_{0.2-x}Ca_xCe_{0.8}O_{1.9-x/2}$ compositions, with x = 0, 0.025, 0.05, 0.1, 0.15 and 0.2, were prepared by solid-state reaction, and the influence of the additive content on densification and ionic conductivity was investigated. All compositions were found to have cubic fluorite-type structure. The optimal composition was $Sm_{0.175}Ca_{0.025}Ce_{0.8}O_{1.888}$, which showed a relative density of 97%, single phase and higher ionic conductivity than the $Sm_{0.2}Ce_{0.8}O_{1.9}$ parent electrolyte.

(ICACC-S3-P014-2018) Aerosol Deposition of barium-based perovskites as solid electrolyte film for fuel cells

J. Exner*[1]; T. Nazarenus[1]; H. Pöpke[2]; F. Fuchs[2]; J. Kita[1]; R. Moos[1]

1. University of Bayreuth, Department of Functional Materials, Germany
2. Kerafol Keramische Folien GmbH, SOFC Department, Germany

Solid oxide fuel cells (SOFC) are of interest for clean and reliable energy conversion technologies. State of the art SOFCs based on ScSZ or YSZ (scandia or yttria stabilized zirconia) electrolytes that conduct oxide ions. Typically, they require high temperatures above 800 °C. In contrast, barium- or strontium-based perovskites are receiving increased attention due to their potential use as proton conducting membranes that could already be operated at temperatures of 500 °C and even below. However, a major drawback of these materials limiting their commercial use is the high sintering temperature of 1500 °C to 1700 °C. To overcome the necessity of sintering, we intended to form dense and well-adhering perovskite films by Aerosol Deposition (AD). The unique feature of this spray coating technique is the possibility to form dense, nanocrystalline ceramic films directly from the ceramic powder without the need for a heat treatment during or after deposition. Three different compounds were synthesized, namely barium zirconate ($BaZrO_3$), barium cerate ($BaCeO_3$) and barium stannate ($BaSnO_3$). Each compound was doped with 10 % and 20 % yttrium, respectively. Resulting films are dense and between 2 µm and 10 µm thick. The crystal structure of the powder was retained during coating and was still present in films. Especially $BaZrO_3$ and $BaCeO_3$ films feature high conductivities of up to 10^{-2} S/cm at 800 °C.

(ICACC-S3-P015-2018) Residual Stress Measurement of 8 mol % YSZ Coating for SOFC Application

Z. Ruhma[1]; K. Yashiro[1]; F. Iguchi*[2]; T. Kawada[1]

1. Tohoku University, Graduate School of Environmental Studies, Japan
2. Tohoku University, Graduate School of Engineering, Japan

As for SOFC application, 8 mol% YSZ coating usually is fabricated by using wet ceramic or physical deposition route. In contrast with wet ceramic method, physical deposition route such as PLD and plasma spraying, leaves residual stress because of difference in temperature and material properties of coating-substrate during deposition process. By knowing changes in residual stress, deposition condition, material properties, and defects inside a coating, it is possible to explain how the defects were formed, therefore the desired dense, crack-free YSZ coating can be fabricated. We tried to quantify residual stress inside PLD deposited 8 mol% YSZ by $\sin^2$ psi XRD and raman method. As for XRD method, by using (422)-cubic YSZ, respectively, we are able to quantify the residual stress inside the deposited film. However, as for raman method, one has to know the raman peak shift with variation of stress inside material, so that by measuring how much a raman peak is shifting, one can quantify the residual stress that is contained inside of material.

(ICACC-S3-P016-2018) Reactive Spray Deposition Technology (RSDT): A flamed-based process for SOFC diffusion blocking layer and cathode

T. Ebaugh*[1]; L. Bonville[2]; R. Maric[2]

1. University of Connecticut, Chemical Engineering, USA
2. Center for Clean Energy Engineering, USA

An important focus for SOFCs is the improvement of cell lifetime and reduction in materials costs by developing cells capable of running in an intermediate temperature range (773-923K). In this range, LSCF ($La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_{3-\delta}$) is a promising cathode material due to its good electronic and ionic conductivity. A GDC ($Ce_{0.9}Gd_{0.1}O_{1.95}$) blocking layer must be applied between the YSZ ($Zr_{0.92}Y_{0.08}O_{2-\delta}$) electrolyte and the LSCF cathode to prevent the formation of $SrZrO_3$. The presence of $SrZrO_3$ leads to resistive losses. RSDT is used to deposit a GDC diffusion blocking layer and a LSCF cathode layer on half cells with NiO,YSZ anode support, NiO,YSZ anode functional layer, and YSZ electrolyte. The RSDT process uses inexpensive solvents and precursors (i.e. toluene and metal 2-ethylhexanoates) in open atmosphere. With RSDT, a dense GDC layer with good adhesion is achieved at a relatively low temperature (~1273K) without sintering. This leads to decreased production costs and limits the inter-diffusion of the YSZ and GDC. The cathode is deposited using a slurry of pre-synthesized LSCF nanoparticles, which, in some experiments is mixed with LSCF nanoparticles directly from the RSDT flame. Test data suggest that RSDT cathodes with this mixture of LSCF nanoparticles have better performance (1.4 W/cm^2) than RSDT cathodes made from only the LSCF slurry.

(ICACC-S3-P017-2018) Development of Fe/Cr Alloy-Supported Solid Oxide Fuel Cell by Plasma Technique

S. Yang*[1]; C. Tsai[1]; C. Chang[1]; C. Fu[1]; R. Lee[1]; H. Lee[2]

1. Institute of Nuclear Energy Research Atomic Energy Council, Taiwan
2. Porite Taiwan Co., Ltd., Taiwan

In this study, the large-scale mold for compression molding process is prepared and a compression load of 200 ton is applied to form a porous alloy specimen with dimension of 11×11×1.2 cm. The pyrolyzable filler is used in the period of manufacturing processes in order to produce porous interconnected networks of iron (Fe)-containing chromium (Cr)-based alloy. The specimens are sintered in hydrogen at the temperature of 1350°C to obtain porous alloy substrate. The flexural strength of porous alloy supporting component is measured in this work. The strength of porous alloy substrate (Sintered at 1350°C) in three-point flexure at 25°C and 750°C are 158 MPa and 52.5 MPa, respectively. Metal-supported solid oxide fuel cells (MS-SOFCs) are fabricated by thermal plasma spraying technique and the anode ($Ce_{0.55}La_{0.45}O_{2-\delta}$ -NiO,LDC-NiO), electrolyte ($La_{0.8}Sr_{0.2}Ga_{0.8}Mg_{0.2}O_{3-\delta}$, LSGM) and cathode ($Sm_{0.5}Sr_{0.5}CoO_{3-\delta}$, SSC) functional layers are deposited onto porous alloy substrate. The 10×10 cm^2 MS-SOFC with effective electrode area of 81 cm^2 shows the open circuit voltages is 1.03 V at 750°C. The measured maximum output power densities (@0.68V) of this cell has reached 411 mW/cm^2 at 750°C by employing hydrogen as fuel and air as oxidant.

(ICACC-S3-P018-2018) Effects of composite ratio of vermiculite/talc seal material on gas leak properties

J. Xu*[1]; S. Suda[1]

1. Shizuoka University, Electronics and Materials Science, Japan

Glass-free high-temperature gas seals are attractive for SOFC application because of easy set-up and excellent thermal cycle durability. Mica is a traditional candidate for SOFC compression seals, but relatively large compression load is required and gas leakage through the interface between swelled or rough substrate would often occur when mica is used as SOFC compression seals. We then investigated vermiculite/talc composite seals to overcome the interfacial gas leakage issues. Vermiculite irreversibly expands by firing at high temperatures and calcined vermiculite shows excellent compression properties. The vermiculite calcined at moderate temperature would expand still more during it heats at an SOFC operating temperature. This self-inflation property would settle the interfacial gas leakage issues. The composite seals of talc and vermiculite that was calcined at various temperatures were then prepared by cold and hot pressing with mixed powder. The expansion rate by heating the composite seals from room temperature to 800°C much depended on the calcination temperature and particle size of vermiculite. The self-inflation composite seal with no delamination or cracks were obtained by adjusting these parameters. The vertical expansion rate of the seals was 0.4-30% whereas the horizontal expansion was less than 1.7%. This vertical self-inflation would much diminish the interfacial gas leakage.

(ICACC-S3-P019-2018) Computational Phase Studies in the La-Sr-Ga-Ni-O System

G. Soydan[1]; E. Kondakci[1]; N. Solak*[1]

1. Istanbul Technical University, Metallurgical and Materials Eng, Turkey

Strontium and magnesium doped lanthanum gallate (LSGM) perovskite-type compounds are considered as promising solid electrolytes for intermediate temperature fuel cell (IT-SOFC) applications. For the practical use of these materials not only the thermodynamic stability of the electrolyte and electrodes themselves, but also the reactivity between the electrolyte and electrodes are needed. In the presents work, reactivity in the Sr and Mg ions incorporated $LaGaO_3$ with Ni system, which is the main constituent of the anodes for intermediate temperature solid oxide fuel cells (IT-SOFC), has been investigated both computationally and experimentally. An isobarothermal section of the La-Sr-Ga-Ni-O system was constructed. It has been found that NiO reacts with $LaGaO_3$ and there exist extended solid solutions of $La(Ga,Ni)O_3$, $La(Ga,Ni)_2O_4$ and $La_4(Ni,Ga)_3O_{10}$. It has also been found that, in the presence of Sr ions and direct contact conditions, NiO diffusion from the anode and cathode materials to $LaGaO_3$ is inevitable.

(ICACC-S4-P020-2018) DFT Study of Yttrium Adsorption on Boron suboxide (0001) Surface

J. S. Dunn*[1]; K. D. Behler[1]; J. LaSalvia[1]; M. P. Harmer[2]; C. J. Marvel[2]

1. U.S. Army Research Laboratory, USA
2. Lehigh University, Materials Science and Engineering, USA

In this paper we developed a first principles Density Functional Theory (DFT) model of the (0001) oriented boron suboxide (B_6O) surface and studied the structural and electronic reconstructions along the surface as a result of surface termination (boron terminated vs oxygen terminated), icosahedral stacking (ABCABC vs ABAB), and Yttrium adsorption. We then performed a qualitative analysis of the Electron Localization Function (ELF), Electron Density Difference (EDD), band structure, and partial density of states (PDOS) to determine the effect of these different surface reconstructions on the chemistry and bonding behavior at the surface. Finally, to validate the model, we compare model predictions to aberration-corrected Scanning Transmission Electron Microscopy (AC-STEM) with High-Angle Annular Dark Field (STEM-HAADF) measurements of the atomic positions for the surface adsorbed Yttrium along B_6O grain boundaries.

(ICACC-S4-P021-2018) The Effect of Heating on Indented Boron Carbides of Varying Stoichiometry

M. C. Schaefer*[1]; V. Domnich[1]; R. A. Haber[1]

1. Rutgers University, Materials Science and Engineering, USA

Boron carbide is a great material for armor applications thanks to its high hardness, low density, and high Hugoniot Elastic Limit (HEL). However, past the HEL limit, boron carbide fails from the formation of pressure-induced nanometer thick amorphous bands. This led to research into failure mechanisms from plate impact experiments. Additionally, it is believed that the material can undergo extensive heating during a high impact, leading many to wonder how heating effects the performance of the material. Using boron carbide samples spanning the homogeneity range, the effect of heating was investigated on the amorphous phase of boron carbides of varying stoichiometries. Samples ranged from 9.6 at.% to 19.7 at.% carbon, and were examined under raman spectroscopy. Vickers indentation (5 N) were made on polished sample surfaces to induce the amorphization in the samples. Samples were heated in situ to temperatures in excess of 800 °C to observe the stability of the amorphous phase, and the disappearance of the amorphous raman signature at higher temperatures. The hot stage was vacuumed down and backfilled with inert gas to minimize oxidation from the atmosphere. The results suggest lower temperature stability of the amorphous phase in boron-rich boron carbide.

(ICACC-S4-P022-2018) Optimization of Consolidation Parameters and the Resulting Mechanical Properties of Bulk Silicon Doped Boron Carbides

M. Gagnepain*[1]; A. M. Etzold[1]; A. U. Khan[1]; V. Domnich[1]; C. Hwang[1]; K. D. Behler[2]; J. LaSalvia[2]; R. A. Haber[1]

1. Rutgers University, Materials Science and Engineering, USA
2. US Army Research Laboratory, USA

Previous work at Rutgers University has shown that silicon doping in boron carbide may mitigate the issue of amorphization in high pressure events. In the present study, iterative trials varying temperature, pressure, and dwell time in hot pressing and spark plasma sintering of powder mixtures containing boron carbide with low free carbon content, silicon hexaboride as well as elemental boron

were performed in an effort to create dense bulk Si-BC samples. The prepared samples were characterized by X-ray diffraction and Raman spectroscopy to determine their stoichiometries and possible changes in the lattice structure. Energy Dispersive X-ray Spectroscopy was used in conjunction with a Scanning Electron Microscope to map elemental distribution. Vickers micro-indentations helped us investigate the effect of Si inclusions on fracture toughness and Raman spectroscopy was used to probe those indents from an amorphization aspect in the Si-BC system. Optimization of the sintering protocol will lead to better understanding of the limits of Si doping in the BC system, and a dense amorphization resistant boron carbide with a possible path to scale up to industry level production.

(ICACC-S4-P023-2018) Reactive Hot-Pressing of B_6O: Effect of Excess B_2O_3 on Phases, Microstructure, and Properties

H. E. Payne*[1]; K. D. Behler[2]; T. Shoulders[3]; L. R. Vargas-Gonzalez[4]; J. LaSalvia[4]

1. U.S. Army Reseach Lab, College Qualified Leaders (CQL) and The Pennsylvania State University, Ceramics and Transparent Materials Branch, USA
2. U.S. Army Research Lab and SURVICE Engineering, Ceramics and Transparent Materials Branch, USA
3. U.S. Army Research Lab and ORISE, Ceramics and Transparent Materials Branch, USA
4. U.S. Army Research Lab, Ceramics and Transparent Materials Branch, USA

Boron suboxide (B_6O) exhibits potential for ceramic armor due to its intrinsic properties (low density, high hardness). However, high costs for boron (B) and processability are challenges. In this study, reactive hot-pressing of B_6O was investigated as a lower cost alternative compared to the conventional approach (separate powder synthesis, processing, and densification steps). Reactive hot-pressing is based on the common reaction for B_6O: $16B + B_2O_3 \rightarrow 3B_6O$. Due to volatilization of B_2O_3 at higher temperatures, excess B_2O_3 might be necessary to decrease oxygen (O) deficiency. Amorphous B and 0–12 wt.% excess B_2O_3 powder mixtures were hot-pressed at 1850°C for 3hr and 54MPa. A 1hr temperature hold at 1400°C was performed to convert the reactants to B_6O. The resulting samples were characterized for densities, phases, microstructures, and 2 kgf Knoop hardness values. Excess B_2O_3 had a strong effect on phases, microstructures, and properties. Experimental procedures and results are presented.

(ICACC-S4-P024-2018) Computer modeling of projectile's penetration into discrete armor panel

E. Kartuzov[1]; V. Kartuzov*[1]; O. Mikhaylov[1]

1. Frantsevich Institute for Problems in Materials Science NAS of Ukraine, Ukraine

This effort brings the results of numerical experiment of projectile's penetration into hybrid armor panel with absorbing layer made from various discrete elements. Modelling was performed at two scales. Deformation of each discrete element was considered in terms of continuum approach, hybrid armour panel was represented by a number of discrete elements with given law of interaction. Numerical experiment consisted in variation of micro porosity of Al damping particles and variation of projectile's shape and velocity as well as a variation of material and shape of discrete reinforcing elements.

(ICACC-S4-P026-2018) Molecular-dynamic simulation of shock wave propagation in B13-C2 ceramics

E. Kartuzov*[1]; V. Kartuzov[1]

1. Frantsevich Institute for Problems in Materials Science NAS of Ukraine, Ukraine

This effort is to consider the properties of shock waves of high intensity in B13-C2 boron carbide. The study is based on molecular dynamics computer simulation with the use of the appropriate Tersoff interatomic potential. The objective is to elucidate the impact of presence of defects and porosity on cracks formation as a result of shock waves depending on loads intensity. Samples of various geometries and direction of wave propagation as to crystal orientation with porosity in terms of 1-15% were considered. The dependences of threshold loads causing samples fracture on porosity degree were obtained. Simulation results are used to explain microscopic fracture mechanisms of cracks formation in boron carbide ceramics exposed to shock loads.

(ICACC-S4-P027-2018) Observations of metastable explosion and wurtzite phases of boron nitride formed by emulsion detonation synthesis

M. Ornek*[1]; C. Hwang[1]; K. Xie[3]; S. Da Silva[2]; J. Calado[2]; M. K. Reddy[3]; A. Burgess[4]; V. Domnich[1]; S. Miller[5]; K. Hemker[3]; R. A. Haber[1]

1. Rutgers University, Material Science and Engineering, USA
2. Innovnano, Materiais Avançados, Portugal
3. Johns Hopkins University, Mechanical Engineering, USA
4. SprayWerks Technologies Inc., Canada
5. H&M Analytical Services, Inc., USA

Emulsion detonation synthesis (EDS) is a novel and recently developed process for the synthesis of nano-sized ceramics based on detonation of two water-in-oil emulsions. This process can provide high pressure and high temperature along with fast cooling, thus creating ideal environment for metastable phase formation. Here we report the application of this process for the first time on hexagonal boron nitride (h-BN). X-ray diffraction (XRD) and Fourier transform infrared spectroscopy (FTIR) and transmission electron microscopy(TEM) with selected area electron diffraction(SAED) revealed the formation of metastable explosion BN (e-BN) and metastable wurtzite BN (w-BN) phases at calculated pressures of 5 and 7 GPa, respectively. It was observed that e-BN phases are 10-20 nm in size and embedded in h-BN matrix. Further, scanning transmission electron microscopy (STEM) coupled with electron energy loss spectroscopy (EELS) revealed the presence of w-BN phase with varying grain sizes, from nano to micron scale, and either embedded in or coexists with h-BN. These observations demonstrated EDS as a promising pathway for the synthesis of e-BN, w-BN and other metastable phases.

(ICACC-S4-P028-2018) A 1-D Analytical Model for Hypervelocity Penetration of Thick Ceramic Targets

S. Bavdekar*[1]; G. Subhash[1]

1. University of Florida, Mechanical & Aerospace Engineering, USA

The Walker-Anderson model captures the one-dimensional transient and steady-state response of thick targets to penetration. This analytical model is based on the solution of the time-dependent, cylindrically-symmetric Eulerian momentum equation along the axis of penetration and, in the case of ceramics, utilizes a Drucker-Prager yield surface with a maximum flow stress cut-off to characterize the constitutive response. A modified version of this model is proposed by utilizing the extended Mohr-Coulomb constitutive model, which captures the exponential pressure-shear response of ceramics in a normalized universal strength model. The extended M-C model is preferred due to its ability to capture the experimentally observed non-linear response of confined comminuted ceramics at ultrahigh pressure loads using a single set of universal parameters, applicable to all ceramics. By incorporating this model, the requirement on (often expensive) experimental data is reduced, which is especially beneficial in the research of new materials. The target response is assumed to occur in a hemispherical region containing nested comminuted, cracked and elastic zones of deformation. An expanding cavity model, recently developed by the authors, is utilized to estimate the extent of these zones within the target. The model results are compared to numerical simulations and experimental penetration data on brittle materials.

(ICACC-S4-P132-2018) Reliable Measurement of Fracture Toughness of Armour Ceramics at the Microstructural Scale

J. Jiang*[1]; S. Falco[1]; N. Petrinic[1]; R. I. Todd[1]

1. University of Oxford, United Kingdom

Toughness measurements of individual microstructural components of armour ceramics are indispensable to a full understanding of their mechanical properties and impact behaviour. Such measurements can be made by testing microcantilever beams manufactured using focused ion beams (FIB) and including a notch at the chosen microstructural feature. However, residual stress caused by Ga-ion implantation at the notch root is thought to influence the results and therefore causes inaccurate toughness values. The present study attempts to solve this problem by two methods: (a) clarifying the extent of the effect of the implantation stress (IS), and (b) enabling stable crack growth prior to failure. In the first, the IS has been quantitatively characterised in alumina, silicon carbide and silicon by measuring beam curvatures in a dual-beam FIB. The effects of FIB settings and crystal orientations are investigated. These measurements enable toughness values to be corrected for the IS. Alternatively, stable crack growth could allow the fracture toughness to be measured out of the implanted region. The feasibility was investigated using a range of notch types and specimen geometries, based on analytical analysis as well as numerical simulations. The implications of the results for microstructural toughness measurements are described.

(ICACC-S6-P030-2018) A two-step synthesis process of thermoelectric alloys for the separate control of carrier density and mobility

S. Lim[1]; S. Baek[1]; C. Park[2]; Y. Lee[3]; J. Kim*[1]

1. Korea Institute of Science and Technology, Republic of Korea
2. Seoul National University, Republic of Korea
3. Korea Research Institute of Chemical Technology, Thin Film Materials Research Group, Republic of Korea

It is challenging to improve the thermoelectric figure-of-merit as its constituent terms such as Seebeck coefficient, electrical conductivity, and thermal conductivity, are inter-related in the way that the enhancement of one term leads to the degradation of others. Therefore, it is highly desirable to design a new synthesis process that allows us to independently control these terms. Here, we report a simple, two-step process combining spark plasma sintering (SPS) and post-annealing (PA) to separately control the carrier density and mobility in the p-type $(Bi_{0.2}Sb_{0.8})_2Te_3$. High-temperature SPS enables enhancing the carrier mobility by reducing scattering sites such as grain boundaries. Then, the following PA at a lower temperature allows tailoring the carrier density without the degradation of mobility. Beyond bismuth telluride-based, room-temperature thermoelectric materials, we believe that our result will provide an insight for the performance enhancement of other thermoelectric materials such as oxide and skutterudite.

(ICACC-S6-P031-2018) Fabrication and thermoelectric characterization of Bi_2Te_3 and Sb_2Te_3 films grown on graphene substrate by plasma-enhanced chemical vapor deposition

Y. Lee*[1]; J. Kim[2]

1. Korea Research Institute of Chemical Technology, Advanced Materials Division, Republic of Korea
2. Korea Institute of Science and Technology, Republic of Korea

A study on the substrate effect on the thermoelectric properties of Bi_2Te_3 and Sb_2Te_3 thin films grown by plasma-enhanced chemical vapor deposition (PECVD) was performed. Graphene substrates having small lattice mismatch with BT and ST were used for the preparation of highly oriented BT and ST thin films. Carrier mobility of the epitaxial BT and ST films grown on the graphene substrates increased as the deposition temperature increased, which was not observed in that of SiO_2/Si substrates. Seebeck coefficients of the as-grown BT and ST films were observed to be maintained even though carrier concentration increased in the epitaxial BT and ST films on graphene substrate. Although Seebeck coefficient was not improved, power factor of the as-grown BT and ST films was considerably enhanced due to the increase of electrical conductivity resulting from the high carrier mobility and moderate carrier concentration in the epitaxial BT and ST.

(ICACC-S6-P133-2018) Sb nano particles in Silicon Oxycarbide matrix as an Anode materials for Sodium-ion Batteries

W. Choi*[1]; Y. Lee[1]; H. Kim[1]; H. Lim[1]; J. Park[1]; Y. Kwon[1]; K. Lee[2]; D. Byun[2]

1. Korea Institute of Science and Technology, Center for Energy Convergence, Republic of Korea
2. Korea University, Republic of Korea

Sodium-ion batteries (SIBs) have recently attracted significant attention for electrochemical energy storage and conversion owing to the environmental friendliness, natural abundance and low cost of sodium compared to lithium. Among the various materials for SIBs, antimony (Sb) has been considered to be a promising candidate for anodes due to its high theoretical capacity. Despite the high gravimetric capacity advantage, these alloy-based anodes suffer from unsatisfactory electrochemical performance originating from inherent huge volume changes and sluggish kinetics during repeated sodiation/desodiation. Herein, we synthesize the Sb-embedded SiOC composites by pyrolysis of antimony acetate with SiOC precursor to improve the sodium storage performance of Sb as anode for SIBs. The structural and morphological characterizations reveal the Sb nanoparticles are homogeneously embedded into the amorphous SiOC matrix. The Sb-embedded SiOC composite electrodes exhibits high sodium storage capacity and maintain the stable cycling performance. Further analyses indicate that buffering matrix is effective to release the mechanical stress during sodiation/desodiation and suppress the agglomeration of Sb particles. This facile structural design and synthetic method can potentially be extended to other sodium-alloy materials for high capacity-SIBs.

(ICACC-S8-P032-2018) Complex shaped SiC-diamond components for thermal management, high energy lasers, and solar thermal applications

A. McCormick*[1]; P. Karandikar[1]; M. Aghajanian[1]

1. M Cubed Technology, Inc., R&D, USA

As the demand for higher power electronic devices increases, so does the need for dissipating the heat produced. To achieve this goal, chips are mounted on heat sinks. These heat sinks are made from highly thermally conductivity (K) metals such as Cu and Al. Unfortunately, these metals have high CTE. As a result, differential-CTE induced stress is generated resulting in failures after fewer than desired heating –cooling cycles. To overcome this problem thermally conductive adhesives are used to mitigate the residual stress, but they reduce heat dissipation efficiency. Other lower CTE materials such as ceramics (e.g. AlN, BeO) and composites (e.g. –Cu-W, Al-SiC, Cu- diamond) have been evaluated. These, have lower K or high cost. Reaction bonded SiC -diamond composites provide extremely high K and low CTE in addition to low cost due to net-shape manufacturing. This work will present systematic properties of reaction bonded SiC-diamond composites with varying diamond content (15% to 70% by weight). Theoretical analysis will be conducted to predict the thermal properties of the SiC-diamond. Ability to produce complex-shaped SiC-diamond components will be demonstrated. With their unique thermal properties and high temperature capability, complex SiC-diamond components can be enabling for high energy lasers mirrors, solar thermal energy, photonics, etc.

(ICACC-S8-P033-2018) Boron – challenges to a fascinating element

T. Schmidt*[1]; S. E. Vogel[2]

1. H.C. Starck Surface Technology and Ceramic Powders GmbH, Application Engineering, Germany
2. H.C. Starck North American Trading LLC, USA

Elemental Boron is a very fascinating element due to its unique structure and polymorphism as well as its complex chemistry. It occurs in several allotropic forms, most of them are not easy to access. The preparation of pure boron is demanding and requires a well-designed production process because of its increased reactivity at high temperatures, inevitable for the reduction from its compounds. Large volumes of amorphous boron are used in pyrotechnics (airbag igniters, flares, propellants), explosives, and advanced ceramics, whereas crystalline boron qualifies for permanent magnets (Nd-Fe-B), refractory borides, targets, or neutron shielding in nuclear industry. An increasing demand for purer and finer qualities is the challenging task for today's manufacturers. H.C. Starck – Germany, a renowned boron producer for more than 60 years has accepted that challenge and took the opportunity to improve their broad portfolio of amorphous and crystalline boron. Based on a state-of-the-art production facility, progress has been made to further increase the lot to lot consistency and offer a more consistent performance. The distinct grades revealed purity improvements due to a combination of various preparation and purification techniques available through our versatile technology platform. We will discuss the results of chemical and physical powder characterization, with an emphasis on the improvements achieved and their potential benefits.

(ICACC-S8-P034-2018) Consolidation in micro-nano technologies modeled by difference-differential transformation method

V. Mitic*[1]; Z. Vosika[2]; L. Kocic[2]; G. Lazovic[3]; V. Paunovic[2]

1. Serbian Academy of Sciences, Institute of Technical Sciences, Serbia
2. Faculty of Electronic Engineering, Serbia
3. Faculty of Mechanical Engineering, Serbia

Time scale calculus (TSC) is a relatively new mathematical method, especially, in the discreetly-continuous case, is widely utilized for description of various types of systems and processes: 1) kinetics: from classical mechanics (including classical time crystals), to the biochemistry kinetics (tumor genesis); 2) quantum mechanics (discrete and continuous Schrodinger equation) 3) open quantum systems (photonic and phononic crystals); 4) non-equilibrium systems in general (professor Norman Yao said: "This is a new phase of matter, period, but it is also really cool because it is one of the first examples of non-equilibrium matter. For the last half-century, we have been exploring equilibrium matter, like metals and insulators. We are just now starting to explore a whole new landscape of non-equilibrium matter."); 5) topological states (the quantum Hall efect) etc. Generally speaking, processes in those systems are modeled with discrete time scale. Of Particular interest is the fractal and similar scales. All these phenomena are the basis for the development of new technologies. The difference-differential transformation method, as a dominantly numeric method, could be used for solving corresponding discrete or continuous or fractal equations. It is a useful mathematical tool for examining the properties of previous phenomena (microphysics and macroscopic quantum systems).

(ICACC-S8-P035-2018) Thermal expansion coefficient controlled Cu-$ZrW_{2-x}Mo_xO_8$ cermet material prepared using spark plasma sintering method

H. Wei*[1]; R. Inoue[1]; A. Aimi[2]; K. Fujimoto[2]; K. Nishio[1]

1. Tokyo University of Science, Department of Materials Science and Technology, Japan
2. Tokyo University of Science, Department of Pure and Applied Chemistry, Japan

The coefficients of thermal expansion (CTE) of cermet materials differ. This difference in the CTE of these materials causes the destruction of modules. One solution is to control the CTE. In this study, we fabricated cermet materials consisting of copper and ZrW_2O_8 and $ZrMo_2O_8$ with a negative thermal expansion using a spark plasma sintering method (SPS method), and we controlled its coefficient of thermal expansion. Cubic $ZrW_{2-x}Mo_xO_8$ (0%x%1) shows isotropic negative thermal expansion (NTE) behavior not only over a wide temperature range but also without a phase transition temperature of $ZrW_{2-x}Mo_xO_8$ (0.7%x%1) above room temperature. Volume fraction change enabled Cu- $ZrW_{2-x}Mo_xO_8$ cermet materials to be successfully prepared at 673 K–773 K using SPS process. Also, the CTEs of the samples depended on containing the amount of $ZrW_{2-x}Mo_xO_8$ in the sample; the CTE increased from $-2.5\times10^{-6}[K^{-1}]$ (cubic $ZrWMoO_8$ 100 vol%) to $17.78\times10^{-6}[K^{-1}]$ (Cu 100 vol%). The results showed that the CTE of the sample could be changed from negative to positive. Moreover, the composite material has the potential of zero thermal expansion over a wide temperature range.

(ICACC-S8-P036-2018) New Damage Sensing Method of CNT Coated Glass Fiber or Carbon Fiber/PP-PA Composites via 2D and 3D Electrical Resistance Mapping

J. Park*[1]; J. Kim[1]; P. Shin[1]; Y. Baek[1]; H. Park[1]; L. K. DeVries[2]

1. Gyeongsang Natl University, Materials Eng. & Convergence Technology, Republic of Korea
2. The University of Utah, Mechanical Engineering, USA

Evaluation of sensing for electrical conductive composites has been implemented using electrical conductive nano materials such as graphene, CNT and carbon fiber. Electrical resistance (ER) measurement for nondestructive evaluation (NDE) was developed using self-sensing composites because method of damage sensing and crack prediction of composites under external load is possible to use at aerospace, heavy industry, and automobile. In this research, diverse damage sensing from mechanical impact and thermal aging for electrical conductive composites was investigated by using ER method. To have the test, electrical conductive materials such as CNT coated GF and carbon fiber and matrixes such as epoxy and vinyl ester were used for damage sensing and finding optimum materials for improving the bonding force. 2D and 3D ER mapping was used to sense and predict damages from tensile, compressive, impact and drilling force. The differences in ER from different force were compared to explore their usage for real time monitoring and sensing of damages. Enhance optimum materials and conditions from diverse force were confirmed by ER method. The observation of the fractured surfaces exhibited an acceptable consistency with ER results. Ultimately ER mapping should be useful for evaluating damages in CF/PP-PA composites under various other loading conditions.

(ICACC-S8-P037-2018) Forming limit diagram of vinyl coated metal

J. Yoon*[1]

1. Hanyang University, Department of Mechanical Engineering, Republic of Korea

Vinyl coated metal (VCM) which is sheet metal coated with PET and PVC on galvanized steel sheet has been widely used in home appliances such as washing machines and refrigerators since it guarantees superior appearance and good surface finish. However, there

are severe issues concerning PET film fracture or delamination during the drawing and bending processes. In this research, we're going to provide the forming limit diagrams of base metal and PET film, separately, based on the Nakazima tests to take into consideration of various failure tendencies with respect to deformation modes, which enables analysis of formability prior to manufacturing with VCM metals.

(ICACC-S8-P038-2018) Preparation of polyborosilazane-derived SiBNC ceramic fibers by electron beam irradiation curing

J. Wang*[1]

1. National University of Defense Technology, China

Polymer-derived SiBNC ceramic fibers have attracted increasing attentions due to its high tensile strength and excellent resistance to oxidation at high-temperature. The high-temperature durability of SiBNC ceramics is significantly influenced by Si/B ratios and the synthetic procedures. Single-source synthetic routes can yield homogeneous ceramics, but the Si/B ratio cannot be efficiently adjusted. Herein, we report the SiBNC precursor polyborosilazanes (PBSZs) with different Si/B ratios has been established via a one-pot reaction involving boron trichloride, dichloromethylsilane and hexamethyldisilazane in different molar ratios. After melt-spinning, PBSZ green fibers were obtained and the method of electron beam irradiation was used to crosslink green fibers. The molecular structures evolution from green fibers to cured fibers and the properties of sintered SiBNC fibers have been investigated in detail. The cured fibers performed a high ceramic yield of 80.4 wt%. After pyrolysis at 1500 °C, SiBNC ceramic fibers were acquired, which exhibited a good flexibility with diameter of 12 μm and tensile strength of 1.22GPa. The obtained fibers could keep amorphous up to 1700 °C and showed no mass loss at this temperature.

(ICACC-S8-P039-2018) Si-O-C compact with reduced carbon content obtained by Spark Plasma Sintering

R. Hanatani*[1]; M. Narisawa[1]; H. Inoue[1]; H. Segawa[2]; T. Nishimura[2]

1. Osaka Prefecture University College, Japan
2. National Institute for Materials Science (NIMS), Japan

Si-O-C ceramics derived from Si-O-C(H) powder by hot press at 1600°C was reported to have high creep resistance at 1100°C to 1300°C which is 2 order of magnitudes higher than that of silica. In our research, we focused on the SPS (Spark Plasma Sintering) method which popular to produce a dense sintered compact in a short time by pressurization and rapid temperature rising. We tried to prepare sintered compact from Si-O-C(H) powder by this SPS method at the lower temperatures below 1600°C. The temperatures used for SPS were 1300 - 1600°C with the pressure of 60 MPa. The bulk density of them showed rapid increase beyond 1350°C as temperature increased and the bulk density reached 2.36 g/cm^3, which is almost the same as that of the material Si-O-C(-H) particle. Cross sections of compacts were smooth at 1400°C. There were no traces of the original particle. So we thought dense sintered compacts were obtained beyond 1350°C which was consistent with the reported glass transition temperature of Si-O-C ceramics. Thermal conductivity of the synthesized compacts was ca. 4.5 W/m/K and Young's modulus was about ca. 90 GPa. These value are bit larger than the values of silica and are much smaller than those of silicon carbide.

(ICACC-S11-P040-2018) Development of geopolymer composites reinforced with fiber felts

A. Conte*[1]; G. Passante[2]; P. Colombo[1]

1. University of Padova, Industrial Engineering, Italy
2. Trucker Subforniture s.r.l., Italy

Geopolymers are synthetic inorganic alumino-silicate materials generally formed by reaction of an aluminosilicate with an alkali (Na,K) silicate solution. The reaction occurs at room temperature, so geopolymer can be considered as a type of bi-component inorganic resin. Considering their inorganic structure, geopolymer composites have better thermal properties than organic resins, which typically decompose by oxidation starting from ~400°C. In collaboration with Trucker Subforniture s.r.l., geopolymer based composites reinforced with different kinds of felt, have been developed as alternative to GFRP and CFRP, for applications where a high thermal resistance is required. Different kind of fibers were considered as reinforcement; using felts based on recycled fibers it is possible to keep the cost lower than that of virgin fibers, in particular for carbon and basalt fibers. Geopolymer composites were developed using RTM and infusion technology. In order to have a good infiltration of the felts, the rheology of a potassium based geopolymer resin was optimized in terms of water and alkalinity. Density, mechanical properties and thermal stability of the different composites were tested.

(ICACC-S11-P041-2018) Mophology control of hydrothermally-grown zinc oxide nanowires on aramid fabrics

H. Hwang*[1]

1. University of Michigan, Aerospace Engineering Department, USA

Zinc oxide (ZnO) nanowires have received continuous research interests because of thier piezoelectricity and ability to be synthesized in low temperatures. However, there was not much research has been done on methods to control of the ZnO's morphology in most common nitrate-based hydrothermal growth solutions. In this work, a method to induce different morphologies of the ZnO nanowires synthesized on the aramid fabrics is introduced. Utilizing size control of ZnO quantum dots which performs as a nucleation sites for the growth of the nanowires and a preferential adhesion of a metal ion impurity on (0002) surface of ZnO nanowires, drastic changes in shape of the nanowires has been observed, recording change of aspect ratio of 20. Interestingly, despite of relatively strong basic environment owing to use of ammonium hydroxide in the growth solution, the testing results on fibers and fabrics indicate that the synthesis procedure does not give any negative influence on tensile strength or elastic modulus of the aramid fabrics.

(ICACC-S11-P042-2018) The properties of nanocomposite Ti-Al-V-N coating synthesized by magnetron sputtering process with single composite target

H. Lee*[1]; H. Yoon[1]; G. Bang[1]; K. Moon[1]

1. Korea Institute of Industrial Technology, Republic of Korea

With its progress of machine tools and cutting technology, study of multi-functional materials with high efficiency is becoming increasingly important in terms of productivity, cost reduction and from an environmental point of view. The role of adding third elements have been studied to improve the properties of Ti-Al-N films. In this study, Ti-Al-V based single alloying targets were prepared by powder metallurgy of mechanical alloying and SPS. Ternary Ti-Al-V-N based films were deposited by unbalanced magnetron sputtering method with various alloying targets. During deposition, the substrate temperature was kept constant at 25degree in the mixture of Ar+N_2 atmospheres. The composition of the films was almost the same with that of the target. Their micro-structures and mechanical properties were investigated by XPS, XRD, SEM, nano-indenter, tribometer, corrosion test and etc. Also, the effects of the vanadium on the mechanical and corrosion properties of the Ti-Al-V-N films were studied by changing Ar:N_2 ratio. They showed the highest hardness at the gas ratio of Ar:N_2 being 9:1. It was found that adding vanadium elements results in increasing its hardness by 5~10GPa compared with that of TiAlN films itself. Finally characterization of films was studied by comparing the single nitride films with its graded nitride films.

(ICACC-S11-P043-2018) Zr-base amorphous alloys Correlation between grain size and mechanical, Electrochemical property

H. Lee*[1]; H. Yoon[1]; G. Bang[1]; K. Moon[1]

1. Korea Institute of Industrial Technology, Republic of Korea

As materials with excellent mechanical and chemical properties are required studies on BMG have been carried out. recently, studies have been carried out to improve mechanical properties by crystallizing. [1-6] In this study, Zr-base amorphous alloys Correlation between grain size and mechanical, Electrochemical property was investigated. Zr-base amorphous alloy ingots were fabricated by arc melting and amorphous powders were fabricated by high pressure gas atomization method. Through the DSC and XRD analysis, the amorphous, hyperplastic and crystallization temperatures of the powders were investigated. The sintering temperature and the retention time were controlled to produce a sintered body having a grain size of nm to um, and the mechanical strength and the corrosion test were conducted. Microstructures were observed by optical microscope and scanning electron microscope, and grain size was measured by scanning electron microscope SEM_BSE. The hardness and grain boundary strength were measured using a Vickers hardness tester, Corrosion tests were carried out on the electromagnetically polarized test.

(ICACC-S11-P044-2018) Effect of load and sliding speed on tribological behavior of a semi-carbonized Cu/phenolic-derived semi-metallic friction material

C. Ju*[1]; C. Lee[2]; H. Lin[1]; K. Lee[2]; J. Chern Lin[1]

1. National Cheng-Kung University, Materials Science and Engineering, Taiwan
2. I-Shou University, Materials Science and Engineering, Taiwan

Effects of load and sliding speed on the tribological behavior of a semi-carbonized copper/phenolic resin-derived semi-metallic friction material used for bike brakes are reported. The material used for the study was prepared by dry-mixing appropriate amounts of phenolic resin powder, pure copper fiber, pure copper powder, along with additions of graphite and vermiculite powders, followed by hot-pressing, post-curing and semi-carbonization. The tribological performance of the material was evaluated using a disk-on-disk sliding wear tester at ambient temperature. Experimental results showed that, under the same test conditions (0.11-0.37 MPa; 121-482 RPM), the present semi-carbonized semi-metallic friction material demonstrated significantly higher COF values (0.34-0.51) than its commercial high temperature-sintered metallic counterpart (0.24-0.40). Another primary advantage of the present material over the commercial metallic material is its dramatically reduced noise level during testing. Under the same test conditions, the chance to pick up noticeable noise during testing of the commercial sintered metallic product was about 50%, while the chance to pick up noticeable noise for the present semi-carbonized material was only about 1%. The research is supported by the Ministry of Science and Technology of Taiwan, ROC (MOST 106-2221-E-006-070).

(ICACC-S11-P045-2018) Osteoporotic goat spine implantation study with a newly-developed calcium phosphate/calcium sulfate-based bone void filler

J. Chern Lin*[1]; B. Yang[1]; S. Lan[2]; C. Lin[3]; C. Ju[1]

1. National Cheng Kung University, Materials Science and Engineering, Taiwan
2. National Cheng-Kung University Hospital Dou-Liou Branch, Department of Orthopedics, Taiwan
3. Joy Medical Devices Corp., Taiwan

A synthetic, inorganic, highly osteoconductive and resorbable calcium phosphate/calcium sulfate-based bone substitute material was developed.by a NCKU/JMD joint project. The porous granular product has a fast resorption rate, while the cement product is featured by its non-dispersive behavior in blood/body fluid. Safety and efficacy of both devices were confirmed by a series of chemical/physical characterization and biocompatibility tests. This presentation reports the results of an osteoporotic goat spine implantation study. 2-3 year old ovariectomized goats were treated with Dexamethasone, fed with low calcium diet and bred away from light. The goats of the control group of same age were fed and bred normally. After 18 months, bone voids of vertebral bodies were surgically created in the osteoporotic goats and the aforesaid Ca-based granular and cement products were separately implanted into the surgically-created bone voids. The goats were sacrificed after 23W. Histopathologic examination showed that osteoporosis had been successfully induced by the present treatment. The trabeculae in vertebral body of the implant-filled experimental group were significantly thicker than the unfilled, osteoporotic experiment group and comparable to the healthy control group. The research was funded by Southern Taiwan Medical Device Industry Cluster (BY-02-06-27-105).

(ICACC-S11-P046-2018) Stretchable electrodes based on the carbon/polymer/metal composite for wearable devices

S. Mhin*[1]

1. Korea Institute of Industrial Technology, Heat treatment R&D group, Republic of Korea

Recent technological progress towards the application of the wearable devices leads to the development of flexible electrodes. So far, various technical approaches for the flexible electrodes to be improved mechanical and electrical reliability have been reported. However, transferring the lab-scale processing to industrialization is still limited due to the complex and tedious processing steps for the preparation of flexible electrodes. Herein, we introduce facile engineering steps to prepare the flexible electrodes, which is consisted of carbon/polymer/metal composites for the potential application to wearable devices. Simply, the composites were screen-printed on metal substrates and then, were transferred to the polymer matrix. The prepared composites show the electrical reliability within 15% up to 30% elongation. Also, constant change of the electrical resistance was observed depending on the degree of elongation, which can be further applied as strain sensor.

(ICACC-S14-P047-2018) Photoluminescence of Activator-Doped Glass-Ceramics

H. Masai*[1]; T. Yanagida[2]

1. National Institute of Advanced Industrial Science and Technology (AIST), Department of Materials and Chemistry, Japan
2. Nara Institute of Science and Technology, Japan

We have examined the physical and luminescent properties of oxide glass-ceramics depending on the preparation condition. The crystallization process of oxide glass consists of nucleation and crystal growth, which are directly correlated with the precipitation behavior of crystallites. The spatial distribution of activators in glass and glass-ceramics is also affected by both this crystallization process and the kind of activators. In this paper, we have two activators: Sn^{2+} and Ce^{3+} in glass and glass-ceramics. Although two activators possess the parity-allowed transition, which is affected by the local coordination field, the degree is dependent of the cation. Since crystallization of the glass is affected by each transient state, the evaluation of the structural analysis will be more important in the near future.

(ICACC-S14-P048-2018) Luminescence properties of BaO-TiO_2-SiO_2 glass-ceramics

H. Masai*[1]; T. Yanagida[2]

1. National Institute of Advanced Industrial Science and Technology (AIST), Department of Materials and Chemistry, Japan
2. Nara Institute of Science and Technology, Japan

Glass-ceramic can possess not only the physical properties of both glass and crystalline phases, but also the structural components of both phases. In the case of BaO-TiO_2-SiO_2 glass, fresnoite $Ba_2TiSi_2O_8$,

can be obtained by a heat-treatment. Since $Ba_2TiSi_2O_8$ possesses TiO_5 square pyramidal structure that shows its polarity along the c-axis, it shows various unique properties, such as fluorescence, piezoelectricity, elasticity, and nonlinear optical property. In this study, we focus on the luminescence properties of the BaO-TiO_2-SiO_2 glass and the glass-ceramics. As luminescent properties, not only conventional photoluminescence but also X-ray induced luminescence (XL) such as scintillation and stimulated luminescence were measured. Since the XL inherently contains energy transfer from the host matrix, it is expected that the crystallization affects the XL properties. Several BaO-TiO_2-SiO_2 glasses were prepared by melt-quenching method in an ambient atmosphere. The $40BaO$-$20TiO_2$-$40SiO_2$, which is the stoichiometric chemical composition of $Ba_2TiSi_2O_8$, a large enhancement of X-ray induced scintillation was observed by the heat-treatment of the T_g + 50°C. On the other hand, in the $30BaO$-$15TiO_2$-$55SiO_2$ glass, which showed "surface crystallization behavior", no significant difference was observed after the same heat-treatment. The property change by the crystallization was also observed in the internal quantum yield.

(ICACC-S14-P049-2018) Crystal growth of $Sr_3Zr_2O_7$ by TSFZ method

I. Fukasawa*[1]; M. Nagao[1]; S. Watauchi[1]; I. Tanaka[1]

1. University of Yamanashi, Center for Crystal Science and Technology, Japan

$Sr_3Zr_2O_7$ is expected as a candidate of Hybrid Improper Ferroelectric (HIF) substances. $Sr_3Zr_2O_7$ single crystals are necessary to experimentally clarify the ferroelectricity mechanism. However, information for crystal growth of $Sr_3Zr_2O_7$ from the melt is uncertain. In this work, we investigated on the melting behavior of the $Sr_3Zr_2O_7$ phase, and tried to grow $Sr_3Zr_2O_7$ single crystals by the traveling solvent floating zone (TSFZ) method. Melting-solidification experiments were performed by the slow-cooling floating zone (SCFZ) method using an infrared heating furnace. The samples solidified from the melt of the composition in the range of 60~80mol%SrO were analyzed by SEM&EDS. On the basis of the results, we tried to grow crystal of $Sr_3Zr_2O_7$ by the TSFZ method using the solvent of 70mol%SrO. As the result of SCFZ experiments, it was found that the $Sr_3Zr_2O_7$ phase melts incongruently to $SrZrO_3$ phase and a liquid phase. Also the result suggests that the $Sr_3Zr_2O_7$ phase coexists with a liquid near 70mol%SrO. In crystal growth of $Sr_3Zr_2O_7$ by TSFZ method, we were confronted with some problems such as evaporation of strontium component from the molten zone and stability of the molten zone. To solve these problems, the growth conditions such as the amount of the solvent and heating technique were optimized. We will discuss on the crystal growth in details at the presentation.

(ICACC-S14-P050-2018) Investigating the Mechanical and Piezoelectric Properties of Combinatorially Deposited $Al_{1-x-y}Sc_xB_yN$ Thin Films

Y. Chen*[1]; K. Talley[1]; S. Manna[1]; C. Ciobanu[1]; C. Packard[1]; G. L. Brennecka[1]

1. Colorado School of Mines, USA

The aluminum-scandium nitride material system has been the attention of significant focus for its promise of improved performance in wireless bandpass filter devices. Limitations to the aluminum-scandium nitride systems adoption include the reduction in material stiffness, reduction in film orientation, and the cost of scandium metal. This study investigates the addition of boron to the alloy system as to improve each of these limitations. Boron nitride is known as a very hard material and is hypothesized to improve the mechanical properties. Boron is smaller in size than aluminum and should compensate the strain induced by the larger scandium atoms, improving the crystal quality. Boron is also cheaper to produce, which can lower the cost to produce these films in micro electromechanical systems. Thin film combinatorial synthesis and characterization was used to investigate the $Al_{1-x-y}Sc_xB_yN$ system. Here we present the calculated structural, piezoelectric, mechanical, and electrical properties and the dependence on the composition of the material, along with initial observations from experimental synthesis.

(ICACC-S14-P051-2018) Scintillation detector properties of undoped and Eu-doped SrI_2 crystals

T. Yanagida*[1]; M. Koshimizu[2]; G. Okada[1]; T. Kojima[3]; J. Osada[3]; N. Kawano[1]; N. Kawaguchi[1]

1. Nara Institute of Science and Technology, Japan
2. Tohoku University, Japan
3. Oxide Corp., Japan

SrI_2 has been known to have excellent scintillation properties characterized by the high light yield and energy resolution. In this work, undoped and Eu 3% doped SrI_2 crystals were synthesized by the vertical Bridgeman method. The radioluminescence (RL) of Eu-doped one exhibited an intense emission peak at 430 nm while the spectral features of the undoped one were complicated. In order to discuss the emission origin of the undoped one, the temperature dependence of the RL was investigated from 10 to 300 K. In addition, by using several gamma-ray sources, pulse height spectra were investigated as a scintillation detector property.

(ICACC-S14-P052-2018) Growth and scintillation responses of $EuAlO_3$ crystal

T. Yanagida*[1]; D. Nakauchi[1]; G. Okada[1]; N. Kawano[1]; N. Kawaguchi[1]

1. Nara Institute of Science and Technology, Japan

In this study, Eu 1% doped $YAlO_3$ (Eu:YAP), $Eu_{0.5}Y_{0.5}AlO_3$ (EYAP) and $EuAlO_3$ (EAP) crystals were prepared by the Floating zone method, and optical and scintillation properties of them were studied. After the crystal growth, we conducted XRD analysis, and confirmed that any impurity phases were included. When we observe photoluminescence (PL) and radioluminescence (RL) spectra, some emission bands due to the perovskite host, divalent and trivalent Eu ions were observed. The PL and scintillation decay time profiles consisted of several exponential decay components. The fast (ns) component group was possibly due to host emission, and especially Eu:YAP demonstrated a very fast PL decay time of 16 ns. In addition, we investigated thermally simulated luminescence properties of them, and Eu:YAP showed the most intense TSL response with a major glow peak at highest temperature at 257 °C, and the dose response functions showed a good linearity (R^2>0.99) over the range 0.1 mGy to 100 mGy. ->

(ICACC-S14-P053-2018) Synthesis of Transition Metal Doped Alumina and Dopant Impact on Phase Transformation

A. L. Fry*[2]; N. Ku[2]; C. A. Moorehead[3]; V. L. Blair[1]; R. E. Brennan[1]

1. U.S. Army Research Laboratory, USA
2. U.S. Army Research Laboratory, ORISE, USA
3. University of Washington, USA

While extensive research has focused on property characterization and manipulation of alumina (Al_2O_3), the Army Research Laboratory has pioneered work on achieving alignment of rare earth doped alumina (RE: Al_2O_3) under varying applied magnetic fields. As a next step, research has shifted to synthesis and characterization of transition metal doped alumina (TM: Al_2O_3) in order to obtain similar responses at a lower cost. TM: Al_2O_3 powders were synthesized using a precipitation process, followed by calcination and characterization. Calcination experiments were conducted to develop time-temperature-transition (TTT) diagrams for the synthesized materials to investigate dopant impact on phase transition parameters, magnetization, and powder morphology. While the transformation temperature of theta to alpha alumina changed significantly when a larger cation was placed within the lattice, it was hypothesized that the addition of a dopant with a similar size to aluminum would result in a less drastic shift in temperature.

(ICACC-S14-P054-2018) The role of microstructure in dichroic properties of the Lycurgus cup glass

A. Drozdov*[1]; M. Andreev[1]

1. Lomonosov Moscow State University, Chemistry, Russian Federation

Lycurgus cup (LC) is is a roman diatreta cut from a blank of typical NCS glass. The glass is dichroic, appearing green in reflected daylight and red in transmitted light. The dicroism of the LC glass is usually derived from the presence of mixed Ag-Au nanoparticles (AuAgNPs). To study the optical properties of LC the glass was prepared starting from the batch calculated using the data published. The glass was molten at 1500°C, poured and quenched with further annealing. The material obtained is a transparent green glass without visible dichroism and phase separation. The colour is caused by the Fe(+2) and Fe(+3) ions in its composition (total Fe 1,14%, ICP MS). During striking the glass becomes dichroic and semiopaque due to the secondary phase separation. The resulted multiphase structure consists of discrete inclusions of glass 1 in a continuous silicate matrix of glass 2. The heat-treatment results in devitrification of a glass 1 that crystallizes giving α-quartz and α-cristobalite microcrystals (confirmed XRD) that strengthen the material. Although iron ions don't enter the crystal phases, they from an important role in phase separation. This fact proves the importance of iron as a glass micro component. The red colour of LC glass in transmitted light is due to the light absorption of AuAgNPs while the green colour in reflected light is caused by scattering on glass 2 droplets and microcrystals.

(ICACC-S14-P055-2018) Development of Yttria Nanopowders for the Photoluminescence Applications as PTC material

S. Kumar*[1]; U. Batra[1]; J. Sharma[1]

1. PEC University of Technology, Materials & Metallurgical Engineering, India

Light scattering due to birefringence has prevented the use of polycrystalline ceramics with anisotropic optical properties in applications such as laser gain media. However, continued technological advancements in ceramic processing technology have made it possible to tailor the microstructural, mechanical and optical properties of different transparent medias. More recently, interest has focused on the development of high optical polycrystalline transparent ceramics (PTC) materials. The PTC are being produced through wet chemical synthesis routes as well as solid state routes. The wet route processes are capable to counter the challenges of purity and chemical stability requirements to achieve high-purity final products. The present work deals with the preparation of Yttria (Y_2O_3) nano-powders by using Ammonium Hydrogen Carbonate (AHC) as a precipitant. The powder characterization of Y_2O_3 nano-powders were examined by using various techniques such as TGA/DSC, XRD, FESEM, EDX, and TEM. It is found that the final phases of the precursors and morphologies were evidently influenced by the molar ratio of (AHC)/(Y^{3+}). An important theme of the work that Y_2O_3 nanopowders produced exhibit morphologies either like needles, rods or spherical. Results obtained in this work theme can contribute to the controllable synthesis of Y_2O_3 nano-powders for advanced transparent ceramics applications.

(ICACC-S15-P056-2018) Polymer-derived ceramic/graphene oxide 3D structures

J. Moyano-Subires*[1]; M. I. Osendi[1]; P. Miranzo[1]; M. Belmonte[1]

1. Institute of Ceramics and Glass, CSIC, Ceramics, Spain

3D printing methods allow the development of complex computer-designed geometries by sequential addition of material. These methods are attractive for building tridimensional stable structures of basically 2D materials, in particular graphene-based structures. Starting from graphene oxide sheets self-supported spanned structures were constructed by a computer controlled filament deposition method. GO water based inks (about 5 wt. % solids content) with a shear thinning rheology provided by the addition of polyelectrolytes were employed. These 3D GO structures are used as a platform for making hybrid composites by infiltrating with a preceramic polymer. After several heat treatment steps structured GO /ceramic composites were achieved. These composites are electrically conductive and, in addition, the ceramic hybridization allows increasing the mechanical resistance up to a 750% with respect to the initial GO structure and the thermal stability up to 1000ºC, thus obtaining structures highly conductive and stable at very high temperatures.

(ICACC-S17-P057-2018) Melting gel for encapsulation applications towards high UV and thermal-stable with low processing temperature

J. Wang*[1]

1. NSYSU, Taiwan

The melting gels are mainly based upon a mixture of low-molecular weight of methyltriethoxysilane(MTES) and dimethyldiethoxysilane(DMDES). A very unique rheology property of the MTES-DMDES gel is rigid at room temperature, flow at temperatures above 110°C, but can become rigid at temperatures above 110°C after processing at temperatures higher than the consolidation temperature about 160°C. As an example, encapsulation application of the melting gel in UV-LED is studied. The preliminary results are described below. For silicone, thermal degradation started at approximately 200°C, and the transmittance was 85.5% at 365nm. The transmittance decreased by 55% after thermal aging at 250°C for 72 h, and it decreased further by 2.5%, even at room temperature, under continuous exposure to UV light at 365 nm for 72h. By contrast, for the MTES-DMDES-gel, thermal degradation started at approximately 300°C, and the transmittance was 90% at 365 nm. The transmittance decreased negligibly after thermal aging at 250°C for 72 h and it did not decrease further even at 75°C under continuous exposure to UV light at 365 nm for 72 h. Hence, the MTES-DMDES gel clearly reveals a better encapsulation material. Furthermore, the latest result of exploiting MTES-DMDES-based melting gel composition effect on higher UV and thermal-stable with lower processing temperature is further discussed.

(ICACC-GYIF-P058-2018) ZrO_2- Y_2O_3 Phase Diagram and Properties Relevant to Ceramic Dental Crown

S. Uwanyuze*[1]; M. K. King[1]; M. Mahapatra[1]

1. University of Alabama at Birmingham, Materials Science and Engineering, USA

3 mol% yttria stabilized tetragonal zirconia is commonly considered for ceramic dental crown due to its superior mechanical properties, chemical durability, and translucency. ZrO_2- Y_2O_3 phase diagram is being studied to find the effect of simultaneous presence of tetragonal and cubic polymorphs of zirconia on densification, mechanical properties, and translucency. The implications of the polymorphs on dental crown will be discussed although no quantitative relationship between the amount of different polymorphs and the properties has been observed.

(ICACC-GYIF-P059-2018) Investigations of matrix-platelets interactions during sintering of ice-templated ceramics and relation to macroscopic compressive response

M. Banda*[1]; H. Kang[1]; D. Ghosh[1]

1. Old Dominion University, Mechanical and Aerospace Engineering, USA

Ice-templated ceramics exhibit a macroscopic structural anisotropy, but at microstructural length-scale contain equiaxed grains within the lamella walls and lamellar bridges. Our recent work has revealed that introduction of anisotropic grains (platelets) has significant effects on the structural evolution at multiple length-scales of ice-templated alumina ceramics. Moreover, grain-level modifications can result in marked improvements of macroscopic mechanical response. However, there is a need to thoroughly understand the interactions of fine-grained alumina matrix and anisotropic alumina

platelets, and microstructure evolution during high-temperature sintering. Presence of fine, equiaxed grains can lead to templated grain growth, significant coarsening of the platelets, and generation of microporosity within the lamella walls. In this work, our goal is to understand the progressive interactions of fine-grained alumina matrix and platelets, and microstructural modifications within the lamella walls of ice-templated scaffolds during sintering. Moreover, our aim is to connect the local structural modifications to the macroscopic mechanical response, and identify the parameters that are critical to tailor the properties. The results will further advance the process-structure-property relationships of ice-templated materials.

(ICACC-GYIF-P060-2018) On the design of novel engineered composites for multifunctional applications

A. Bosco[1]; A. Holland[1]; C. Borillo[1]; S. Abualdam*[1]; S. Gupta[1]

1. University of North Dakota, Mechanical Engineering, USA

This poster will report the synthesis and characterization of novel 3D Printed structures and particulate reinforced composites. Microstructure studies by SEM studies will be performed to understand the dispersion of these particulates in the resin matrix and their mechanical behavior. Tensile testings and tribological behavior study by pin-on-disk method will be performed to understand the mechanical and tribological behavior of these solids.

(ICACC-GYIF-P061-2018) Design of Novel Multifunctional Materials for Sustainable Applications

M. Ahmann*[1]; M. Kringstad[1]; M. Platt[1]; A. Miles[1]; S. Gupta[1]

1. University of North Dakota, Mechanical Engineering, USA

Recently, National Academy of Engineering (NAE) has come up with 14 Grand Challenges. Different schools are developing Global Challenge Scholar's Program (GCSP) to incorporate some of the key elements of grand challenge in the educational program. Some of the important elements of the GCSP program are: "(a) A creative learning experience connected to the Grand Challenges such as research or design projects, (b) Authentic experiential learning with clients and mentors that includes interdisciplinary experience in fields such as public policy, business, law, medicine, ethics, and communications, (c) Entrepreneurship and innovation, (d) Global and cross-cultural perspectives, and (e) Development of social consciousness through service-learning ". In this poster, we will present the recent progress in undergraduate (UG) research as an integral component of GCSP in the grand challenge areas of Carbon Sequestration Methods, Water purification/harvesting, and Engineering tools for scientific discovery by developing collaboration with scientists. http://www.engineeringchallenges.org/File.aspx?id=15680&v=c29105cb

(ICACC-GYIF-P062-2018) Effect of ball milling on microstructure of pressureless sintered GNP/3YTZP composite

C. Lopez Pernia*[1]; A. Morales Rodríguez[1]; Á. Gallardo López[1]; R. Poyato Galán[2]

1. Universidad de Sevilla, Física de la Materia Condensada, Spain
2. Instituto de Ciencia de Materiales de Sevilla, Spain

Recently, the addition of carbon allotropes into different ceramic matrices has attracted the interest of the scientific community. The incorporation of nanomaterials, such as graphene nanoplatelets (GNP) or few layer graphene (FLG), results in a reinforcement of the ceramics. Due to the tendency of these carbon nanomaterials to agglomerate, it becomes necessary to develop a process to obtain a good dispersion of the graphene phase in the ceramic matrix. One of the major routes to fabricate graphene based ceramic composites is the wet powder processing, which typically involves ultrasonic agitation and milling. This is usually followed by a pressure sintering technique, such as hot pressing or spark plasma sintering. However, the published studies about the effect of the homogenization method on composites prepared by pressureless sintering(PLS) are scarce. In this work, composites of 3YTZP with different amounts of GNPs were prepared by wet powder processing and pressureless sintered. The homogenization of the GNP/3YTZP mixture in dry state was carried out by planetary ball milling. GNP size in the composite powder after milling was evaluated by laser granulometry. The effects of ball-milling conditions and sintering temperature on densification and microstructure of the composites were discussed.

(ICACC-WW-P064-2018) Effects of Alkaline Earth Metal Substitution on the Surface Acoustic Wave Properties of Fresnoite Glass-Ceramics

F. Dupla*[1]; M. Renoirt[1]; M. Gonon[1]; N. Smagin[2]; M. Duquennoy[2]; G. Martic[3]

1. University of Mons, Department of Materials Science, Belgium
2. University of Valenciennes and Hainaut-Cambrésis, IEMN-DOAE, France
3. INISMA, CRIBC, Belgium

Surface acoustic wave sensors are used to measure different physical properties such as temperature, pressure or humidity. They rely on a piezoelectric substrate and metallic interdigital transducers (IDT). When a sinusoidal signal is applied to the input IDT, it creates surface acoustic waves (SAW) on the substrate that are modified by the environment and then monitored by the output IDT. This technology is widely used below 600 K, but current piezoelectric materials cannot withstand higher temperatures. Preferentially oriented fresnoite crystals $Sr_2TiSi_2O_8$ in a surface crystallized glass-ceramic are non-ferroelectric and show no depolarization over time and at high temperatures. $Sr_2TiSi_2O_8$ glass-ceramic shows a (002) orientation near the surface that tilts into (201) after a few hundred micrometers. In order to obtain more repeatable samples without plane tilt, the strontium is partially substituted with calcium or barium. The influence on orientation is observed by XRD, and SEM is used to observe the microstructural changes. The piezoelectric properties of the Sr-Ba-fresnoite and Sr-Ca-fresnoite are compared to a non-substituted sample. The SAW properties of these different fresnoite glass-ceramics are measured at high temperatures.

(ICACC-WW-P065-2018) Improvement of Piezoelectric Properties of Sr-Fresnoite Glass-Ceramics

M. Renoirt*[1]; F. Dupla[1]; M. Gonon[1]; N. Smagin[2]; M. Duquennoy[2]; G. Martic[3]

1. University of Mons, Department of Materials Science, Belgium
2. University of Valenciennes and Hainaut-Cambrésis, IEMN-DOAE, France
3. CRIBC, INISMA, Belgium

Most of piezoelectric sensors are made of ferroelectric ceramics. For high temperature applications, a problem occurs due to the depolarization of the ceramic at a temperature higher than its Curie temperature. To overcome this problem, pyroelectric non-ferroelectric ceramics can be used for these applications. However, the preferential orientation of the dipole moments needs to be induced during the fabrication process. It is possible through the glass ceramic synthesis process, which consists in crystallizing a parent glass to obtain a textured glass-ceramic. Strontium Fresnoite $Sr_2TiSi_2O_8$ is a non-ferroelectric piezoelectric phase in which the dipole moment of the unit cell is oriented along the [00l] direction to induce a macroscopic polarization of the crystal phase in this direction. In the present research, crystallization within the glass-ceramic was achieved to study its piezoelectric properties and microstructure, by experimenting different crystallization temperatures. It was demonstrated that a thermal treatment at 1123 K leads to a finer microstructure and a better control of crystals orientation during the crystallization step. A tilt from (002) orientation to (201) orientation was also observed during this work.

(ICACC-WW-P066-2018) Optimisation of SiCp/SiCf preforms prior to matrix formation using microwave enhance chemical vapour infiltration

M. Porter*[1]; A. D'Angio[1]; J. Binner[1]; M. Cinibulk[2]; B. Garcias Banos[3]; A. Aktas[4]

1. University of Birmingham, Metallurgy and Materials, United Kingdom
2. Air Force Research Lab, USA
3. Universidad Politecnica de Valencia, DIMAS - ITACA Institute, Spain
4. National Physical Laboratory, United Kingdom

Current work has been dedicated to the preparation of SiC fibre preforms with a supplementary aqueous SiC slurry added prior to microwave processing. This study has been a multi-disciplinary approach building on previous research that concluded slurry could be successfully added, consolidated and dried using vacuum bagging. Important parameters that have been considered including the impregnation process method, the resultant porosity distribution and size, dielectric properties of constituents and the consequential permeability of the preforms for chemical vapour infiltration. The study looked at all five of these characteristics using a combination of scanning electron microscopy, micro computed tomography, modelling and simple porosity calculation to determine the optimal combination of the particle size, solid loading, impregnation method and fibre type. Results showed that the addition of a powder reduced the average pore size by more than 50% and decreased the porosity significantly reducing the amount of time required for CVI to fill the remaining macro porosity to near fully dense. This supplementary pore filling effect combined with the use of microwave energy to prevent premature pore closure has the potential to considerably reduce the processing time of SiC/SiC composites from months to a matter of days.

(ICACC-WW-P067-2018) Extending the lifetime of T-EBC coatings during thermal cycling above 2400°F

J. Deijkers*[1]; H. Wadley[1]

1. University of Virginia, Materials Science & Engineering, USA

Growth of a cristobalite thermally grown oxide in silicon-based EBCs results in mud cracking and eventual delamination of the EBC system due to high thermal stresses. A proposed composite bond coating of silicon and hafnia will be used to transform excessive cristobalite growth upon oxidation into hafnon (hafnium silicate), which is thermally stable and compatible with the currently longest-lasting EBC system (silicon bond coat, ytterbium disilicate (YbDS) top coat). Silicon-hafnia powder mixtures have been consolidated using spark plasma sintering (SPS) to demonstrate the growth of hafnon during oxidation of this system at the minimum temperature of interest at 2400°F (1316°C) in a consolidated pellet. Cristobalite-hafnia powder mixtures have been consolidated using SPS and annealed in air and argon, demonstrating the growth rates of hafnon in different oxygen environments. Silica volatility reduces YbDS to YbMS, forming silicon hydroxide gas. HfO_2 and gadolina-stabilized HfO_2 TBCs are deposited on the YbDS using EB-DVD, growing a columnar microstructure in an attempt to reduce silica volatility. The evolution of the coating structure has been observed during steam cycling up to 750 hours, showing sintering and subsequent cracking of the HfO_2-based TBC. No reaction between the TBC and YbDS coating has been observed. A reduction in TGO growth has been found during the steam cycling of these coatings.

(ICACC-WW-P068-2018) Bulk Nanoporous Silicon Carbide: A Study of Processing Temperature's Effects on Microstructure and Mechanical Properties.

C. Kassner*[1]; H. Wadley[1]

1. University of Virginia, Materials Science and Engineering, USA

A novel class of architectured metamaterials have been fabricated and have potential to be a major advancement in structural, thermal, and chemical material design applications. The advent of these materials will exploit mechanical properties that approach the theoretical strength. This phenomenon can be realized through the reduction of component size. In particular, nanoporous structures are a lightweight alternative to bulk materials. Additionally, due to their mesostructure, they possess unique properties due to their ligament size and high surface area. However, scalability has been an issue for most techniques Nanoporous SiC, with pore diameters of 500-600nm, have been fabricated using a polycarbosilane preceramic polymer, rhoelogical nano β-SiC seed particles, and the Powder Space Holder Method (PSH). The resulting structures were approximately 2cm in length and had a 1cm diameter. The samples were then heat treated at temperatures ranging from 1200-1500°C resulting in apparent densities ranging from 0.95 – 1.15 g/cm^3. The compressive strength was measured and values exceeding 100MPa were achieved. By understanding these processes, we will be able to reduce defect populations on the macro, meso, and nano scale leading to high strength low density ceramics.

Wednesday, January 24, 2018

S1: Mechanical Behavior and Performance of Ceramics & Composites

Oxidation and Fatigue of CMCs

Room: Coquina Salon D

Session Chairs: Marina Ruggles-Wrenn, Air Force Institute of Technology; Brian Donegan, Air Force Institute of Technology; Dietmar Koch, Institute of Structures and Design

8:30 AM

(ICACC-S1-029-2018) Fatigue of Advanced SiC/SiC Ceramic Matrix Composites at Elevated Temperature in Air and in Steam

M. Ruggles-Wrenn*[1]; N. Boucher[1]; C. P. Przybyla[2]

1. Air Force Institute of Technology, USA
2. Air Force Research Lab, USA

High-temperature mechanical properties and tension-tension fatigue of three SiC/SiC ceramic composites are discussed. Effects of steam on high-temperature fatigue are evaluated. The three composites consist of a SiC matrix reinforced with SiC (Hi-Nicalon™) fibers. Composite 1 was processed by chemical vapor infiltration (CVI) of SiC into fiber preforms coated with BN. Composite 2 had an oxidation inhibited matrix consisting of alternating SiC and B_4C layers and was processed by CVI. Fiber preforms were coated with pyrolytic carbon with B_4C overlay. Composite 3 had a melt-infiltrated (MI) matrix consolidated by combining CVI-SiC with SiC particulate slurry and molten Si infiltration. Fiber preforms were coated with BN. Tension-tension fatigue was investigated at 1200°C in air and in steam. Steam significantly degraded the fatigue performance of composites 1 and 3, but had little influence on the fatigue performance of composite 2. Composite microstructure, as well as damage and failure mechanisms were investigated.

8:50 AM

(ICACC-S1-030-2018) Mechanical Characterization of SiC/SiC Ceramic Matrix Composites Under a Unique Combustion Facility

R. Panakarajupally*[1]; M. Kannan[1]; G. N. Morscher[1]

1. University of Akron, Mechanical Engineering, USA

The efficiency of the advanced turbine engines has to be improved to meet the future demands. The turbine efficiency increases with the increase in Turbine Inlet Temperature (TIT). This demand for the higher efficiency pushed the research to develop a new class of lightweight materials which can withstand higher temperatures with the reduced cooling. The conventional Nickel based superalloys which

are currently employed in the hot section parts are operating at their maximum limit. Ceramic Matrix Composites (CMC's) are considered as the primary candidates for the advanced turbine engines. These are light in weight, has higher temperature capability and requires reduced cooling. However, these are prone to oxidation under the harsh combustion environments. A unique experimental facility which simulates to some degree the combined mechanical and combustion environment of the jet engines was developed to test these CMC's. NDE techniques such as acoustic emission and electrical resistance have been incorporated into the facility to monitor damage during testing in addition to temperature monitoring via an infra-red camera. Results of different CMCs subjected to various conditions will be discussed.

9:10 AM

(ICACC-S1-031-2018) Oxidative Degradation of SiC/SiC Composites

K. Kawanishi*[1]; S. Muto[2]; E. B. Callaway[3]; F. W. Zok[3]

1. IHI Inc., USA, Aero Material Technology Department, USA
2. IHI Corporation, Materials Technology Department, Japan
3. University of California, Santa Barbara, USA

SiC/SiC composites are known to exhibit strength degradation at intermediate temperatures (800–900°C), especially in water-laden environments. Here we present results of an experimental study on stress rupture and fatigue response of a 3D SiC/SiC composite at relevant temperatures. Microscopy and fractography are used to probe the degradation mechanisms. Additionally, effects of environmental exposure on interface properties are examined through fiber push-in tests. Effects of test conditions (time, temperature, environment, stress) as well as location relative to external free surfaces on the interfacial sliding stress and debond toughness are studied. The results are integrated in an effort to elucidate the key degradation mechanisms and to identify mitigation strategies.

9:30 AM

(ICACC-S1-032-2018) Strength degradation model of an orthogonal 3-D woven SiC fiber/SiC matrix composite under constant and cyclic tensile loads at elevated temperatures

Y. Ikarashi*[1]; T. Ogasawara[1]; T. Aoki[2]

1. Tokyo University of Agriculture and Technology, Japan
2. Japan Aerospace Exploration Agency, Japan

SiC fiber reinforced SiC matrix composites (SiC/SiC) are expected to be applied to hot section parts of turbo fan engines. For the designing of actual parts, a methodology that enables the estimation of the lifetime of SiC/SiC composites is necessary for various loading conditions. In the present study, monotonic, constant, and cyclic tensile loading tests were conducted at 1100 °C in air to elucidate the factors dominating the rapture behavior of an orthogonal 3-D woven SiC/SiC composite. Rupture strength and lifetime of the SiC/SiC composite were strongly degraded due to the oxidation of the BN interphase and fibers within load-bearing 0° yarns under constant loading tests. It was also observed that the imposition of unloading-reloading cycles during the constant loading tests further reduced the lifetime of the composite. Microstructural observations and single fiber push-out tests before and after the tests revealed that the interfacial shear stress at BN interphase degraded significantly with the number of unloading-reloading cycles, probably due to the wear of the interphase. A model was proposed to predict the lifetime of the composite under various tensile loading conditions based on the estimated crack growth rate within load-bearing 0° yarns and the degradation of interfacial shear stress.

10:10 AM

(ICACC-S1-033-2018) Evaluation of CMC Failure Mechanisms in Rotating Detonation Engines

D. King*[1]; G. Wilks[2]; T. A. Parthasarathy[1]; M. Cinibulk[2]; R. Rao[1]; B. Maruyama[2]; C. Stevens[3]

1. UES, Inc., USA
2. Air Force Research Lab, USA
3. ISSI, Inc., USA

Pressure gain combustion engines, such as rotating detonation engines (RDEs), offer improved thermodynamic efficiency compared to constant pressure combustion engines; however in a RDE, materials are subjected to intense thermal and acoustic loads. Such conditions make ceramic matrix composites (CMCs) prime candidates for insertion into air cooled RDEs, where metallic components would require active cooling schemes. Modeling of the thermal effects predict localized thermal stresses in the region close to the site of detonation that can result in interlaminar failure of 2D CMCs. Based on these analyses, carbon fiber based CMCs were evaluated as potential RDE materials, where their low CTE and elastic modulus limit the magnitude of accumulated thermal stresses during RDE operation. Nevertheless, prolonged RDE operation resulted in CMC failure. Light optical microscopy and X-ray CT techniques were utilized for three-dimensional reconstruction and analysis of CMC failure. Laser heating experiments, combined with Raman spectroscopy measurements were also utilized to determine the stability and oxidation behavior of the matrix and fiber, in a simulated post-combustion environment.

10:30 AM

(ICACC-S1-034-2018) Temperature Dependence of Modulus and Creep Behavior of SiC/SiC Ceramic Matrix Composite Constituents

V. Christensen*[1]; M. Schuster[2]; G. Henson[2]; J. Gao[2]

1. The Pennsylvania State University, Materials Science and Engineering, USA
2. GE Global Research, USA

Nanoindentation is used to understand the properties of silicon veins and silicon carbide matrix within a SiC/SiC ceramic matrix composite as a function of temperature. The temperature-mechanical property relationship provides input parameters that are crucial to the development of a multi-physics model of CMC failure behavior. Modulus and creep curves at RT, 400°C and 800°C tested under 5mN and 10mN peak loads were acquired using a nanoindenter with a Berkovich diamond tip and a high-temperature stage. The area function was monitored based on a fused quartz control, and a calibration control chart was frequently updated to ensure accurate data. Silicon creep rate increased with temperature while the reduced modulus decreased from 158GPa at RT to 139GPa at 400°C. The SiC matrix showed no statistically significant difference in modulus at high temperatures, yielding a reduced modulus consistently around 300GPa. With the current method, we are able to measure, with confidence and reasonable variation, the temperature dependence of modulus and creep behavior of individual constituents within a bulk CMC sample. This is based upon work supported by ARFL under Contract F865011C5227. Any opinions, findings and conclusions or recommendations expressed in this material are those of the author(s) and do not necessarily reflect the views of the USAF/AFMC Air Force Research Laboratory.

10:50 AM

(ICACC-S1-035-2018) Damage Evaluation of Ceramic Matrix Composites during Tension-tension Fatigue Loading using Non-destructive Health Monitoring Tools

Y. P. Singh[1]; M. Kannan*[1]; M. J. Presby[1]; G. N. Morscher[1]

1. University of Akron, Mechanical Engineering, USA

The need for understanding the damage accumulation in ceramic matrix composites are in the rise due to their properties at high temperature. In this article, the use of a technique called direct current electrical resistance (ER) via a multi-lead configuration is discussed. This technique is used to capture the details of damage initiation and progression during the tension-tension fatigue of a SiC/SiC ceramic matrix composite (CMC). The results from this method is then compared with the results recorded by acoustic emission (AE) and digital image correlation (DIC) and ER data was as sensitive to damage as the other techniques. Thus, ER could be used as a viable health monitoring tool to study and monitor damage in ceramic matrix composites.

11:10 AM

(ICACC-S1-036-2018) Influence of Thermal Cycles, Creep and Fiber Volume Fraction on Electrical Resistivity of SiC/SiC Minicomposites

A. S. Almansour*[1]; G. N. Morscher[2]

1. NASA Glenn Research Center, Ceramic & Polymer Composites Branch/ LMC, USA
2. University of Akron, Department of Mechanical Engineering, USA

Fiber-reinforced Ceramic Matrix Composites (CMCs) are currently being used in high-temperature applications such as jet engine components. In such extreme environment applications, materials are exposed to many mechanical and thermal loading cycles during the life of the component. This exposure can degrade the component, and thus, components need to be monitored and inspected during service. The electrical resistivity nondestructive evaluation technique has been shown to be very sensitive to damage in CMCs, and although its performance is highly dependent on temperature, this temperature-dependence is still not well understood. Therefore, this current study aims to establish the influence of thermal cycles, creep, CVI-SiC matrix cracks and fiber volume fraction on the electrical resistance of Hi Nicalon and Hi-Nicalon Type S SiC fiber-reinforced minicomposites with BN interphases. Minicomposites with 97% CVI-SiC matrix volume fraction were used to characterize the electrical resistivity of the CVI-SiC matrix. Next, models were constructed to calculate fiber resistivity and onset and evolution of damage during tensile testing at room-temperature. The thermal cycles were achieved by heating as-received and precracked specimens to 1200°C and then cooling them to room temperature, with or without applied stress on fibers.

11:30 AM

(ICACC-S1-037-2018) Effect of Porosity on Crack Propagation in SiC/SiC Ceramic Matrix Composites

M. J. Lancaster*[1]; A. L. Chamberlain[2]; R. Trice[1]; M. D. Sangid[1]

1. Purdue University, Materials Science and Engineering, USA
2. Rolls-Royce Corp, USA

Processing of woven SiC/SiC CMCs using chemical vapor, slurry, and silicon melt infiltration has the potential to introduce microstructural inhomogeneities. These inhomogeneities include intrinsic features, such as fiber tows constituent interfaces, but also defects including porosity, silicon rich areas, and variations in CVI thickness. Mechanical testing of SiC/SiC CMCs concentrating on bulk mechanical properties has revealed unexplained differences in the responses of the material with respect to these microstructural variations. Lack of understanding of the relationship between the microstructural features and the mechanical response prohibits establishment of quality criteria and continuous improvement strategies required to quickly bring SiC/SiC components to market. This ongoing research is focused on establishing the relation between the mechanical response of woven, infiltrated, SiC/SiC CMCs and one common microstructural feature induced in material processing: porosity. This work uses the damage monitoring capabilities of digital image correlation to track the damage evolution in and around porosity during bend testing. By monitoring the locations and extent of plastic strain accumulation at defects of controlled, size, shape, and location in the weave, the research hopes to highlight criterion for analyzing porosity to predict what effect, if any, it will have on the mechanical response.

S2: Advanced Ceramic Coatings for Structural, Environmental, and Functional Applications

CMAS Degradation of T/EBC & Mitigation Strategies II

Room: St. John

Session Chairs: Peter Mechnich, DLR - German Aerospace Center; Douglas Wolfe, Pennsylvania State University

8:30 AM

(ICACC-S2-031-2018) Effect of Porosity on Synthetic Sand Infiltration within Yttria-Stabilized Zirconia Pellets

A. Wright[1]; M. J. Walock*[2]; A. Ghoshal[2]; J. Luo[1]; A. Nieto[2]; M. Murugan[2]

1. University of California, San Diego, Nanoengineering, USA
2. US Army Research Laboratory, Vehicle Technology Directorate, USA

Yttria-stabilized zirconia pellets of varying composition (3 mol% and 8 mol% yttria) and porosity (0.5% - 60%) were processed by pressureless sintering, cold sintering, and spark plasma sintering. The consolidated pellets were characterized non-destructively through x-ray diffraction (XRD), scanning acoustic microscopy, confocal laser scanning microscopy, and with a scanning electron microscope equipped with an energy dispersive x-ray spectrometer preceding and following testing. Each pellet was exposed to AFRL-02 synthetic sand at high temperature (≈ 1300 °C) through means of either stationary or accelerated contact with an exposure time of approximately 15 minutes per pellet. Post-test characterization results were compared to the corresponding pre-test data to draw conclusions on the degree of infiltration and mechanism. Density measurements were calculated through Archimedes method and compared to the pre-test values to determine the mass/volume of sand infiltration along with any clues hinting at possible sintering. XRD results show the introduction of new crystal structures suggested to be synthetic quartz along with a negative peak shift of $2\theta = 0.5°$, which may be compressive strain.

8:50 AM

(ICACC-S2-032-2018) Development of an Integrated Framework to Design Thermal and Environmental Barrier Coatings Resistant to Molten Silicate Degradation

D. L. Poerschke*[1]

1. University of Minnesota, Chemical Engineering and Materials Science, USA

The development of thermal and environmental barrier coatings (TBCs and EBCs) offering improved resistance to accelerated degradation caused by molten silicate (CMAS) deposits is critical to increasing gas turbine efficiency. The process of identifying suitable coating architectures is complicated by both the multiple competing coating design requirements and the variability in the nature of the deposits arising in service. Efforts to accelerate the development of improved coatings require an integrated approach with models capturing the influence of service conditions, the coating material

and microstructure, and the deposit composition on the severity of degradation. The present work describes progress toward developing and validating a design framework integrating the phase equilibrium and reaction dynamics models with descriptions of the relevant failure mechanisms. The findings provide insight into the relative efficacy of various prospective coating materials in mitigating the deposit-induced degradation.

9:10 AM

(ICACC-S2-033-2018) Calcium-Magnesium-Alumino-Silicate (CMAS) viscosity effects on the lifetime of thermal barrier coatings

B. Jun*[1]; E. H. Jordan[2]; N. Jonsson[1]

1. University of Connecticut, Materials Science Engineering, USA
2. University of Connecticut, Mechanical Engineering, USA

Thermal barrier coatings can experience reduced lifetimes due to the infiltration of calcium-magnesium-alumino-silicate (CMAS). The infiltration of CMAS depends on viscosity. We perform experiments on thermal barrier coatings in a thermal gradient rig that simulates conditions of turbine blades exposed to environmental CMAS conditions. Two CMAS compositions with different predicted viscosities will be compared. The viscosities will be measured and compared to model predictions. Infiltration depth of the CMAS is monitored over the lifetime of the thermal barrier coatings

9:30 AM

(ICACC-S2-034-2018) Thermo-Corrosive Properties by CMAS and Volcanic Ash of RE-doped Silicate Environmental Barrier Coatings (Invited)

B. Jang*[1]; S. Ueno[2]; K. Lee[3]; S. Kim[4]; Y. Oh[4]; H. Kim[4]

1. National Institute for Materials Science, Research Center for Structural Materials, Japan
2. Nihon University, College of Engineering, Japan
3. Kookmin University, School of Mechanical Systems Engineering, Japan
4. Korea Institute of Ceramic Engineering and Technology, Republic of Korea

The improvement of the gas turbine inlet temperature is a key factor involved in increasing the fuel efficiency and reducing the carbon emissions of a gas turbine. Due to the high limit point of temperature capability, non-oxide silicon-based ceramics, such as SiC/SiC_f, Si_3N_4 and SiC, have been investigated extensively as potential structural material for hot gas parts for next-generation gas turbines. However, abundant investigations have indicated that the disadvantageous factor of Si_3N_4 and SiC as it applies to gas turbines is that they lose observable weight in the combustion environment. In the present study, the influence of isothermal heat treatment on thermo-chemical properties of Y_2SiO_5 EBCs (environmental barrier coatings) on SiC was investigated. The hot corrosion between Y_2SiO_5 coatings and artificial CMAS ($CaO–MgO–Al_2O_3–SiO_2$) was examined by isothermal heating at 1400°C in air during 1~50 hrs. The evaluation of hardness and Young's modulus was performed on the cross-section of Y_2SiO_5 coatings by nano indentation method. The isothermal heat treatment improves the hardness and Young's modulus of Y_2SiO_5 coatings. In addition, high-temperature corrosion behavior of volcanic ash and CMAS on sintered Yb_2SiO_5 and mullite are investigated.

10:20 AM

(ICACC-S2-035-2018) Environmental Barrier Coating Ceramics and their High-Temperature Interactions with Calcium-Magnesium-Aluminosilicate (CMAS) Glass

L. R. Turcer*[1]; A. Krause[1]; H. Garces[1]; N. Padture[1]

1. Brown University, Engineering, USA

Ceramic-matrix-composites (CMCs) are replacing current metallic hot-section components, which allows for higher engine-operating temperatures. In the presence of water vapor, active-oxidation of CMCs is prevalent. Therefore dense environmental barrier coatings (EBCs) are needed to protect CMCs. At temperatures above 1200 °C, silicate particles (sand, volcanic ash, fly ash, etc.) that enter the engine melt on the hot surfaces and form calcium-magnesium-aluminosilicate (CMAS) glass deposits. These molten CMAS deposits can penetrate grain boundaries and/or cause reactions, which leads to premature EBC failure. New EBC ceramics are needed that are resistant to CMAS attack. A new model based on optical basicities has been used to predict the reactivity between CMAS and potential EBC ceramics. Based on this analysis, several potential EBC ceramics have been identified. Their high-temperature interactions with CMAS are studied, and the nature of these interactions, damage mechanisms, and their mitigation effects are elucidated.

10:40 AM

(ICACC-S2-036-2018) Examining the Role of Process Induced Porosity Differences on CMAS Interactions in Plasma Sprayed Thermal Barrier Coatings

E. J. Gildersleeve*[1]; S. Sampath[1]

1. Stony Brook University, Materials Science, USA

The continued need for higher gas turbine operating temperatures has necessitated developments in new thermal barrier (TBC) materials and their processing. With increasing operating temperatures comes the possibility for ingested siliceous debris (CMAS) to become molten and adhere to the surface of the TBC. Depending on the coating material and deposition process, these molten debris can flow freely into the entirety of the microstructure or be arrested at the surface. Of interest are yttria stabilized zirconia (YSZ) and Gadolinium Zirconate (GDZ), which elicit these two independent interactions with the molten silicates. In each of the cases, beyond their unique interaction with CMAS, the inherent microstructure of the coating will have an additional role on the movement of the melt. For example, coatings with high porosity (>15%) or coatings which are dense with vertical cracks (DVC) will have different penetration mechanisms. This study seeks to understand this interplay between the molten CMAS flow and the process-induced microstructure. Different types of YSZ & GDZ single and multilayer coatings were fabricated using different processes (Air Plasma Spray; Suspension Plasma Spray) and exposed to CMAS through isothermal and gradient mechanisms. Results indicate a dependence on the process-induced microstructure and the CMAS interaction.

11:00 AM

(ICACC-S2-037-2018) Characterization of a Volcanic Ash Glass Related to Particulate Degradation of Protective Barrier Coatings

R. Webster*[1]; V. L. Wiesner[2]; J. Salem[2]; N. P. Bansal[2]; E. J. Opila[1]

1. University of Virginia, Materials Science & Engineering, USA
2. NASA Glenn Research Center, USA

Calcium magnesium alumino-silicate (CMAS) attack is a pressing issue in the development of Thermal/Environmental Barrier Coatings (T/EBCs) for Ceramic Matrix Composites (CMCs). CMAS originates as siliceous debris such as sand or volcanic ash which can be ingested into aircraft engine turbines. At temperatures nearing 1200°C, CMAS becomes molten and can penetrate and thermo-chemically interact with T/EBC materials. In this study, ash from the 2010 Eyjafjallajökull volcano eruption was obtained and melted

into glass. The intrinsic properties of this glass were studied. Bulk glass pieces were exposed to temperatures between 900-1300°C and were either cooled at 10°C/min or air quenched. The resulting samples were crushed for phase analysis by x-ray diffraction (XRD) or mounted in epoxy and polished for microstructural investigation by scanning electron microscopy (SEM). Preliminary results indicate the main crystalline phases that form in this temperature region include iron oxides Fe_3O_4 and Fe_2O_3. Bulk and powdered glass was also analyzed via differential scanning calorimetry (DSC) at variable heating rates to determine crystallization kinetics. Additionally, glass discs were mechanically tested to determine properties such as hardness, toughness, and elastic modulus. The results of this study will be compared to those of a synthetic sand glass.

11:20 AM

(ICACC-S2-038-2018) Resistance of APS Y_2O_3 EBC against two artificial volcanic ash variants

P. Mechnich*[1]

1. DLR - German Aerospace Center, Institute of Materials Research, Germany

Ceramic matrix composites (CMC) are being developed for next generation aero-engines to substitute conventional superalloy components. However, the synergistic attack of increasing operation temperatures and water-rich exhaust gases demands for environmental barrier coatings (EBC) for protecting CMC components against thermochemical degradation. At DLR, a model CMC combustor liner with air-plasma-sprayed (APS) Y_2O_3 EBC has been demonstrated recently. Beyond heat and water-rich exhaust gases, ingestion and subsequent deposition of inorganic aerosols such volcanic ash (VA) is considered a major issue for EBC lifetime. In particular infiltration of open porosity by molten VA deposits is considered a major reason for "cold shock" fracture and subsequent EBC spallation. The behavior of APS Y_2O_3 EBC attacked by two test dusts similar to recent volcanic eruptions in Iceland, (Ejafjalla, 2010 and Grimsvotn, 2011), is presented in the light of phase formation, solid solubility, and microstructural evolution. APS Y_2O_3 EBC exhibit excellent resistance versus both VA-types at temperatures up to 1500°C at minimum. Melt infiltration is effectively mitigated by a low-permeable microstructure and rapid crystallization of Y-rich phases such as oxyapatites and silicates. Therfore, the thermochemical attack is occurring only close to the EBC surface.

11:40 AM

(ICACC-S2-039-2018) Lifetime evaluation of EB-PVD 7YSZ coatings in thermal gradient rig tests under the influence of CMAS and VA attack (Invited)

R. Naraparaju*[1]; U. Schulz[1]; L. Hochstein[1]

1. DLR - German Aerospace Center, Materials Research, Germany

CMAS deposits have been found to be detrimental to the durability of Thermal barrier coatings (TBCs). The hot-corrosion attack of this siliceous liquid with the TBC material is very severe and leads to the ultimate failure of the TBCs. From the previous experimental knowledge on the interaction between CMAS and EB-PVD 7YSZ, it is known that CMAS melts at high temperatures and infiltrates through the porous inter columnar TBC structure with the help of capillary forces. This infiltration causes severe mechanical stresses within the TBC upon thermal cycling, subsequently leading to crack formation and TBC spallation. Most of the life time assessment tests were so far conducted in isothermal atmosphere which generally does not represent real engine atmosphere. In this study a thermal gradient test rig has been used to study the TBC life time behaviour under the influence of CMAS and volcanic ash. EB-PVD 7YSZ coatings on Ni-based superalloys protected by a NiCoCrAlY bond coat are infiltrated with CMAS/VA and thermal cyclic tests in isothermal and thermal gradient conditions are performed. It was found out that the CMAS infiltration depth has a direct impact on the life time of the coating and a fully infiltrated sample has a life time reduction up to 90% compared to non-infiltrated coating. All coatings were investigated under SEM and failure modes are analysed.

S3: 15th International Symposium on Solid Oxide Fuel Cells (SOFC): Materials, Science and Technology

Coatings I

Room: Crystal

Session Chair: Kevin Huang, University of South Carolina

8:30 AM

(ICACC-S3-026-2018) Electrodeposition of Mn-Co alloys for SOFC Interconnect application (Invited)

J. Wu[1]; X. Liu*[1]

1. West Virginia University, Mechanical & Aerospace Engineering, USA

Electrodeposition of Mn-Co alloy film followed by controlled oxidation to achieve the desired spinel phase offers a deposition option that is both cost effective and adaptable to work piece geometry. This work presents Mn/Co alloys deposition by DC and pulse plating. The dramatic difference of deposition potentials of Mn and Co makes it difficult to co-deposit them in thermodynamics aspect. Potentiodynamic polarization and CV were conducted to characterize the reactions occurring during deposition. Effects of current density and pulse cycle on the surface morphologies and compositions of coatings were studied. Mn content increases with the on-time increasing, and surface morphologies changes from flake like structure to crystalline structures with less pores. ASR measurement is used to characterize the conductivity of the coating. In tests of 1200 h, ASR is stable with a slight increase. The ASR value at 40,000was predicted to be 0.0460_Ω cm^2, which is well below the industry accepted goal of 0.1 Ω cm^2. Following and interconnect on-cell tests have substantiated that the coating by electrodeposition can successfully block chromia evaporation, maintain high conductivity and survive thermal cycles. Therefore, the simple and cost effective coating technique is a great option for SOFC interconnects.

9:00 AM

(ICACC-S3-027-2018) Protective Coatings for SOFC Metallic Interconnects

M. K. King*[1]; M. Mahapatra[1]

1. University of Alabama at Birmingham, Materials Science and Engineering, USA

Corrosion and chromium evaporation from commonly used metallic interconnects in humidified oxidizing environment during SOFC operation remains an issue. We have studied the role of pretreatment of AISI 430 alloy with or without nickel coating on the oxidation resistance and formation of interfacial compounds at 650-800°C in moisture-saturated air. It is found that electrodeposited nickel coated AISI 430 alloy pretreated in dry hydrogen (Ar-3% H_2) improves the oxidation resistance but not preoxidized alloy. Nickel coating by electroless method does not improve oxidation resistance. The underlying mechanisms for improved oxidation resistance will be discussed using structural and microstructural analysis supported by thermochemical calculations.

9:20 AM

(ICACC-S3-028-2018) Evaluation of Fe-doped $(CuMn)_3O_4$ Coatings on Metallic Interconnects as a Protective Barrier to Prevent Cr-poisoning in Solid Oxide Fuel Cells

Z. Sun*[1]; R. Wang[1]; Y. Gong[1]; S. Gopalan[1]; U. Pal[1]; S. Basu[1]

1. Boston University, MS&E, USA

Ferrite stainless steels, used as interconnects in intermediate temperature solid oxide fuel cells (IT-SOFCs), form Cr_2O_3 scales at operating temperatures. This leads to the formation of Cr^{6+} containing vapor species that poisons the SOFC cathodes. Applying a coating on the surface of interconnects to act as a protective barrier is an effective solution to this problem. Fe-doped $(CuMn)_3O_4$ spinel was investigated as a potential coating material. The conductivity, coefficient of thermal expansion (CTE) and phase stability of bulk Fe-doped $(CuMn)_3O_4$ spinel were first studied. Then coatings of this composition were deposited on interconnects by electrophoretic deposition (EPD) and densified by reduction and re-oxidation. To study the effectiveness of these coatings, they were analyzed for area specific resistance (ASR), weight gain, Cr distribution, and changes in morphology as a function of oxidation time. Comparison of the effectiveness of $(CuMn)_3O_4$ and Fe-doped $(CuMn)_3O_4$ will also be discussed.

9:40 AM

(ICACC-S3-029-2018) Microstructural and electrical characterization of Cu and Fe-doped Mn-Co spinel protective coatings for solid oxide cell interconnects

F. Smeacetto*[1]; A. Sabato[1]; H. Javed[1]; B. Talic[2]; S. Molin[2]

1. Politecnico di Torino, Applied Science and Technology, Italy
2. DTU, Energy Conversion and Storage, Denmark

MnCo-based coatingsare considered as one of the best materials to limit chromium evaporation and to reduce corrosion on the oxygen side of the interconnect in solid oxide cells. However, there is a lack of real conditions long term studies, where effects of contact pastes, electrical resistance and oxidation kinetics, are evaluated to assess the overall efficiency of coating protection. In this work, Mn-Co (un-doped, Fe and Cu-doped) coated metallic interconnect samples are successfully processed by electrophoretic deposition, with an optimized thickness around 10 μm. Suspensions with different amounts of Cu and Fe-doped Mn-Co spinels are optimized to improve the Mn-Co-based coatings in terms of densification and electrical properties. Structural and compositional modifications in the manganese cobaltite spinel and in the coating properties due to the Cu and Fe doping are reviewed and discussed. EPD coated metallic interconnect samples are tested up to 2500-5000 hours at 800°C under current load, to determine their area specific resistance increase as a result of the growing chromia scale. Oxidation kinetics are periodically evaluated after 1000 hrs. Samples are morphologically analysed by post-mortem SEM-EDS and TEM, to determine the oxide scale thickness, possible new phases formation and to examine for evidence of chromium diffusion.

Coatings II / Contacting

Room: Crystal

Session Chair: Joseph Barton, University of Utah

10:20 AM

(ICACC-S3-030-2018) Aluminized stainless steel as a corrosion-resistant material for application in high-temperature fuel cells

M. Kusnezoff[1]; E. Medvedovski*[2]; M. Vinnichenko[1]; C. Folgner[1]; V. Sauchuk[1]

1. Fraunhofer IKTS, Germany
2. Endurance Technologies Inc., Canada

High-temperature corrosion of metallic alloys in oxidizing gas is one of the important factors limiting performance, stability and lifetime of solid oxide and molten carbonate fuel cells. Interaction with molten salts in the latter case makes corrosion protection especially challenging. The present work focuses on the investigation of the aluminizing thermal diffusion process as an approach to resolve this problem. This method was applied to protect samples of 316-type austenitic stainless steel (SS), which were subsequently tested in alkali carbonate melt at 650°C in the O_2 and CO_2 containing gas mixture for 1000 h. The corrosion behavior of the aluminized SS, as well as the aluminized SS with additional thin top ceramic layers, was systematically compared to the reference SS samples coated with Al by PVD and hot dip methods. The analysis of the samples' cross-section morphology (FESEM) and elemental distribution (EDX mapping) shows superior corrosion resistance of the aluminized steel. Neither signs of diffusion of the steel components (Fe, Cr, Ni) into the carbonate melt nor degradation of the aluminized coatings' surfaces after the tests were found. The high-temperature corrosion tests in air without carbonate melt are underway.

10:40 AM

(ICACC-S3-031-2018) Development of Cathode Contacting for SOFC Stacks

K. Sick (Schönauer)*[1]; N. Grigorev[1]; N. H. Menzler[1]; O. Guillon[1]

1. Forschungszentrum Juelich, Institute for Energy and Climate Research (IEK-1), Germany

In SOFC stacks the electrical contact between the ceramic cathode layer and the metallic interconnector (IC) has to be adjusted carefully to minimize the contact resistance and slow down degradation mechanisms. In JÜLICH, ICs made of Crofer22 are coated with a $MnCo_{1.9}Fe_{0.1}O_2$ spinel (MCF), normally applied by atmospheric plasma spraying (APS), which successfully prevents the diffusion of volatile Cr species from the steel into the cell. Materials for the cathode contact layer have to be sufficiently good electronic conductors, stable in oxidizing atmosphere, chemically stable and compatible with the adjacent layers, and show a thermal expansion behavior similar to MCF and the cathode material, i. e. $La_{0.58}Sr_{0.4}Co_{0.2}Fe_{0.8}O_{3-x}$ (LSCF). To obtain an optimal contact layer, different aspects such as material properties, microstructure, and processing have to be considered. Here we compare various materials regarding their electrical conductivity and compatibility with each other and discuss different processing routes for the application of the cathode contact layer. DC conductivity measurements and scanning electron microscopy (SEM) imaging give information on the correlation of material properties and microstructure. We conclude that an LSCF contact layer with coarse porosity on top of the fine structured LSCF cathode is the most suitable contacting for JÜLICH SOC stacks.

11:00 AM

(ICACC-S3-032-2018) Investigation of zirconia fiber felt and cloth as cathode contact material

Y. Chou*[1]; J. F. Bonnett[1]; J. W. Stevenson[1]

1. Pacific Northwest National Lab, Materials, USA

The mechanical integrity of the cathode contact plays an important role in the performance of solid oxide fuel cells. This is because the final stack firing is typically a few hundred degrees lower than normal sintering temperatures, which leads to poor solid-state sintering and low strength. In previous work at PNNL, we have demonstrated that the strength can be improved by mechanical interlocking with surface roughening as well as the use of Bi2O3 as a sintering aid. Both have approaches improved the contact strength and thermal cycle stability. Recently, we have continued the work by employing flexible porous fiber felt and cloth impregnated with a conducting phase such as LSM and LSCo. YSZ fiber felt and woven cloth were dip coated with slurry and calcined at elevated temperatures. Simple linear relationship of the weight of coated material versus number of dip coating was determined. The impregnated samples were further bonded with candidate contact paste to various substrates. Contact strength was evaluated with a peel off

test using scotch tape for as-sintered and 10 thermal cycled samples. The impregnated zirconia fiber felt and cloth was further tested for electrical conductivity, and correlated with porosity. Microstructures of the impregnated sample and interfaces were characterized to assess the viability of fiber felt/cloth as contact materials for SOFC applications.

11:20 AM

(ICACC-S3-033-2018) Quantitative Defect Chemistry and Electronic Conductivity Analysis of $(La_{1-x}Ca_x)_yFeO_{3\pm\delta}$ and $(La_{1-x}Sr_x)_yMnO_{3\pm\delta}$ Perovskite

S. Darvish*[1]; Y. Zhong[2]

1. Florida International University, Mechanical and Materials Engineering, USA
2. Worcester Polytechnic Institute, Mechanical Engineering Department, USA

The defect chemistry inside the $(La_{1-x}Ca_x)_yFeO_{3\pm\delta}$ and $(La_{1-x}Sr_x)_yMnO_{3\pm\delta}$ perovskite was analyzed using the La-Ca-Fe-O and Ls-Sr-Mn-O quaternary thermodynamic database, respectively. The quantitative Brouwer diagram for LCF and LSM were developed to show the concentration of all the species in A-site, B-site, and O-site of the perovskite with the change of oxygen partial pressures at different temperatures. In addition, the detailed defect chemistry analysis has been adopted on the prediction of the electronic conductivity of LCF and LSM with the compound energy formalism model by applying the CALPHAD approach. The calculations on the electronic conductivity of LCF and LSM show agreement with the high temperature experimental data in the literatures. The effect of ionic conductivity has been also investigated for these two systems and compared.

11:40 AM

(ICACC-S3-054-2018) Investigation of $(La_{1-X},Ca_X)(Ni_{0.25}Fe_{0.25}Cr_{0.25}Co_{0.25})O_3$ for SOFC Cathode Materials

S. Gajjala*[1]

1. Southern Illinois University Carbondale, Mechanical Engineering and Energy Processes, USA

The purpose of this research is to develop cathode material for use in SOFC which demonstrates desired properties of high electrical conductivity, good chemical stability at elevated temperatures, desirable thermal expansion characteristics and which can be easily manufactured by sintering in conditions acceptable with other cell components. In the present research, stoichiometry's of La1-xCax(Fe0.25Co0.25Ni0.25Cr0.25)O3(x=0-0.3) were synthesized by using polymerizable precursor method proposed by Pechini. The structure and morphology of the powder samples were characterized by X-ray diffraction and SEM. X-ray diffraction results revealed the formation of single phase orthorhombic distorted perovskite structure in all four samples. Pressed pellets were sintered in air at 1400°C for 2 hours. SEM analysis showed densification of the pellets with the addition of calcium. D.C. electrical conductivity measurements on the sample were made using the AC resistance bridge between the temperature range of 100-900°C in air. Study of electrical conductivity showed an increasing trend in electrical conductivity with increase in temperature and the amount of calcium doped on A-site up to 20mol% and the sample with calcium 30mol% on A-site, showed sharp increase in electrical conductivity reaching a maximum of 50 S/cm at 800°C, showing that the present materials can be used as cathode material in SOFC.

S4: Armor Ceramics - Challenges and New Developments

Synthesis and Processing II

Room: Coquina Salon F
Session Chair: Steve Kilczewski, Army Research Laboratory

8:30 AM

(ICACC-S4-031-2018) Slip casting of submicron SiC powder

V. Johannessen[1]; B. Watremetz*[1]; C. Bousquet[1]

1. Saint-Gobain Ceramic Materials, R&D, Norway

Saint-Gobain Ceramic Materials is supplying a large number of SiC powders, including Densitec® ready to press mixes. Our R&D team has recently been working to develop a new type of mix based on submicron powder that can be used to prepare a slurry. This new slurry can then be slip casted to make dense, high performance, SiC components for a variety of applications. The effort has included both the solid state and the liquid phase sintering systems. The preliminary tests and literature study showed that it was very difficult to reach a solid content higher than 70% by weight and at the same time keep the viscosity low. Low viscosity and high solids loading is needed to achieve the high green density required to get high sintered density and reduce shrinkage during firing. The work presented in this paper has focused on developing the optimum recipe for industrial applications. This includes production of a tailor made SiC powder as well as a balanced addition of sintering aids and binders. As a result, we managed to produce greens parts with a density higher than 1.9 g/cm^3, leading to a sintered density above 95%. It will also be shown that the final mechanical properties of such components have comparable performance to parts made out of our standard pressed Densitec® powders. This new family of castable products opens up new possibilities for producing dense sintered SiC components of complex shape.

8:50 AM

(ICACC-S4-032-2018) Single-mode microwave sintering of traditionally resistant materials

V. L. Blair*[1]; M. Kornecki[2]; S. Raju[3]; R. E. Brennan[1]

1. US Army Research Laboratory, Weapons and Materials Research Directorate, USA
2. SURVICE Engineering, USA
3. ORAU, USA

Microwaves can be exploited by designing a resonant cavity to produce a standing wave in the sample chamber in which the electric and magnetic field components are spatially shifted, leading to discrete regions in which the ratio of these components ranges from a minimum to a maximum. Processing of ceramics and metals under microwave frequencies has been pursued for some time, but with limited success, as only a few ceramics typically sinter through the use of microwave energy. These ceramics generally have high dielectric losses, which causes heat to build up in the material to promote uniform densification and produces a fine grain structure. However, most materials are not microwave-susceptible, and require an external susceptor to harness the microwave energy and transfer the heat to the sample. Alternatively, internal susceptors can provide localized heating of the microwave transparent ceramics thereby reducing sintering time which can help maintain finer grain size. In this work, efforts to blend hybrid ceramic materials, in which a ferromagnetic material acts as an internal susceptor and the other component is microwave-transparent, are being pursued to obtain dense materials with fine microstructures. This work will also use a single-mode microwave cavity to gain unique processing parameters by separating out the electric and magnetic field contributions.

9:10 AM

(ICACC-S4-033-2018) Machineable Graphene Incorporated Ceramic (MaGIC) for Light Armour Applications

J. Kenny*[1]; J. Binner[1]; I. Chang[2]; S. Marinel[3]; N. McDonald[1]

1. University of Birmingham, Materials & Metallurgy, United Kingdom
2. Brunel University London, Institute of Materials and Manufacturing, United Kingdom
3. ENSICAEN, CRISMAT, France

Homogenous nanocomposite samples have been produced from reduced graphene oxide (rGO) and boron carbide (B_4C) – derived from both commercially available and low carbon (C) low temperature synthesised (LTS) sources - using spark plasma sintering (SPS). This was achieved via the colloidal processing of aqueous graphene oxide (GO) and boron carbide (B_4C) suspensions before the samples were filtered, dried and consolidated, during which the in-situ reduction of GO to rGO was also achieved. Elemental analysis demonstrated that the LTS B_4C exhibited a slightly lower C content overall compared to its commercial counterpart. Both B_4C variants were sintered at 1900ºC over 40 mins without any additives. The addition of 2, 4.5 & 9.0 wt% GO to both variants reduced the overall sample sintering time to 15 mins. Using these regimes, every sample below 9 wt% GO was made to within 98.5% of its theoretical density. Physical and chemical characterisation methods, such as scanning electron microscopy, zeta potential analysis, Raman spectrometry and X-ray diffraction demonstrated that all of the samples were homogeneously dispersed - both before as well as after sintering. The physical properties of each sample were also investigated by measuring their respective Vickers microhardness and corner crack-propagation resistance values under a 1kg loading force.

9:30 AM

(ICACC-S4-034-2018) Microstructural study in B_4C-SiC ceramic-ceramic composites

T. Shoulders*[1]; K. D. Behler[1]; J. LaSalvia[1]; L. Vargas[1]

1. US Army Research Laboratory, USA

Body armor research has moved increasingly towards engineered structures at length scales ranging from the atomic scale (defects and grain boundaries) all the way to the macro-scale (layering, segmenting). One of the most popular trends in experimental armor blends is modification at the micro-scale. Although there have been some successes, our understanding of the structure-dependent mechanisms acting during a ballistic event is still lacking. This work takes a bottom-up approach to study processing and structure as a first step in understanding these complex relationships. A series of B_4C-SiC composites with compositional variation between the monolithic ceramics in increments of 10 wt% have been hot-pressed. Microstructures are controlled by the agglomeration and sieving of component powders. Full structural and chemical characterization is performed using optical microscopy, scanning election microscopy (SEM), x-ray fluorescence (XRF), x-ray diffraction (XRD), and micro-Raman spectroscopy. Knoop hardness values were also determined. Experimental procedures and results are presented.

9:50 AM

(ICACC-S4-035-2018) Synthesis, sintering and mechanical properties of Boron suboxide (B_6O)

A. U. Khan*[1]

1. Rutgers University, Materials Science and Engineering, USA

Boron rich compounds are attractive materials for armor related applications. Boron suboxide (B_6O) is one of the most promising candidates for this purpose, owing to its low density and high hardness. However, sintering of this compound has been challenging over the years. We successfully synthesized boron suboxide by mixing amorphous boron and boric acid powders, and heating at 1400 °C. This synthesized powder was then sintered with different sintering aids, in spark plasma sintering (SPS) at 1800 °C under 50MPa, in a graphite die, with BN sprayed on all surfaces in contact with the sample. More than 99% of the theoretical density was achieved. Elastic constants were measured by employing ultrasound. Average Vickers hardness was found to be >30GPa under 1Kg load. Sintered pellets were characterized by X-ray diffraction, scanning electron microscope, and Raman spectroscopy.

10:10 AM

(ICACC-S4-036-2018) Processing, Microstructure, and Properties of Pressure-Assisted Sintered Boron Suboxide with Oxide Additives

K. D. Behler*[1]; C. J. Marvel[2]; T. Shoulders[3]; J. LaSalvia[4]; L. R. Vargas-Gonzalez[4]; M. P. Harmer[2]

1. SURVICE Engineering and U.S. Army Research Lab, Ceramics and Transparent Materials Branch, USA
2. Lehigh University, Materials Science and Engineering, USA
3. U.S. Army Research Lab and ORISE, Ceramics and Transparent Materials Branch, USA
4. U.S. Army Research Lab, Ceramics and Transparent Materials Branch, USA

The intrinsic fracture resistance of boron suboxide is inherently low due to strong atomic bonding and a relatively open crystal structure. Fracture resistance can be improved extrinsically by microstructural engineering at the atomic, nano, and micro length-scales. To this end, the processing, microstructure, and properties of boron suboxide processed with silica and rare-earth oxides were studied. Boron suboxide powders with 1-5vol.% silica and 2 and 5vol.% silica plus varying amounts of rare-earth oxides additions were hot-pressed between 1700-1850°C for 3hr and 50MPa. All samples were fully dense at 1850°C. Dense samples were metallographically prepared and examined by XRD, SEM, and EDS. Additives were well dispersed and primarily located at triple junctions. Samples with rare-earth additions showed several phases and exhibited grain boundary segregation. The addition of 1vol.% silica resulted in a maximum Knoop hardness of ~21MPa, while higher silica contents were comparable to boron suboxide without additives. Experimental procedures and results are presented.

10:30 AM

(ICACC-S4-037-2018) Incorporation of TiB_2 as Intergranular Phase in Boron Carbide

C. Hwang*[1]; S. DiPietro[2]; K. Xie[3]; Q. Yang[1]; A. U. Khan[1]; V. Domnich[1]; K. Hemker[3]; R. A. Haber[1]

1. Rutgers University, Dept. of Materials Science and Engineering, USA
2. Exothermics, Inc., USA
3. Johns Hopkins University, Department of Mechanical Engineering, USA

Boron carbide has proven its versatility for a wide range of technical applications, from lightweight armor ceramics to high temperature semiconductors, because of its unique properties, such as high hardness, low density, and high melting point. However, it has critical issues that have limited its further widespread applications, which are catastrophic failure under high-velocity impacts, poor fracture behavior, and poor sinterability. Here we report the progress of a new approach that is incorporate a secondary hard phase (TiB_2) as intergranular or, ideally, grain boundary phase in boron carbide to improve its fracture behavior. We synthesized boron carbide powder with nanoscale surface coating of TiB_2 using DC magnetron sputtering process and densified the surface-coated powder using hot pressing process. We will address i) how to improve sintering density of sputtered powder, ii) the effect of variations in sputtering amount and configuration on microstructure development, and iii) possible effect of TiB_2 surface coating on the modification of grain boundary, iv) the effect of TiB_2 intergranular or grain boundary phase on the fracture behavior of boron carbide. Characteristics of surface coated powder and its resultant dense body have been studied using XRD, FE-SEM/EDS, Raman spectroscopy, TEM/EDX, and indentation method.

10:50 AM

(ICACC-S4-038-2018) Forming Transparent Alumina by Hot-Pressing Aligned Platelet-Grain α-Al_2O_3: Processing and Preliminary Results

A. Schlup*[1]; R. Trice[1]; J. P. Youngblood[1]

1. Purdue University, Materials Engineering, USA

Transparent polycrystalline ceramics have many useful military applications, such as ballistic blast shields and sensor protection in aircraft. The transparency of alpha-alumina (α-Al_2O_3) is known to be improved by aligning the grains along their basal planes. In the present work, shear flow in a thermoplastic polymer is utilized to align platelet grain α-Al_2O_3 along its basal planes. Subsequent polymer burnout and hot-pressing is used to form the final part. This unique processing method will be discussed, as well as the effect of sintering time, temperature, and pressure on the density and transparency of the formed parts.

Terminal Ballistics

Room: Coquina Salon F

Session Chair: Brady Aydelotte, US Army Research Laboratory

11:10 AM

(ICACC-S4-039-2018) Cone fragment formation in ceramics by steel sphere impact: Experiments and simulations

E. Carton*[1]; G. Roebroeks[1]; E. Simons[2]; B. Sluys[2]; J. Weerheijm[1]

1. TNO, Netherlands
2. Delft University of Technology, Civil Engineering, Netherlands

At low velocity the impact of a small steel sphere on a brittle material often results in a cone crack in the latter. If the impact velocity is high enough the cone crack propagates until the rear of the tile and a single cone fragment is created. As the cone formation also seems to play a role in the ballistic velocity range, its formation and parameters that affect the cone angle have been explored both experimentally and using finite element simulations. Several types of ceramics with various tile thicknesses have been used. The sphere impact tests were performed in the velocity range of 50-800 m/s. The cone angle changes with impact velocity and the amount of cracks of the cone increases, eventually generating a fragment cloud rather than an intact cone. The low velocity impact of a steel sphere on a ceramic tile has also been simulated using a modified JH-model. When studying crack initiation and propagation with the JH-model spurious localization and mesh bias may introduce serious errors. To prevent mesh dependency a modified model has been developed where a viscosity is introduced to provide regularization. The simulations showed the formation of the cone crack, ring cracks and internal damage of the cone fragment formed. The induced crack pattern has been studied varying the impact velocity and the material properties and compared with the experimental results.

11:30 AM

(ICACC-S4-040-2018) Influence of Hardness and Toughness of Ceramic on Ballistic Performance of Ceramic Armour

W. Goh*[2]; B. Luo[1]; Z. Zeng[1]; J. Yuan[1]; K. Ng[2]

1. Nanyang Technological University, Temasek Laboratories, Singapore
2. Nanyang Technological University, School of Materials Science and Engineering, Singapore

A series of experiments were conducted to investigate influence of hardness and toughness on ballistic performance of ceramic armour. The experiments were conducted using ceramics from 3M technical ceramic using the tiles grade T, T+, F and F+, with dimensions of 100mm X 100mm X 20mm. Hardness and fracture toughness of the ceramic were measured using the indentation technique. The ballistic performance of the ceramic samples were assessed using depth of penetration tests and mass efficiency index. The ceramic samples were tested against tungsten alloy long rod projectiles at a nominal velocity of 1.25km/s. Through a single tile configuration module test, mass efficiency measured increased from 1.53 to 1.73 when hardness of ceramic tiles increased from HV 20.6±0.33GPa to HV 24.4±0.95GPa, while fracture toughness had no observable influence. However, fracture toughness of the ceramic was observed to have significant influence on the damage radius of the ceramic armour, which is an important consideration for mitigating multiple impacts. Through a six-tile configuration module test, grade F+ SiC (K_{Ic} = 2.87±0.23MPa.$m^{1/2}$) exhibited a damage radius of 420mm while grade T+ SiC (K_{Ic} = 6.99±0.78MPa.$m^{1/2}$) recorded a damage radius of 138mm. These experimental results were analysed by computer simulations using LS-Dyna.

S6: Advanced Materials and Technologies for Direct Thermal Energy Conversion and Rechargeable Energy Storage

Thermoelectrics I

Room: Tomoka A

Session Chair: Michihiro Ohta, National Institute of Advanced Industrial Science and Technology (AIST)

8:30 AM

(ICACC-S6-024-2018) Continuous efforts towards high performance half-Heusler thermoelectric materials (Invited)

T. Zhu*[1]

1. Zhejiang University, School of Materials Science and Engineering, China

Half-Heusler (HH) compounds are important high temperature thermoelectric (TE) materials having attracted considerable attention in the recent years. High figure of merit zT values of ~1.0 have been obtained in n-type ZrNiSn based HH compounds. However, developing high performance p-type HH compounds with low cost is a big challenge. In this talk, we first show that a new p-type HH solid solutions with a high band degeneracy, Ti doped FeV0.6Nb0.4Sb, can achieve a high zT of 0.8. Further investigation shows that increasing Nb content in the Fe(V1-yNby)Sb solid solutions can achieve lower valence band effective mass and consequently higher carrier mobility. Moreover, the decrease in band effective mass can lead to the decrease in optimal carrier concentration, which is favorable for p-type Fe(V,Nb)1-xTixSb due to the limited solubility of Ti. Thus we obtain a high zT of 1.1 at 1100K for FeNb1-xTixSb without V substitution. More recently, we found that Hf doped FeNbSb exhibits a record high zT of 1.5 at 1200K due to simultaneously optimized electrical power factors and reduced lattice thermal conductivity. In view of abundantly available elements, good stability and high zT, FeNb1-xTixSb alloys can be great promising for high temperature power generation.

9:00 AM

(ICACC-S6-025-2018) Application of Ab initio Methods in the Development of Advanced Technical Ceramics (Invited)

J. Goldsby*[1]

1. NASA Glenn Research Center, Chemistry and Physics, USA

A significant need exists to develop materials not only capable of providing desired electronic and mechanical properties but also survival at extreme temperatures during service and device fabrication, such as co-firing. Computational methods offers an efficient and systematic manner to design new materials and guide their development. As an example computational -based material approaches can be used to determine the suitability of a given materials as a practical thermoelectric for energy harvesting. In this presentation examples are given of applications to relevant technical ceramics such as thermoelectrics, dielectrics, and magnetic systems. The calculation were based on density functional theory and carried out with norm conserving and projector augmented wave (PAW)

methods using commercial codes Materials Studio (Biovia, Inc) with the Cambridge Serial Total Energy Package (CASTEP) and MedeA (Materials Design Inc.) utilizing the Vienna Ab-initio Simulation Package (VASP) as the respective computational engines. This study makes predictions of relevant technical properties of ceramic materials

9:30 AM

(ICACC-S6-026-2018) Advances in Thermoelectric Complex Sulphides (Invited)

E. Guilmeau*[1]; C. Bourgès[1]; V. Kumar[1]; L. Paradis-Fortin[1]; P. Lemoine[2]; O. Lebedev[1]; T. Barbier[1]; B. Raveau[1]; B. Malaman[3]; G. Le Caer[4]; M. Ohta[5]; K. Suekuni[6]; A. Supka[7]; M. Fornari[7]; R. A. Al Orabi[7]

1. CNRS CRISMAT, France
2. Institut des Sciences Chimiques de Rennes (ISCR), France
3. Institut Jean Lamour, France
4. Institut de Physique de Rennes (IPR), France
5. National Institute of Advanced Industrial Science and Technology (AIST), Research Institute for Energy Conservation, Japan
6. Kyushu University, Department of Applied Science for Electronics and Materials, Interdisciplinary Graduate School of Engineering Sciences, Japan
7. Central Michigan University, Department of Physics and Science of Advanced Materials Program, USA

The design and optimization of thermoelectric (TE) materials rely on the intricate balance between thermopower (S), electrical resistivity (r) and thermal conductivity (k); perfecting such a balance is key to improve energy recovery systems and TE cooling devices. Complex copper sulfides can provide an eco-friendly high-performance low cost alternative by using elements that are abundant in naturally occurring minerals. Most of these materials exhibit low thermal conductivity possibly determined by local structural distortions, rattling phenomena, or strong bond anharmonicity. However, the improvement of the TE performances of these materials remains a challenge, due to the interdependent and contrary effects of their parameters S, r and k. The presence of structural defects, the deviation to stoichiometry, the nature of the chemical bonds, and of the distribution of charges in these complex structures are still a matter of debate, which is of capital importance for the optimization of their TE properties. In this respect, recent progress on some thermoelectric complex sulphides derived from mineral compounds (stannoidite, germanite, colusite) will be described. Synthesis, processing, shaping, as well as structural and microstructural features will be reported, together with electrical, thermal properties. Band structure and vibrational dispersions from first principles calculations will be discussed.

Thermoelectrics II

Room: Tomoka A

Session Chair: Anthony Powell, University of Reading

10:20 AM

(ICACC-S6-027-2018) Effect of cell size and basis set on the simulation of atomic dynamics in tetrehedrite thermoelectrics

J. Li[1]; W. Lai*[1]

1. Michigan State University, Chemical Engineering and Materials Science, USA

Cu12Sb4S13 tetrahedrite based materials are promising candidates as high-performance thermoelectrics containing environmentally friendly and earth-abundant elements. Understanding its atomic dynamics that contributes to its low lattice thermal conductivity is essential to improve its performance and provide guidelines to discover other promising thermoelectric materials. First-principles molecular dynamics is an indispensable tool to reveal atomic-scale details of dynamics of different species in complex materials. However, the effect of size of the simulation cell and basis set on the simulated atomic remains elusive. In this talk, we will discuss our results on the investigation of two different cell sizes (1x1x1 cell with 58 atoms vs 2x2x2 cell with 464 atoms) and two different basis sets (plane wave vs atomic orbital). We will compare various structural, dynamic, and thermodynamic properties such as vibrational density of states, heat capacity, EXAFS, etc.

10:40 AM

(ICACC-S6-028-2018) Praseodymium Telluride: A new high ZT, high temperature material

S. Bux*[1]; D. A. Cheikh[2]; B. E. Hogan[1]; B. Dunn[2]; T. Vo[1]; J. Fleurial[1]

1. Jet Propulsion Laboratory/California Institute of Technology, USA
2. University of California, Los Angeles, Materials Science, USA

Lanthanum telluride ($La_{3-x}Te_4$) has recently emerged as a high efficiency thermoelectric material. The performance of the $La_{3-x}Te_4$ system stems from a complex thorium phosphide (Th_3P_4) crystal structure resulting in an inherently low lattice thermal conductivity with tunable electronic properties. By optimizing the La:Te ratio (La vacancy ratio), the ZT of this system can attain values of 1.2 at 1275K. Computational modelling indicates that the conduction band states are dominated by the La d-orbitals. Praseodymium telluride ($Pr_{3-x}Te_4$) is a $La_{3-x}Te_4$ analogue and density functional theory (DFT) calculations indicate that it may have a sharp peak in the conduction band near Fermi level from the f-electrons. We utilized the mechanochemical approach to synthesize $Pr_{3-x}Te_4$ with various vacancy concentrations. The powders were compacted using spark plasma sintering (SPS) and the compacts were characterized using X-ray diffraction, scanning electron microscopy, and electron microprobe analysis. The temperature dependent electrical resistivity, Seebeck coefficient, and thermal conductivity will be presented. Preliminary data indicates that there is a large increase in the Seebeck coefficient from the f-orbitals, resulting in increased ZT values of 1.7 for $Pr_{3-x}Te_4$ at 1275 K with optimized vacancy concentrations.

11:00 AM

(ICACC-S6-029-2018) Correlating the process, structure and thermoelectric properties of thermal spray synthesized sub-stoichiometric TiO_{2-x} deposits

H. Lee*[1]; R. Chidambaram Seshadri[1]; S. Sampath[1]

1. Stony Brook University, Materials Science and Engineering, USA

Thermoelectric properties of plasma spray synthesized sub-stoichiometric Titania were investigated from RT to 750 K. TiO_{2-x} deposits are formed through the in-situ reaction of the $TiO_{1.9}$ or $TiO_{1.7}$ feedstock within the high-temperature plasma flame and manipulated through the introduction of varying amounts of H_2 in the plasma. Although the TiO_{2-x} particles experience reduction within the plasma, it can also re-oxidized through interaction with the surrounding ambient atmosphere. These deposits predominantly contain rutile phase with the presence of Magneli phases especially under significantly reducing plasma conditions. XRD results show a pattern indicating that higher H_2 in the plasma torch leads to greater decomposition and formation of the Ti_4O_7 phase which is known to have higher electrical conductivity but comes with a concomitant loss in Seebeck coefficient. From the result, keeping the original phase and reducing decomposition is beneficial to retain the high Seebeck coefficient in the TiO_2 system. The process attributes that lead to this is at low or no H_2 flow in the plasma and lower overall particle temperature. Despite the process condition, the deposits were comprising of well-bonded splats resulting in reasonable electrical conductivities while it has sufficient nano scale interfaces to retain relatively low thermal conductivity.

S7: 12th International Symposium on Functional Nanomaterials and Thin Films for Sustainable Energy Harvesting, Environmental, and Health Applications

Integration of Functional Metal Oxide Nanostructures in Devices

Room: Coquina Salon C

Session Chairs: Yoon-Bong Hahn, Chonbuk National University; Mauro Epifani, CNR-IMM

8:30 AM

(ICACC-S7-026-2018) Thin Films of Carbides and Carbon-based Nanocomposites (Invited)

W. Gulbinski*[1]

1. Koszalin University of Technology, Depertment of Technical Physics and Nanotechnology, Poland

Binary, ternary and more complex carbides form a group of compounds offering diverse, sometimes unique mechanical, tribological and electrical and thermal properties. They are main constituents of advanced cutting tools, high-power electronics or high temperature ceramics. These materials gain new properties when deposited in thin film form. In particular use of PVD methods, where coatings are grown in conditions which are far from thermodynamic equilibrium, opens new possibilities for design of carbon-based composites and fine tuning their properties. Thin films of binary metal carbides and carbon-based nanocomposites derived from them had found numerous applications as wear resistant and selflubricating coatings. Addition of third element has led to formation of nanocomposite structures where release of free amorphous carbon can be controlled to meet various application needs. In the talk properties and structural features of binary and ternary carbides or carbide-amorphous carbon thin film composites are systematically reviewed and discussed. Furthermore, plasma based deposition methods and conditions are presented.

9:00 AM

(ICACC-S7-027-2018) Electrochemical fabrication of nanostructured thin-film for renewable energy applications (Invited)

Y. Yang*[1]

1. University of Central Florida, NanoScience Technology Center, USA

Freestanding and robust thin-film electrodes are the basis for future renewable energy technology owing to their unique merits: i) additive-free features enable facile fabrication route and long lifetime; ii) can be easily recycled; iii) significant reduce the weight and volume of the energy devices, especially for batteries and supercapacitors; iv) can be integrated into wearable electronic systems. However, previous efforts fail to make significant progress in freestanding metal compound thin-films due to a lack of fundamental understanding and technical breakthrough in freestanding metal compound thin-films. In this work, transformtive thin-film nanomanufacturing technology has been developed to fabricate freestanding metal compound thin-films using a facile and scalable electrochemical route. Both experimental and computational studies indicate that a supreme electrochemical performance can be achieved by tuning the microsturcture and composition of thin-films. Outstanding energy storage and hydrogen generation properties are therefore obtained by directly using these advanced thin-films as electrodes.

9:30 AM

(ICACC-S7-028-2018) Sr doping in perovskite oxides $LaCrO_3$ and $LaFeO_3$ for controlled functionalities (Invited)

Y. Du*[1]; S. Spurgeon[1]; L. Wang[1]; H. Zhang[2]; D. Wu[3]; M. Bowden[1]; K. Stoerzinger[1]; P. Sushko[1]; S. Chambers[1]

1. PNNL, USA
2. Xiamen University, China
3. Nanjing University, China

Perovskite oxides exhibit a diverse range of properties and play critical roles in energy conversion and storage applications. In our research, we focus on the deposition of structurally and compositionally well-defined perovskites to understand their intrinsic electronic, magnetic, and optical properties. In this talk, I will discuss our efforts in fabricating and characterizing epitaxial Sr doped LaCrO3 (LSCO) and LaFeO3 (LSFO) films. Aliovalent doping of Sr introduces holes into the Cr3d (Fe3d) t2g-derived top of the valence band, resulting in enhanced conductivity, and reduction in bandgap. At low x, LSCO is an excellent p-type transparent conducting oxide, which can form a coherent p-n junction with NbSTO substrate with type II staggered band alignment. LSCO becomes metallic above x = ~0.6. A facile, reversible topotactic phase transition between SrCrO2.8 and SrCrO3 has been realized by controlling the annealing temperature and oxygen partial pressure. In contrast, an insulator-to-metal transition has not been observed in LSFO. The LSFO/NbSTO heterojunctions appear to be affected more by O vacancies, and exhibit resistive switching behavior. To take full advantage of such materials, it is essential to characterize individual defects and extended defect structures, understand their origin and the nature of interaction with internal interfaces, and to develop protocols for their control.

Metal Oxide Nanostructures for Chemical and Biological Sensors

Room: Coquina Salon C

Session Chairs: Witold Gulbinski, Koszalin University of Technology; Yang Yang, University of Central Florida

10:20 AM

(ICACC-S7-029-2018) Metal-oxide Nanostructures Based Chemical and Biological Sensors for Environmental and Biomedical Applications (Invited)

Y. Hahn*[1]

1. Chonbuk National University, School of Chemical Engineering, Republic of Korea

It is well recognized that abnormal levels of glucose, choresterol and uric acid in biological fluids, and nitrite and potassium ions in water affect millions of people worldwide causing several diseases and disorders in human body as well as in environments. Thus, there is a high need to detect different analytes routinely for disease screening, monitoring and treatment. Nanotechnology revolution has led to the fabrication of nanosensor devices for rapid and specific identification of chemical and biological species. However, the development of multiplexed nanoscale sensors for simultaneous, selective detection of different analytes still remains a major challenge at the nanotechnology frontier. In this lecture, the synthesis of functional metal oxide nanomaterials and their applications for chemical and biological sensors will be presented in terms of electrochemical sensors and field-effect transistors array sensors.

10:50 AM

(ICACC-S7-030-2018) Controlled Processing of Sol-Gel Precursors for the Synthesis of W and Mo Oxides and their Combination with Titania Nanocrystals (Invited)

M. Epifani*[1]

1. CNR-IMM, Italy

One of the reasons of the success of chemical processing of materials is the range of synthetic possibilities allowed by the control of the molecular structure of the precursor. For instance, hydrolytic processing may allow 3D development of the material structure, through progressive inorganic cross-linking. On the other hand, if the cross-linking is constrained, suitable processing will then result in different materials typologies, depending on the allowed growth degrees of freedom. These general considerations have different outcomes, depending on the actual reactivity of the considered precursor. In this work, the exploitation of the chemical properties of simple W and Mo chloroalkoxide precursors will be illustrated for the synthesis of different WO_3 and MoO_2 structures. While simple powders and thin films are common in sol-gel chemistry, it is possible to go further in the range of obtainable structures by more advanced processing. Monoclinic WO_3 and MoO_2 naked nanocrystals can be prepared by solvothermal processing in suitable conditions. Finally, layers of WO_x and MoO_x species can be deposited onto TiO_2 nanocrystals, resulting in radical modification of the naked anatase nanostructures. The gas-sensing properties of the resulting materials will be reviewed, enhanced by the peculiar surface chemistry induced by the chemical processing.

11:20 AM

(ICACC-S7-031-2018) Surface modification of magnetic nanoparticles for enhanced hyperthermia cancer therapy (Invited)

H. Srikanth*[1]

1. University of South Florida, Physics, USA

Magnetic nanoparticles are being developed for applications ranging from high density recording, spintronic devices to nanomedicine. Magnetic hyperthermia is one of the most promising new techniques for cancer treatment. Although spherical iron oxide nanoparticles are commonly studied for a variety of biomedical applications, their relatively small saturation magnetization and small effective anisotropy result in a relatively low heating efficiency or Specific Absorption Rate (SAR). In order to boost the heating efficiency of iron oxide nanoparticles, different options have been considered, such as increasing their saturation magnetization, changing their size, shape, anisotropy etc. In this talk I will present our strategies to tailor the surface of nanoparticles and demonstrate that core-shell structures, high aspect ratio nanorods and exchange coupled hybrid nanoparticles have enhanced SAR. There is a need to improve the specific absorption rate (SAR) and heating efficiency of nanoparticles for hyperthermia and at same time retain the biocompatibility afforded by iron oxide. Overall our recent work has highlighted the role of surface and interfacial anisotropy in magnetic nanoparticles and their direct influence on heating efficiency.

11:40 AM

(ICACC-S7-032-2018) Controlling texture in lead-free KNN ferroelectric thin films by aqueous chemical solution deposition

N. H. Gaukås*[1]; S. M. Dale[1]; J. Glaum[1]; M. Einarsrud[1]; T. Grande[1]

1. Norwegian University of Science and Technology, Department of Materials Science and Engineering, Norway

Here we report on the control of texture and microstructure of KNN thin films by aqueous chemical solution deposition (CSD) by spin coating on single crystal STO. Two different aqueous precursor solutions were developed, differentiated by the complexing agent for Nb, continuing our previous work on bulk Nb-based materials. Thermogravimetric analysis of the hybrid gels formed from the two precursor solutions demonstrates fundamentally different thermal decomposition kinetics. The decomposition plays a crucial role in the promotion of either homogeneous or heterogeneous nucleation of KNN and also parasitic secondary phases, yielding polycrystalline or textured films, respectively. Characterization of the thin films using different methods proved that gel decomposition dynamics and nucleation kinetics are closely linked, and that texturing in the thin films can be tuned by alternation of the precursor solution chemistry.

S8: 12th International Symposium on Advanced Processing and Manufacturing Technologies for Structural and Multifunctional Materials and Systems (APMT12)

Unique Processing II

Room: Coquina Salon A

Session Chairs: Eugene Medvedovski, Consultant; Vojislav Mitic, Serbian Academy of Sciences

8:30 AM

(ICACC-S8-024-2018) Boron Nitride–Based Coatings Obtained through Thermal Diffusion Process for Friction and Corrosion Applications (Invited)

E. Medvedovski*[1]

1. Endurance Technologies Inc., Canada

A novel approach of the BN-based coating formation through the thermal diffusion process is presented. The process includes the formation of gaseous B- and N-based species due to high temperature decomposition of the B- and N-rich powders with consequent deposition of B and N onto heated metallic or metal-ceramic substrates. Consequent diffusion of B and N to the substrate and their interaction result in the formation of the BN-based layer. The coatings can be used for the friction and corrosion-related applications. The process is versatile, suitable for different size and shape components and does not require complicated agreement.

9:00 AM

(ICACC-S8-025-2018) Improvement of high-temperature characteristics in WC-FeAl hard materials

R. Furushima*[1]; K. Shimojima[1]; H. Hyuga[1]

1. National Institute of Advanced Industrial Science and Technology (AIST), Structural Research Insititute, Japan

WC-Co is a famous hard material having superior mechanical properties such as hardness, strength and toughness. However, these properties degrades at high temperatures more than 600 degrees Celsius. Combination of WC and intermetallic compound having good heat resistance is one of keys to solve that problem. In this study, we focused on WC-FeAl composites and tried to improve their high-temperature characteristics such as mechanical properties and oxidation resistance. Oxygen is one of key element to improve those properties. Here, we introduce some examples, which indicate the importance of oxygen control to improve the mechanical properties and oxidation resistance of WC-FeAl. Here, we introduce some examples, which indicate oxygen improved the mechanical properties and oxidation resistance of WC-FeAl.

9:20 AM

(ICACC-S8-026-2018) Fabrication and Optical Properties of Highly Transparent MgO Ceramics

X. Chen*[1]; Y. Wu[1]

1. New York State College of Ceramics at Alfred University, Department of Materials Science and Engineering, Kazuo Inamori School of Engineering, USA

Highly transparent MgO ceramics have been prepared via Spark Plasma Sintering (SPS) at 1200 °C for 10-60 min, under a pressure of 100 MPa. All of the prepared ceramics were visually transparent and exhibited relatively high in-line transmittance. The ceramic sintered for 60 min demonstrated the highest in-line transmittance, of 60-80% in the 300 nm to 9 μm wavelength range; values that are close to that reported for MgO single crystals. The average grain size of the sample sintered for 60 min was measured by SEM to be ~144 μm. Photoluminescence measurements revealed a peak located at ~360 nm, which was determined to be due to oxygen vacancy color centers induced by the reducing atmosphere of the SPS apparatus. Results are presented with an emphasis on potential laser applications, based on the unique thermal properties of the MgO host material.

9:40 AM

(ICACC-S8-027-2018) Combustion synthesis of boron nitride via magnesium reduction using additives

S. Chung*[1]

1. National Cheng Kung University, Department of Chemical Engineering, Taiwan

This study is aimed at enhancing the product yield in combustion synthesis of h-BN using Mg reduction of B2O3 as the boron source under a low N2 pressure by using additives. The reactant powders and the additives were placed in perforated aluminum containers without pressing. Due to a loose and highly porous structure of the powder stack, the surrounding N2 can penetrate easily into it and the N2 generated by NaN3 or C3H6N6 escapes easily, addition of these two additives only increases slightly the product yield in the low content region but decreases the product yield in the high content region due to decreasing temperature. Addition of inert particles(i.e., MgO or BN)increases the product yield only when the temperature is higher than the melting point of boron, under which coalescence of molten boron is suppressed due to capillary spreading of the molten boron on the particles. When the temperature is lower than the melting point of boron, addition of the inert particles decreases the product yield because of their cooling effect. NH4X (X¼Cl orBr) was found the most effective in enhancing the product yield because it creates an easier route for the nitridation of boron by first converting boron to BXx, which then reacts with N2 under the reduction of H2. A product yield of 67% was achieved by simultaneous addition of NH4Cl and BN under a N2 pressure of 1.6MPa.

10:20 AM

(ICACC-S8-028-2018) Advanced Powder Processing for High-Performance Thermoelectric Materials (Invited)

J. Li*[1]

1. Tsinghua University, School of Materials Science and Engineering, China

Thermoelectric materials that can be used for direct conversion between heat and electricity have drawn worldwide interests for decades. A good thermoelectric material must have high Seebeck coefficient, good electrical conductivity and low thermal conductivity. However, it is very difficult to control the above three parameters independently, which often counter each other. Last two decades have witnessed significant progress in thermoelectric research, to which materials processing has crucial contributions. Compared with traditional zone-melting method used for fabricating bismuth telluride alloys, new powder-based processes have more freedom for manipulating nanostructures and nanocomposites, leading to thermoelectric performance enhancement. In particular, spark plasma sintering is increasingly used for synthesizing fine-grained thermoelectric bulk materials with reduced thermal conductivity. By focusing on three representative examples, this talk will show the processing importance for thermoelectric materials.

10:50 AM

(ICACC-S8-029-2018) Water vapor-assisted solid-state synthesis and particle shape evolution of ceramic powders

T. Kozawa*[1]; K. Yanagisawa[2]

1. Osaka University, Joining and Welding Research Institute, Japan
2. Kochi University, Japan

Among the synthetic methods for ceramic powders, a solid-state reaction between oxides and/or carbonates is widely accepted as a simple and low-cost method. However, this method typically produces coarse and agglomerated particles due to the high-temperature and long-time heating. It is also difficult to control particle morphologies. Recently, we found that the solid-state reaction was accelerated by introducing of water vapor. The formation rate of various alkaline-earth titanates, silicates, and zirconates in water vapor was faster than in air. In addition, the reaction in water vapor promotes the particle growth and provides different particle morphology from that in air. The solid-state reaction in water vapor is a simple and valuable method for the large-scale production of ceramic powders at a low temperature, where the shape, size, and microstructure can be controlled.

11:10 AM

(ICACC-S8-030-2018) Facile one-step high-temperature spray pyrolysis route toward ultrafine metal carbide nanocrystalline powders

J. Xing*[1]; P. Foroughi[1]; A. E. Behrens[1]; Z. Cheng[1]

1. Florida International University, Mechanical and Materials Engineering, USA

Fine ultra-high temperature ceramic (UHTC) powders have found very important applications in many fields. In this work, a facile high-temperature spray pyrolysis (HTSP) approach is adopted for the synthesis of UHTCs of metal carbides such as tantalum carbide (TaC), hafnium carbide (HfC). It is proposed that, during the HTSP, the precursor solution droplets would continuously undergo rapid drying, thermolysis, and rapid (within ~1 min) in-situ carbothermal reduction (CTR) process, and finally give rise to metal carbide powders. The as-obtained materials are shown as uniform and well-separated nanoparticles with intensive metal carbide XRD peaks, and from TEM images, carbide nanocrystalline particles embedding in carbon can be found. The solvent that was used for the preparation of precursor solution could largely influence the formation of metal carbide through its carbon to oxygen atomic ratio, boiling point, and viscosity etc. Mixing of ethanol with other organic solvents, like 1-pentanol and tri-n-butyl borate, is proved to be helpful for the metal carbide formation. In addition, a suitable conducting temperature or gas flow rate is also very important for the preparation of metal carbide through HTSP. The HTSP method developed in this work is simple, low-cost and efficient, hence it can potentially be employed for the large-scale fabrication of fine UHTC powders.

11:30 AM

(ICACC-S8-031-2018) Fractals Nature and Nano-micro Structure within the Energy Frontiers

V. Mitic*[1]; L. Kocic[3]; V. Paunovic[3]; B. Vlahovic[4]; S. Tidrow[2]; H. Fecht[5]

1. Serbian Academy of Sciences, Institute of Technical Sciences, Serbia
2. Alfred University, USA
3. University of Nis, Serbia
4. North Carolina Central University, USA
5. University of Ulm, Germany

One possible line developed by the authors as the new model using fractal nature of electronics ceramics materials. Using the new experimental-theoretical approach frame, a new topic is developed known as electrochemistry area of fractal microelectronics. As illustration, note that both energy and fractality is omnipresent, unlimited entity concerning space and time. Many evidences that the whole Universe is permeated by fractal structures as well as by energetic fields a systematic approach to create the method for the wind motion and turbulences, prediction the fractal nature of influence. Besides, both entities, energy and fractality exist in micro and macro world. Any fractal is dividable down to nano-scale as well as the energy field. In the case of porous ferroelectric ceramics, the combination of grains and pores constitutes a specific fractal micro-nano structure that can store energy as micro-electric charges. In this way, we confirmed the new fractal frontiers in the area of alternative energy sources, what is very new, precise and powerful approach. The concept design main goal is to reach the inventive ideas for final products with best performances. Through this method it is introduced the new intergranular thin film's fractal nature microelectronics, from the one aspect, and also opening the new "window" towards universality of relationship between forms and energy in the broad sense.

S9: Porous Ceramics: Novel Developments and Applications

Innovations in Processing Methods and Synthesis of Porous Ceramics I

Room: Coquina Salon G
Session Chairs: Paolo Colombo, University of Padova; Manabu Fukushima, National Institute of Advanced Industrial Science and Technology (AIST)

8:30 AM

(ICACC-S9-001-2018) How to engineer porous non-oxide ceramics at various length scales via precursor chemistry? (Invited)

S. Bernard*[1]

1. CNRS, Ceramic Research Institute, France

Design of porous ceramics at various length scales has induced important applications in environmental and green technologies. In such materials, the macropores reduce flow resistance, allowing improvement of the mass transfer into and out of the network, whereas the large surface area of the micro-/mesopores provides selectivity and active sites. Common oxide ceramics have largely dominated the porous material field. In contrast, non-oxide ceramics are significantly less investigated most probably because their strong and predominantly covalent atomic bonds impose high temperature solid-state sintering usually non compatible with the design of well-defined porous materials. The Polymer-Derived Ceramics (PDCs) route based on precursor chemistry may significantly reduce the synthesis temperature and offers the possibility to develop materials bearing tailored porosity through its combination with a pore forming approach. In this talk, we will review the recent strategies developped in the litterature, including materials prepared by our group, to design Si-based non-oxide ceramics with tailored porosity; in particular those holding hierarchical porosity. This will be followed by an overview concerning the applications of these materials. Lastly but not least, some issues that need to be clarified in the near future are proposed in the conclusion.

9:00 AM

(ICACC-S9-002-2018) Synthesis and properties of multiscale porous TiC/SiC ceramics

A. J. Baux*[1]; L. Nouvian[1]; S. Jacques[1]; T. Piquero[2]; D. Rochais[2]; G. Chollon[1]

1. LCTS - CNRS, France
2. CEA, Ripault, France

Open porosity cellular ceramics have a great potential for energy conversion. In spite of their tolerance to mechanical damage, structural applications at high temperature are still limited due to high production costs or properties below requirements. One objective of this work was to investigate a simple manufacturing route, based on the conversion of lignocellulosic materials into porous ceramics, for use as volumetric solar receivers. Balsa wood is selected as the precursor. An original aspect of this work is to create macroporosity in balsa wood, by laser or water jet cutting, in addition to the natural microporosity. Wood specimens of various pore sizes and volume fractions were first pyrolysed. Except shrinkage due to carbonization, the architecture of balsa is retained. The obtained carbon materials were then fully converted into TiC by Reactive Chemical Vapor Deposition using a $TiCl_4/H_2$ source gas. TiC ceramics were finally reinforced by standard CVI of SiC from CH_3SiCl_3/H_2. The 3D multiscale porosity of the TiC and TiC/SiC specimens was analyzed by Scanning Electron Microscopy. Mechanical and thermal properties were examined as well as the oxidation resistance. Although restricted to macropores and near-surface micropores, the SiC coating on the TiC structures was found to strongly improve the crushing strength, heat conductivity and oxidation resistance.

9:20 AM

(ICACC-S9-003-2018) Effect of thermal conductivity of powder compacts on porous structure in direct-foaming method

A. Shimamura*[1]; M. Hotta[1]; N. Kondo[1]

1. National Institute of Advanced Industrial Science and Technology (AIST), Japan

Effect of particle size and thermal conductivity of powder compacts on porous structures in direct-foaming method was investigated in this study. Several ceramics powder, such as silica, cordielite and alumina powder, were used as raw powder in order to controle the thermal conductivity of powder compacts. These ceramics powders were mixed with the resole type phenolic resin powder as the both of binder and thermal foaming agent in dry-condition. Foamed compacts were obtained by pressed molding following by thermal foaming process at 150 °C. Porosity of foamed compacts increased with increased thermal conductivity. Furthermore, the effect of particle size of raw powders on the porous structure was also investigated. Obtained foamed compacts were converted to porous ceramics by debinding and sintering process. The porosity, mechanical strength and microstructural observations of porous ceramics prepared were characterized.

9:40 AM

(ICACC-S9-004-2018) Processing of Eelectrospun Ceramic Nanofiber Mats

O. Elishav[1]; V. Beilin[1]; G. s. Shter[1]; G. Grader*[1]

1. Technion - Israel Institute of Technology, Israel

Nanofibers with desired architecture and properties have been investigated for improving and developing new applications. Electrospinning is a simple approach to produce polymer, ceramic and composite fibers of various diameters and morphologies. Electrospun ceramic fibers can be utilized in applications such as energy harvesting, catalysis and sensing devices. The electrospinning

solution includes at least one or more polymers, solvents as well as ceramic/metal oxides/metal precursor. Usually, the electrospinning stage is followed by polymer removal by thermal treatment. This step often includes shrinkage and potential deleterious deformation. Therefore, understanding the shrinkage origin and minimizing its negative effects on the electrospun fiber mats is highly important. The shrinkage mechanism during thermal treatment was investigated in three different electrospun systems: PZT/PVP, pure PVP and Al_2O_3/PVP. During electrospinning the fibers undergo tremendous stretching aligning the polymer chains along the fiber axis. However, during the thermal treatment the polymer relaxes leading to substantial shrinkage, particularly above the polymer glass transition temperature. Understanding the interplay between the precursor solution composition, the fibers morphology and the mats thermal shrinkage enables process optimization that minimizes the fiber mat deformation during thermal treatment.

Innovations in Processing Methods and Synthesis of Porous Ceramics II

Room: Coquina Salon G
Session Chair: Samuel Bernard, CNRS

10:40 AM

(ICACC-S9-005-2018) Processing and performance evaluation of porous carbon material (Invited)

R. Inoue*[1]; Y. Kogo[1]

1. Tokyo University of Science, Japan

Porous carbon materials (PCMs) have been expected for applying the component of thermal protection system, called ablators. Carbon foams were considered as one of preform materials for ablators, however, polymers, i.e., phenolic resin, polyimide, and PMMA, could not be impregnated. The precursor materials should have open-cell structure as well as high porosity. In addition, mechanical integrity is required because the component is subjected to dynamic pressure at quite high temperature. In this presentation, we develop a new type of PCM with interconnected pillar structure obtained by carbonization of porous phenolic resin. The precursor material was fabricated by phase separation technique. Then, microstructural characteristics and mechanical properties of developed material were also explained. Modeling and characterization of microstructure was also carried out by X-ray computed tomography and focused ion beam-SEM tomography. Elastic properties of developed material were discussed based on homogenization analysis using 3D model and experimental results. The advantage of network structure will be discussed based on experimental and numerical analysis.

11:10 AM

(ICACC-S9-006-2018) Aligned Continuous Cylindrical Pores Derived from Electrospun Polymer Fibers in Titanium Diboride

D. C. Hicks*[1]; Z. Zhou[2]; G. Liu[2]; C. Tallon[1]

1. Virginia Tech, Materials Science and Engineering, USA
2. Virginia Tech, Chemistry, USA

The use of electrospun polystyrene to create long range, continuous, ordered multi-scale porous structures in Titanium Diboride (TiB_2) has been explored. The introduction of electrospun polystyrene fibers as a sacrificial filler into a colloidal suspension of TiB_2 allows for easy control of pore size, volume percent porosity, and long range ordered pore structures within the sintered ceramic. Green bodies were formed by vacuum infiltrating an electrospun-fiber-filled mold with the colloidal TiB_2 suspension. The size, volume, uniform distribution, and dispersion of the pores was optimized by carefully selecting the fibers material, its diameter, the solvent type and the solid content of TiB_2. Samples were partially sintered at 2000° C in argon to form a multiscale pore structure. Continuous aligned cylindrical pores from the fibers (5–10 μm) and random porosity between the ceramic particles from partial sintering (300–900 nm) were revealed under the SEM. TiB_2 near-net-shaped parts with this multi-scale porosity and porosities from 50% to 70% were successfully cast and sintered. This low cost processing technique facilitates the production of thermally and mechanically anisotropic structures into near-net shape parts, for extreme environment applications, such as ultra high temperature insulation.

11:30 AM

(ICACC-S9-007-2018) Controllable Morphology of Multi-Scale Porous Titanium Dioxide

N. Zahed*[1]; J. Foster[1]; C. Tallon[1]

1. Virginia Polytechnic Institute and State University, Materials Science and Engineering, USA

Titania (TiO_2) samples exhibiting a multi-scale pore size distribution were prepared by a facile, rapid technique via hydrolysis of titanium isopropoxide using H_2O/Ti ratio ranging from 5:1 to 60:1, at three different drying rates, and at various percentages of isopropanol as solvent. The effect of these parameters on the pore size, pore size distribution, particle size, morphology, and crystalline phase was systematically studied. Scanning Electron Microscopy reveals the synthesis of nanometric particles on the order of 20nm assembled into structures with macropores and macrochannels on the order of 1-2 μm as well as pores around 20nm in between particles. Hydrolysis in excess H_2O increased the frequency of the macropores and decreased the distance between them, while the introduction of isopropanol dramatically decreased the presence of the macropores, but rendered more homogeneous microstructures. The nanometric particles obtained after thermal treatment at 450°C exhibited anatase phase with good crystallinity. The results from these studies are aligned to identify the optimal synthesis settings for achieving highly porous, self-assembled titania mesostructures whose high surface area promises superior performance as an electrode material in Li-ion batteries or an efficient hydrogen storage material.

11:50 AM

(ICACC-S9-008-2018) Design of Superhyrophobic Conductive Ceramic Fibers

P. Taheri*[1]

1. University of Texas Arlington, Chemistry, USA

Electrospun preceramic Polymethylhydrosiloxane (PMHS) and Divinylbenzene (DVB) with either Poly (methyl methacrylate) (PMMA), Polyvinylpyrrolidone (PVP), or Polystyrene (PS) as the carrier polymer were fabricated followed by annealing to make nanoporous silicon oxycarbide fibers (SiCO). Their fiber morphologies and atomic compositions differed due to their thermal behaviors and decomposition pathways of each carrier polymer. The chemical compositions and structures of the ceramic fibers were characterized by SEM-EDS, XRD and FTIR and the average pore size, cumulative pore volume, and the BET surface area were measure by Porosimetry. The superhydrophobic property of the fibrous mats was studied and showed their potential ability to absorb oils and organic chemicals, which can be used for the application such as marine oil-spill recovery and environmental remediation and also they were rich in Carbon content, which can be used as a conductive material.

S12: Advanced MAX/mxene Phases and UHTC Materials for Extreme and High Temperature Environment

Novel Synthesis Paradigm and Unique Properties of Mxenes-II

Room: Tomoka B
Session Chairs: Geetu Sharma, University of California, Davis; Deniz Cakir, University of North Dakota

8:30 AM

(ICACC-S12-028-2018) Highly Stretchable and Bendable Conductive MXene Multilayers

H. An*[2]; T. Habib[2]; S. Shah[2]; H. Gao[1]; M. Radovic[3]; M. Green[2]; J. Lutkenhaus[2]

1. Texas A&M University, Department of Mechanical Engineering, USA
2. Texas A&M University, Artie McFerrin Department of Chemical Engineering, USA
3. Texas A&M University, Department of Materials Science and Engineering, USA

Stretchable, bendable, and foldable conductive coatings are crucial for wearable electronics and biometric sensors. Such coatings should maintain functionality while simultaneously interfacing with different types of surfaces undergoing mechanical deformation. MXene sheets as conductive 2D nanomaterials are promising for this purpose, but it is still extremely difficult to form surface-agnostic MXene coatings that can withstand extreme mechanical deformation. Herein, we demonstrate conductive and conformal MXene multilayer coatings that can undergo large-scale mechanical deformation while maintaining a conductivity as high as 1250 S/m. MXene multilayers are successfully deposited onto flexible polymer sheet, stretchable poly(dimethylsiloxane), nylon fiber, glass, and silicon. The coating shows a recoverable resistance response to bending (up to 2.5 mm bending radius) and stretching (up to 40% tensile strain). We demonstrated that the MXene multilayer films could be used as strain sensors to topographically sense objects or materials deformation.

8:50 AM

(ICACC-S12-029-2018) Multiscale Damping Properties and Mechanisms of 2D Layered MXene

A. Loganathan*[1]; P. Nautiyal[1]; B. Boesl[1]; A. Agarwal[1]

1. Florida International University, Materials Engineering, USA

Layered 2D MXene has been the fervent topic of interest in the field of energy materials. However, the mechanical properties of MXene phase are poorly understood. In the present work, nanomechanical and multiscale damping properties of MXene phase are investigated as a function of HF treatment period. Nanomechanical properties of MXene were examined using quasi-static nanoindentation and damping behavior was studied at multiscale with loads varying from mN to μN. MXene showed a superior damping behavior (200% more than pure MAX phase) which is ascribed to interplay of multiple mechanisms related to structure of MAX and MXene.

9:10 AM

(ICACC-S12-030-2018) Transparent, conductive solution-processed 2D MXene films

G. Ying*[1]; A. D. Dillon[2]; D. Zhao[1]; S. Mei[1]; S. Kota[1]; G. Michael[1]; C. Li[1]; A. T. Fafarman[2]; M. Barsoum[1]

1. Drexel University, Department of Materials Science and Engineering, USA
2. Drexel University, Department of Chemical and Biological Engineering, USA

MXenes - a new class of solution-dispersable 2D nanomaterials - are produced by selectively etching the A-layers from the MAX phases. Herein, we report on the fabrication of $Ti_3C_2T_x$, Ti_2CT_x and V_2CT_x transparent conductive films by spin coating from aqueous colloidal solutions. The electrical conductivities of $Ti_3C_2T_x$, Ti_2CT_x and V_2CT_x films are, respectively, 6450 S/cm, 5250 S/cm and 3380 S/cm. with optical transmittances that depend on thickness. The figures of merit (FOM) - defined as the ratio of the DC conductivity, σ_{DC}, to a quantity termed the 'optical conductivity', σ_{OP}, - derived from the optical transmittances of the transparent films are quite respectable. The ease by which these films can be deposited from aqueous colloidal solutions from inexpensive raw materials that are non-toxic at room temperature will have important technological implications that are touched upon.

9:30 AM

(ICACC-S12-031-2018) Theoretical investigations on the physical properties and fabrication mechanisms of MXenes

X. Zha*[1]; J. Zhou[1]; L. Feng[1]; P. Eklund[3]; J. Francisco[2]; Q. Huang[1]; S. Du[1]

1. Ningbo Institute of Materials Technology and Engineering, Chinese Academy of Sciences, China
2. Purdue University, USA
3. Linköping University, Sweden

As a new family of two-dimensional materials, MXene has aroused extensive attentions in recent years. Due to their outstanding absorbtion capacities and conductivities, MXenes were proposed to show widespread applications such as in energy storage and electromagnetic shielding. In this presetation, we systematically studied the structural, mechanical and electronic properties of the carbide MXenes firstly. It implied that the physical properties are significantly depended on the surface functional froups. Moreover, some MXene members show semiconducting characteristics. Based on above, we further investigated the possibility of using those semiconducting MXenes as the materials for semiconducting devices. The key properties of carrier mobility and thermal conductivity were studied. The results shown that the oxygen functionalized M_2CO_2 (M=Ti, Zr, Hf) possess relatively high hole mobilities which are of the order of 10^4 $cm^2V^{-1}s^{-1}$, while Sc_2CT_2 (T=F, OH) MXenes present favorable electron mobilities. The lattice thermal conductivities are significantly related to the metal atom M. Noteworthily, we have developed a new approach to synthesis MXenes cooperated with experiment. Instead of etching the traditional MAX phases, the layered ternary carbides $M_nAl_3C_{n+2}$ and $M_nAl_4C_{n+3}$ (n equals to 3 or 4) were adopted as precusors, and we have successfully synthesized the Zr- and Hf-containing MXenes.

UHTC Ceramic Matrix Composites

Room: Tomoka B
Session Chair: William Fahrenholtz, Missouri University of Science & Technology

10:20 AM

(ICACC-S12-032-2018) Ultra-high temperature ceramic matrix composites (UHTCMCs) (Invited)

J. Binner*[1]

1. University of Birmingham, United Kingdom

There is an increasing demand for advanced materials, for aerospace and other applications, with temperature capability ranging from 1500°C to well over 2000°C and able to survive highly corrosive environments whilst subject to intense heat fluxes and mechanical stresses. The interaction of environmental conditions together with the requirement that dimensional stability is maintained makes the selection of suitable materials extremely challenging. This paper discusses the design, development, manufacture and testing of a new class of ceramic matrix composites based on C fibre and SiC preforms enriched with ultra-high temperature ceramic (UHTC) powders and with a matrix infiltrated by either RF- or microwave-heated chemical vapour infiltration (CVI). These composites

will form of suite of materials suitable for application in severe aerospace environments.

10:50 AM

(ICACC-S12-033-2018) Tough salami-inspired UHTCMCs produced by electrophoretic deposition

P. Galizia*[1]; S. Failla[1]; L. Zoli[1]; D. Sciti[1]

1. ISTEC-CNR, Italy

One of the biggest challenges of the materials science is the mutual exclusion of strength and toughness. This issue was minimized by mimicking the natural structural materials. To date, few efforts were done regarding materials that should be used in harsh environments. In this work we present novel continues carbon fiber reinforced ultra-high-temperature ceramics matrix composites (UHTCMCs) for aerospace featuring optimized fiber/matrix interfaces and fibers distribution. The microstructures - produced by electrophoretic deposition of ZrB_2 on unidirectional carbon fibers followed by ZrB_2 infiltration and hot pressing - show a maximum flexural strength and fracture toughness of 330 MPa and 14 MPa $m^{1/2}$, respectively. Microstructures and properties are discussed and possible toughening mechanisms acting in this composite are illustrated.

11:10 AM

(ICACC-S12-034-2018) Thermo-mechanical behavior of novel UHTCMCs with a carbide matrix at temperatures above 1800°C

A. Vinci*[2]; L. Zoli[2]; D. Sciti[2]; W. Fahrenholtz[1]; J. Watts[1]; G. Hilmas[1]

1. Missouri University of Science & Technology, Dept. of Materials Science and Engineering, USA
2. CNR ISTEC, Dip. di Scienze Chimiche e Tecnologie dei Materiali, Italy

Although research on UHTCs has led to successful fabrication of high strength UHTCs, their low fracture toughness and thermal shock resistance remain major concerns for their application under extreme environments. The ceramic matrix composites (CMCs) currently used in aerospace applications, based on carbon or silicon carbide, display excellent thermo-mechanical properties but are not suitable for temperatures exceeding 1600°C due to oxidation of the fibers and the subsequent drop of mechanical properties. To overcome these limits, we are currently designing and manufacturing a new class of composites, the so called UHTCMCs, in order to combine the good fracture toughness and thermal shock resistance of CMCs with the oxidation and ablation resistance of UHTCs. The aim of this work is to study the thermal, electrical, and mechanical properties of carbon fiber reinforced UHTCs based on a ZrC, HfC and TaC ceramic matrix. Four-point bending tests were performed in an induction furnace with graphite test fixtures. Specimens have been cut from the sintered pellets and tested from room temperature to T > 1800°C. Results show an increase in flexural strength with the increase of temperature and a change in the mechanical behavior which was attributed to stress relaxation. Crack deflection is still present even at high temperature as confirmed by SEM analysis.

11:30 AM

(ICACC-S12-035-2018) New Avenues for Near-Net-Shaping of UHTCs

C. Tallon*[1]; S. Leo[2]; G. Franks[2]

1. Virginia Tech, Materials Science and Engineering, USA
2. The University of Melbourne, Department of Chemical and Biomolecular Engineering, Australia

Hypersonics and other extreme applications rely on components that often require a complex geometry and a fully dense, fine-grained microstructure. The current state-of-the-art processing of these materials allow to achieve that microstructure, but the shaping capability remains a challenge. Near-Net-Shaping Techniques involving the colloidal processing of UHTC powders can render near-net-shaped pieces, but because of the shape, the sintering needs to be done using pressureless conditions. This work will discuss, using zirconium diboride (ZrB_2) as a case study, the strategies to enhance the pressureless densification and complex shaping of these materials: i) particle packing in the green bodies (by the control of the interparticle forces in suspension), ii) sintering profiles and iii) incorporation of sintering aids, and how these results compare with the current state-of-the-art processing (hot-pressing). These strategies have allowed to reach 93% TD without sintering aids at 2100°C and 98%TD with sintering aids and optimized sintering profiles. These approaches have been further explored using a range of processing techniques such as slip casting, freeze casting and gelcasting, and other materials such as boron carbide (B_4C) and titanium diboride (TiB_2). The promising results open the path to use this approaches in the next generation of advanced and additive manufacturing technologies and multi-material compositions.

S13: Advanced Ceramics and Composites for Nuclear Fission and Fusion Energy

Novel Ceramics, Radiation Effects II

Room: Coquina Salon H
Session Chair: Lance Snead, ORNL

8:30 AM

(ICACC-S13-027-2018) Neutron Irradiation of Ti_3SiC_2 and Ti_3AlC_2 (Invited)

M. Barsoum*[1]

1. Drexel University, Materials Science and Engineering, USA

The impetus for this work was our conjecture – proven herein - that the presence of the A-layers in the MAX phases would result in extraordinary damage tolerance to neutrons. The focus here is on Ti_3SiC_2 and Ti_3AlC_2 After irradiation to 0.14 dpa at 121 °C and 735 °C, black spots are observed in both ternaries. After irradiation to higher doses, at 735 °C, basal dislocation loops, with a Burgers vector of b = ½ [0001] are observed in Ti_3AlC_2 with loop diameters of the order of the order of 25 nm. In Ti_3AlC_2 larger dislocation loops (75±34 nm) are observed after 3.4 dpa at 735 °C, in addition to stacking faults. In sharp contrast to the MAX phases, the TiC impurity particles in the former form extensive dislocation loops at all conditions. Voids are observed at grain boundaries and within stacking faults after 3.4 dpa irradiation, with extensive void formation in the TiC regions at 1085 °C. Most remarkably, defect free, denuded zones, of the order of 1 μm, were observed after irradiation to 3.4 dpa at 735 °C. Small grains, (3-5 μm) were damage free after irradiation at 1085 °C to this dose. When these results are compared to those of TiC, there is little doubt that the A-layers provide enhanced neutron irradiation tolerance. Based on these it is reasonable to conclude that Ti_3SiC_2 is quite a promising candidate for high (> 500 °C) temperature nuclear applications. At lower temperatures anisotropic swelling results in microcracking.

9:00 AM

(ICACC-S13-028-2018) Low-Temperature Processing and Consolidation of Hydroxyapatite

M. ul Hassan*[1]; H. Ryu[1]

1. Korea Advanced Institute of Science and Technology (KAIST), NQE-NFML, Republic of Korea

One of the issues being faced by used nuclear fuel reprocessing is immobilization of volatile radioisotopes like radioiodine. Recently Wang et al. synthesized iodate substituted hydroxyapatite and compacted this material up-to 89% of relative density at 300°C by using relatively a complex sintering technique of spark plasma sintering. However, the density of the sintered matrices needed to be increased to further reduce leaching rate of the loaded iodate as well as sintering temperature was needed to be further reduced.

We report the densification of >95% at a sintering temperature of 200ºC under a uniaxial pressure of 500 MPa for 10 minutes. We have investigated the role of different parameters in lowering sintering temperature of synthesized hydroxyapatite and have concluded that physiosorbed and chemisorbed water present in dried powder has the vital role in achieving such high density and low-temperature sintering. We have also observed very small grain growth for these sintering conditions. The effect of these low temperature and relatively high-pressure conditions on mechanical strength and other properties of sintered matrices are being studied. Further to enhance structural durability and reduce leaching rate of loaded iodine various type of additives having positive effect on these parameters are also being investigated

9:20 AM

(ICACC-S13-029-2018) Saturation of radiation damage in A-atom layers of Ti_3AlC_2-$Ti_5Al_2C_3$ and Ti_3SiC_2

C. Ang*[1]; P. Edmondson[2]; C. Parish[2]; Y. Katoh[2]

1. University of Tennessee, Nuclear Engineering, USA
2. Oak Ridge National Lab, USA

MAX phases are potential candidates for structural materials in advanced reactors. However, neutron irradiations have been limited, and the effects on the physical processes of recovery are still being speculated. We report irradiation of Ti_3AlC_2-$Ti_5Al_2C_3$ and Ti_3SiC_2 to a displacement dose of ~10 dpa (10^{26} n/m^2, E > 0.1 MeV) at (T_{irr}) ~400 and ~700°C. This is the highest neutron fluence to date. Preliminary analysis included X-ray Diffraction, electrical resistivity and macroscopic swelling measurements. Point defect lattice dilation and subsequent deterioration of electivity resistivity show clear saturation with respect to dose. No change in electrical resistivity and lattice parameters are observed at T_{irr} ~700°C, but macroscopic swelling also appears to saturate ~2 and 4% respectively in Ti_3SiC_2 and Ti_3AlC_2-$Ti_5Al_2C_3$. After T_{irr} ~700°C, TEM shows basal planar defects with a strain field ½ <0001>. These have high Z-atomic contrast relative to the A and X atoms. Therefore, the recovery process along the A-layer also retains point defects that have become prismatic antisite (M_A) discs. Several possibilities explaining saturation of macroscopic swelling with dose will be presented. Research sponsored by the Office of Fusion Energy Sciences, U.S. Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

9:40 AM

(ICACC-S13-030-2018) Effect of neutron irradiation on microstructure evolution of isotopically-controlled titanium diboride ($Ti^{11}B_2$)

A. Bhattacharya*[1]; T. Koyanagi[1]; C. Parish[1]; Y. Katoh[1]; D. King[2]; G. Hilmas[2]

1. Oak Ridge National Lab, Materials Science and Technology Division, USA
2. University of Missouri-Rolla, Materials Science & Engineering Department, USA

Owing to a unique combination of mechanical and physical properties such as high melting temperature (>3000 °C), high thermal and electrical conductivity, transition metal borides are emerging as a novel class of materials for applications in extreme environments. In particular, we believe their properties are potentially useful for structural applications in fusion reactors. However, the key to success for application in nuclear environments lies in the basic understanding of radiation induced defects in the material. For this purpose, a fundamental neutron irradiation study was performed on polycrystalline $Ti^{11}B_2$ at 200 and 600 °C using High Flux Isotope Reactor upto a dose of ~2×10^{25} n/m^2 (E > 0.1 MeV). The samples were produced using > 99 at.% purity of ^{11}B powder instead of ^{10}B to avoid producing transmutant helium. Post-irradiation characterization by transmission electron microscopy revealed that $Ti^{11}B_2$ is resistant to amorphisation at 200 °C. The microstructure mainly consisted of extended defects like dislocation loops. In this work, we present a detailed characterization of the radiation induced defects detected in $Ti^{11}B_2$ and their evolution with irradiation temperature. Research partially sponsored by the U.S. Department of Energy, Office of Fusion Energy Sciences under contract DE-AC05-00OR22725 with ORNL, managed by UT-Battelle, LLC.

10:20 AM

(ICACC-S13-031-2018) Advanced manufacturing of ceramics for nuclear fission systems (Invited)

K. Terrani*[1]; M. Trammell[1]; S. C. Finkeldei[1]; J. Kiggans[1]

1. Oak Ridge National Lab, USA

Aside from the 100 tonnes of urania pellets that fuel the core of each of the 400+ nuclear power plants worldwide, ceramics are rarely used in these systems. The absence of this class of materials in these power systems is not due to their insufficient functionality, but rather the lack of experience in their utilization and difficulty in their production. While utilization of radiation stable ceramics can enhance the performance and safety of the current reactors, advanced reactors, particularly the high temperature variants, require ceramic components to meet their operational targets. Advanced manufacturing of ceramics is a transformative area that enables easier adoption of ceramics in these systems while allowing for the design of the fission systems to be re-imagined. The advanced manufacturing methods allow for distribution of fuel ceramics with strict spatial control to shape the power profile and enable production of complex geometries for ceramic structural components. The potential for these methods is discussed using a number of demonstration examples to date.

10:50 AM

(ICACC-S13-033-2018) Developing Radiation Resistant Ceramics through Microstructural Engineering

N. J. Madden*[1]; K. Hattar[2]; J. A. Krogstad[1]

1. University of Illinois at Urbana-Champaign, Material Science and Engineering, USA
2. Sandia National Laboratories, USA

There is a fundamental need to understand how ceramic materials with realistic microstructural features, such as grain boundaries and pores, respond to and evolve under irradiation. In particular, the role and strength of such features as defect sinks is of continuing interest and the subject of this work. Polycrystalline yttria stabilized zirconia (YSZ) was selected as a model ceramic system. The 20YSZ ceramic bodies were densified to approximately 90% of the theoretical density. Regions with high residual porosity were intentionally targeted for imaging during in-situ ion irradiation within the TEM. This allowed for direct measurements of the defect transport and annihilation kinetics relative to pore surfaces and grain boundaries. Ex-situ experiments on bulk polycrystalline ceramic specimens complement the in-situ TEM experiments by offering a wider perspective on radiation induced or accelerated microstructural evolution. Both the in-situ and ex-situ radiation experiments were done at room temperatures to establish baseline behavior of the model ceramic system in anticipation of future elevated temperature observations aimed at establishing a temperature dependent model for microstructural evolution in polycrystalline ceramics.

11:10 AM

(ICACC-S13-034-2018) Microstructural characterization of nuclear graphite: From the microscale to the nanoscale

J. D. Arregui-Mena*[1]; P. D. Edmondson[1]; G. W. Helmreich[1]; A. Campbell[1]; Y. Katoh[1]

1. Oak Ridge National Lab, Nuclear Materials Science & Technology Group, USA

Multiple reactor designs use graphite as moderator and as structural support. During the usage lifetime irradiation creates defects on the microstructure. The evolution of irradiation defects and mechanical properties can be traced by the pore structure. Pores in graphite

follow a well-known pattern, at the beginning of the irradiation process they close and accommodate the swelling of the crystallites. Then, after the pores are closed then the anisotropic crystallite growth leads to generation of new pores. An understanding of this process would improve the lifetime predictions of graphite components. We present a combination of microscopy techniques to investigate this process at multiple length scales. Optical microscopy, is used to observe the microstructure of the different components of graphite and their porosity content at the larger scale (>10 μm). X-ray computed tomography provides information of the 3D structure, connectivity and content of the voids in the range of 100 μm to 100 nm. FIB-SEM tomography combines the milling and scanning of the eroded surface to characterize smaller pores around 1 μm to 10 nm. This presentation will discuss how these techniques are being combined in an effort to link the mechanical properties changes to the evolution of pores in graphite.

S14: Crystalline Materials for Electrical, Optical and Medical Applications

Piezoelectric Material

Room: Tomoka C

Session Chairs: Elizabeth Kupp, The Pennsylvania State University; Danilo Suvorov, Jozef Stefan Institute

8:30 AM

(ICACC-S14-023-2018) Synthesis of nano $KNbO_3$ perovskite at room temperature (Invited)

K. Toda*[1]

1. Niigata University, Japan

We studied a oxyfluoride layered perovskite K_2NbO_3F as a precursor for synthesis of nanoparticles and thin films. The K_2NbO_3F has a unique layered perovskite structure with an ordered KF block and a KO block between layers. An interlayer KF block composed of fluorine of negative charge 1 has weaker bonding than a perovskite block. The potassium ions and fluoride ions in K_2NbO_3F are removed easily when stirred into water at room temperature. Using this technique, we synthesized $KNbO_3$ nanoparticles and thin film at room temperature. Perovskite-type potassium niobate thin films were prepared by a new nanosheet self-assembling technique on a glass substrate. A homogeneous nanosheet precursor solution was obtained by dissolving layered perovskite K_2NbO_3F in water at room temperature without any intercalant. X-ray diffraction patterns and ultraviolet-visible absorption spectra clearly show a continuous transformation from layered perovskite K_2NbO_3F to perovskite $KNbO_3$, suggesting the growth of a self-assembled multilayer consisting of perovskite nanosheets and K^+ ions.

9:00 AM

(ICACC-S14-024-2018) Formation of secondary phases in lead-free $(Na_{1-x}K_x)_{0.5}Bi_{0.5}TiO_3$-based piezoceramics (Invited)

D. Suvorov*[1]; J. Konig[1]; M. Spreitzer[1]

1. Jozef Stefan Institute, Advanced Materials, Slovenia

Among several alternatives to replace PZT/PLZT is $(Na_{1-x}K_x)_{0.5}Bi_{0.5}TiO_3$ ceramics. To clarify the inconsistencies reported in the literature, we investigated the formation mechanisms of $Na_{0.5}Bi_{0.5}TiO_3$ (NBT), $K_{0.5}Bi_{0.5}TiO_3$ (KBT), and several $(Na_{1-x}K_x)_{0.5}Bi_{0.5}TiO_3$ solid solutions. In the NBT–KBT system, the content of polytitante secondary phase increases with the amount of KBT. Bellow 50 mol% of KBT, no significant effect on the properties of ceramics was observed. We confirmed that in the NBT–$NaTaO_3$ system no secondary phases are formed which connects the formation of secondary phases with the presence of potassium. For modification of the electrical properties we doped NBT with Ta. In the NBT–$NaTaO_3$ system, no secondary phases were formed. In contrast, in the NBT–$KTaO_3$ system the polytitanate phase was observed. A modification of NBT with Ta, shifts the dielectric anomalies to lower temperatures and changes the behaviour of the material from ferroelectric to relaxor. We observed that the solid solution initially starts to form with the formation of the NBT- and $KTaO_3$-rich phases which then react towards the nominal composition with annealing at higher temperatures. In this contribution, the details of the synthesis and their implication on the resulting piezoelectric properties of these materials will be discussed.

9:30 AM

(ICACC-S14-025-2018) Structure-Property Relationships in Texture-Engineered Ceramics (Invited)

G. L. Messing[1]; S. Poterala[1]; Y. Chang[1]; E. R. Kupp*[1]; T. Frueh[1]; B. Watson[1]; R. Walton[1]; M. Brova[1]; A. Hofer[2]; R. Bermejo[2]; R. Meyer[3]

1. The Pennsylvania State University, Materials Science and Engineering, USA
2. MontanUniversityet Leoben, Austria
3. Pennsylvania State University, Applied Research Laboratory, USA

A range of properties exists in the continuum between isotropic bulk ceramics and single crystals when those properties show anisotropy based on crystallographic direction. Single crystals are typically utilized to access maximized properties (e.g., magnetic, piezoelectric, electronic, optical, thermoelectric and structural) in a particular direction. There are, however, multiple physical, chemical and cost issues that limit their wide application. Texture-engineered ceramics allow access to near-single crystal property values by crystallographically aligning the grains in ceramics and overcoming the disadvantages inherent in single crystals. This talk will describe texture quality assessment in texture-engineered ceramics and review properties and applications of these materials.

10:20 AM

(ICACC-S14-026-2018) Field induced phase transition in PMN-xPT single crystal near MPB

Q. Li*[1]; C. Xu[1]; Q. Yan[1]; Y. Zhou[1]

1. Tsinghua University, Department of Chemistry, China

PMN-xPT relaxor-based ferroelectric single crystals have attracted extensive attentions in recent years for the designs of ultrasonic transducers and piezoelectric actuators etc., owing to their ultra-high k_{33} and d_{33}. The origin of the high performance is a vital topic for scientists and the intrinsic structure around the morphotropic phase boundary(MPB) region still remains in dispute till now. In this work, we focus on systematically investigating phase structure, phase transition and phase stability in relaxor-based PMN-xPT single crystal near the MPB via domain structure observations combined with electrical characterizations. Further researches are carried on as-grown single crystals near the MPB before and after poling samples. The results indicate that unpoled MPB single crystals are either in hombohedral(R) or tetragonal(T) phase according to their extinction positions of the domains under the polarized light microscope(PLM). Poling at room temperature, a bridging phase appears near the rhombohedral-tetragonal(R-T)phase boundary. By domain structure and electrical property analysis, the bridging phase is identified to be orthorhombic rather than the reported monoclinicor adaptive regardless of the poling directions. Based on the temperature induced phase transition behavior, the temperature-composition phase diagrams are constructed for both the poled and unpoled PMN-PT single crystals.

10:40 AM

(ICACC-S14-027-2018) Texture control of ceramics with electric field orientation (Invited)

T. Nakayama*[1]; M. Kanno[1]; H. Cho[2]; S. T. Nguyen[1]; T. Suzuki[1]; H. Suematsu[1]; K. Niihara[1]

1. Nagaoka Univ of Tech, Japan
2. Hanyang University, Republic of Korea

The texture and micro structure control of the magnetic fillers is one of the important point on these materials The orientation of Barium-Ferrite particles was controlled in polymer-based nanocomposite film using microscopic molds while applying a DC electric field. The Barium-Ferrite particles were dispersed by sonication in a prepolymer mixture of polysiloxane followed by a high speed mixing. The homogeneous suspension was cast on a microscopic mold with different patterns, which is attached to positive electrode during application of electric field before it became cross-linked. Analysis revealed that filament-like linear assembles of Barium-Ferrite particlewere fabricated in polysiloxane/Barium-Ferrite hybrid films, and Barium-Ferrite particles composing filament-like linear assembles were aligned perpendicular to the film plane with high anisotropy. 3D microstructure imaging on hybrid materials were successfully observed by optimizing the CT condition.

11:10 AM

(ICACC-S14-028-2018) Surface electric fields of bioceramic electrets promote cell adhesion (Invited)

M. Nakamura*[1]; K. Yamashita[1]

1. Tokyo Medical and Dental University, Institute of Biomaterials and Bioengineering, Japan

The surface modification of ceramic biomaterials used for medical devices is expected to improve the osteoconductivity through control of the interfaces between the materials and living tissues. The purpose of this study was to investigate a mechanism through which the surface wettability of biomaterials can be improved and determine the effects of biomaterial surface characteristics on cellular behaviors. Polarization treatment induced surface charges on hydroxyapatite, b-tricalcium phosphate, carbonate-substituted hydroxyapatite and yttria-stabilized zirconia regardless of the differences in the carrier ions participating in the polarization. Characterization of the surfaces revealed that the wettability of the polarized ceramic biomaterials was improved through the increase in the surface free energies compared with conventional ceramic surfaces. In addition, sintering atmosphere affects the polarization capacity of hydroxyapatite by changing hydroxide ion content and grain size. Compared with hydroxyapatite sintered in air, hydroxyapatite sintered in saturated water vapor had a higher polarization capacity that increased surface free energy and improved wettability, which in turn accelerated cell adhesion. We determined the optimal conditions of hydroxyapatite polarization for the improvement of surface wettability and acceleration of cell adhesion.

11:40 AM

(ICACC-S14-029-2018) Reduced Graphene Oxide-Nanoparticles Nanocomposite "Synthesis and Characterization"

A. Nemati*[1]

1. Sharif University of Technology, Materials Science & Eng., Islamic Republic of Iran

Synthesis and characterization of reduced graphene oxide (RGO) based nanocomposites consist of different oxides is reported. After preparation of RGO (carried out via modified Hammer method and proper reduction process), different oxides nanoparticles (Fe_3O_4 and ZnO and TiO_2) were synthesized and decorated on RGO. The phase/s, morphology and characteristics of RGO, nanoparticles and nanocomposite were investigated by FTIR, Photoluminescence (PL), XRD, SEM/FESEM and TEM analysis and VSM. The FTIR studies of nanoparticles and nanocomposites showed O-H peak at about 3400-3500 cm^{-1}, which was related to the absorbed water in the samples. TEM studies revealed the formation of a few layers RGO, as well as nanoparticles. FESEM/SEM and TEM results showed that the average sizes of synthesized magnetite nanoparticles were less than 15 nm. FTIR showed the existence of Fe-O, Zn–O, Ti-O and RGO vibrations bands.

S15: Additive Manufacturing and 3-D Printing Technologies

Slurry & Ink Jet Printing

Room: Coquina Salon B

Session Chair: Tyrone Jones, US Army Research Laboratory

8:30 AM

(ICACC-S15-027-2018) Challenges and Issues in Additive Manufacturing of Ceramic Products (Invited)

T. Ohji*[1]; N. Kondo[1]

1. National Institute of Advanced Industrial Science and Technology (AIST), Japan

For realizing complex-shaped products with reduced lead-time, additive manufacturing (AM) technologies for ceramics products have been developed in "High-Value Added Ceramic Products Manufacturing Technologies" project (sponsored by the Japanese government) since 2014. The project deals with two technologies for producing ceramic green bodies; powder layer manufacturing (powder bed fusion, or indirect selective laser sintering) and slurry layer manufacturing (stereolithography) due to adjustability of green density, relatively good precision, and complex-shaping capability, in addition to ceramic laser sintering (direct selective laser sintering). The paper will describe the up-to-date research achievements in this project, including the unique-structured 3D bodies never attainable in conventional methods. Particularly the paper addresses a number the technical items which should be carefully considered and properly selected, for optimizing the AM procedures. It will show what sorts of technical items we have, how those are connected and correlated each other and what should be considered and selected in each item in order to obtain sound products through AM approach, taking an example of powder layer manufacturing. This work was conducted as a part of "High-value added ceramic products manufacturing technologies project" supported by CSTI, SIP, "Innovative design/manufacturing technologies (managed by NEDO)".

9:00 AM

(ICACC-S15-028-2018) LSD- 3D printing: Powder based Additive Manufacturing, from porcelain to technical ceramics

A. Zocca*[1]; P. Lima[1]; T. Mühler[2]; J. Lüchtenborg[1]; J. Guenster[1]

1. BAM Federal Institute for Materials Research and Testing, Ceramic Processing and Biomaterials, Germany
2. TU Clausthal, LaserAnwendungsCentrum, Germany

Powder based Additive Manufacturing (AM) processes are widely used for metallic and polymeric materials, but rarely commercially used for ceramic materials, especially for technical ceramics. This seemingly contradicting observation is explained by the fact that in powder based AM, a dry flowable powder needs to be used. Technical ceramics powders are in fact typically very fine and poorly flowable, which makes them not suitable for AM. The layerwise slurry deposition (LSD) is an innovative process for the deposition of powder layers with a high packing density for powder based AM. In the LSD process, a ceramic slurry is deposited to form thin powder layers, rather than using a dry powder This allows the use of fine powders and achieves high packing density (55-60%) in the layers after drying. When coupled with a printing head or with a laser source, the LSD enables novel AM technologies which are similar to

the 3D printing or selective laser sintering, but taking advantage of having a highly dense powder bed. The LSD -3D printing, in particular, offers the potential of producing large (> 100 mm) and high quality ceramic parts, with microstructure and properties similar to traditional processing. This presentation will give an overview of the milestones in the development of this technology, with focus on the latest results applied both to silicate and to technical ceramics.

9:20 AM

(ICACC-S15-029-2018) Three Dimensional Ceramics Printed via Ink Jet Methods

D. Crenshaw[1]; X. Wang*[1]

1. Alfred University, School of Engineering, USA

We report new results on ink jet printed three dimensional ceramics with various post forming treatments. First, the precursor materials will be discussed, along with particle sizes and different binders. Second, the shapes and sizes will be adjusted, while porosities being varied. Third, heat treatment conditions will be altered to achieve desired mass densities. Fourth, mechanical properties will be tested, correlated to the morphologies and topologies. Applications will be considered with alumina as the main subject for the research. All experimental results will be given, including SEM/EDS and 3-point bending testing.

9:40 AM

(ICACC-S15-030-2018) 3D Printing of thermal insulating zirconia structures

A. Chrystel[1]; S. Beaudet Savignat*[1]

1. CEA, France

The aim of this study is to develop thermal insulating ceramics of yttrium stabilized-zirconia using the 3D printing technique. Six zirconia powders with different particle sizes, particle morphologies (from fiber to sphere) and specific surface area were selected and mixed with an inorganic solid binder. This binder will react with the printer's infiltrant (mainly composed of water) to form a rigid skeleton and embed the zirconia powder. Powder mixtures of the binder and different zirconia contents were evaluated, according to printing tests of an half cubic lattice to determine the maximum zirconia content. It appears that the maximum zirconia content is obtained for zirconia powders with a particle size around 10 microns and a morphology close to that of the binder (angular particles). Zirconia powders made of porous fibers may inhibit the reaction between the binder and the printer's infiltrant by a preferential absorption of the liquid within the porosity of the fibers. Small particles zirconia powders (particle size ≤ 1 micron) may adsorb on the binder and therefore prevent the accessibility to the infiltrant. The thermal properties of the optimized materials were evaluated using the laser flash method and calorimetric measurements (DSC). The lowest thermal conductivity was obtained using a fibrous zirconia powder. Further work may focus on mechanical reinforcement of the porous structures by impregnation techniques.

Emerging Technologies

Room: Coquina Salon B

Session Chair: Tatsuki Ohji, National Institute of Advanced Industrial Science and Technology (AIST)

10:20 AM

(ICACC-S15-031-2018) Additive Manufacturing of Ceramics for Protection Systems: Technical Challenges and Opportunities (Invited)

T. Jones*[1]

1. US Army Research Laboratory, USA

As the landscape of warfare changes, the US Army must shift its approach to protection strategies to maintain its warfare dominance. New technological advances in manufacturing have enabled the dismounted Soldier to tailor their body armor material needs to the demand of the environment. Traditional manufacturing of ceramics present limitations of long lead times, fabrication of complex geometries, and cost expensive components. The additive manufacturing of ceramics offers engineering-grade ceramic components in approximately 90% less time than traditional ceramics. Typical turn around can be as little as five days depending on complexity of the part. This not only allows for faster time to market, but also allows for more iterations during the design process resulting in a better end product. Additionally, 3-D printed parts can be fabricated with a higher degree of complexity for weight reduction while saving on the cost of the part because of the reduction in material used. Over the last couple of years the US Army Research Laboratory investigated the integrity of 3-D printed ceramics for protection systems. This paper will discuss the technical challenges that were discovered, and provide opportunities to the ceramic community to create robust solutions.

10:50 AM

(ICACC-S15-032-2018) Quality Aspects of Additively Manufactured Medical Implants

J. Wilbig[1]; F. Leonard[1]; G. Bruno[1]; J. Guenster*[1]

1. BAM Federal Institute for Materials Research and Testing, Germany

Rapid fabrication of individualized products is of special interest for the medical sector. Beyond research, only a few medical companies use additive manufacturing (AM) to fabricate medical end products. AM has advanced at a much faster pace than regulations or quality controls. The lack of standards and knowledge gaps in quality control make companies waiting for more evidence of reliability to ensure patient safety before adopting the technology. The European Metrology Programme for Innovation and Research (EMPIR) has set up a project to diminish these gaps. Validated techniques to verify the finished parts, thereby improving processes as well as reliability of the manufacturing chain are the objectives of the project, with the intention to increase the confidence of the medical device industry in the AM technologies. We will report about recent advances within the framework of the EMPIR project (Metrology for additively manufactured medical implants, MetAMMI).

11:10 AM

(ICACC-S15-033-2018) A novel Approach to Facilitate Densification and Strength of 3D Printed SiC Articles

A. El-Ghannam*[1]; T. Schmitz[1]; D. Beasock[1]

1. University of North Carolina at Charlotte, USA

3D printing of SiC provides an alternative manufacturing technique that enables design flexibility, rapid production and reduced machining time compared to subtractive grinding and polishing. However, 3D printing of SiC would still require sintering of the green objects in order to increase density and mechanical strength. Sintering ceramic particles at high temperature would result in shrinking of the printed object due to fusion of particles, a phenomena that results in dimensional changes which defeat the purpose of using 3D printing. In the present study we have taken a novel approach to facilitate densification of 3D printed SiC articles without compromising the dimensions. The enhanced densification of SiC is achieved by growing a secondary silicon oxide fiber and plate-type crystals at moderate temperature and humidity (referred to here as "Amazon crystals"). SEM-EDX, FTIR and XRD analyses indicated that the growth of the amazon crystals starts at the surface of the SiC particles and propagate inwards to bridge the walls of the pores leading to significant increase in density and mechanical strength. The compression strength increased from 76.5 ± 4 to 150.7 ± 6 MPa as the sintering temperature increased from 900C/2h to 1100C/24. Results of the study demonstrate the possibility of designing near-net shape SiC objects for space-based optics and ballistic armor applications.

11:30 AM

(ICACC-S15-034-2018) Considerations on 3D printing of refractories

S. Shi*[1]

1. Materials Technology Innovation, LLC, USA

3D printing, also known as additive manufacturing, has already developed into a rapid growing industry for plastic and metallic objects manufacturing. The newly developed industry would also embrace the fabrication of special refractories, because there are number kinds of refractory articles that have complicated shapes and structures. Furthermore, in-situ installation of monolithic refractories seems more suitable to adopt the 3D printing technology. In this presentation, technical issues for 3D printing of refractories were discussed and the R&D direction in this regard was suggested. They include (1) matrix materials that can provide sufficient bonding force at both of ambient and elevated temperatures; (2) the aggregates that have sufficient flowability, dense packing ability and ability to form strong grips with the matrix materials; and (3) approaches for rapid printing, rapid solidification and rapid drying out.

S16: Geopolymers, Inorganic Polymers and Sustainable Materials

Synthesis, Processing and Microstructure I

Room: Ponce de Leon

Session Chair: Waltraud Kriven, University of Illinois at Urbana-Champaign

8:30 AM

(ICACC-S16-001-2018) Examination of Structural Properties and Thermal Stabilities of Geopolymer Nanomaterials (Invited)

D. Seo*[1]; S. Chen[1]

1. Arizona State University, School of Molecular Sciences, USA

Geopolymer nanomaterials are a new class of inorganic materials and may be listed among the few that can be produced conveniently in a large scale under ambient synthetic conditions. For example, by adjusting the geopolymer resin compositions with high alkali concentrations, geopolymerization process can be controlled to produce submicron-sized highly structured nanoaggregates of geopolymer. The size range and morphology of the geopolymer nanoaggregates are strikingly similar to those of precipitated silica and carbon black. Given the massive production and application of the latter materials in the range of millions of tonnes per year, the advent of the new geopolymer material type has a huge implication in novel large-scale applications of geopolymer. We will discuss the relationship between their synthetic conditions and resulting pore and aggregate structures, as well as their structural changes at high temperatures, by using potassium-based geopolymer nanomaterials as an example.

9:00 AM

(ICACC-S16-002-2018) Additive Manufacturing of Ceramics with Geopolymers (Invited)

P. Colombo*[1]; G. Franchin[1]; P. Scanferla[1]

1. University of Padova, Industrial engineering, Italy

Metakaolin-based geopolymer components foams were fabricated by Additive Manufacturing. Both a direct AM technique (Direct Ink Writing) and an indirect AM technique (powder bed) were used. Printable inks were obtained by controlling the composition, rheology and additives in the formulation as well as taking advantage of the time during which the viscosity of the system was rather constant. High-porosity components were produced for potential use as water purification filters. The use of a liquid geopolymer as the binder in powder-bed AM was also explored, leading to components with improved mechanical properties with respect to using other inorganic binder systems.

9:30 AM

(ICACC-S16-003-2018) Additive Manufacturing of Geopolymers by Local Laser Drying (Invited)

T. Mühler*[1]; J. Lüchtenborg[2]; J. Guenster[2]; P. Hlavacek[2]; P. Sturm[2]; G. J. Gluth[2]

1. Clausthal University of Technology, Institute of Non-Metallic Materials, Germany
2. Bundesanstalt für Materialforschung und -prüfung, Germany

For the additive manufacture of large components typically powder-based methods are used. A powder is deposited layer by layer by means of a recoater, then the component structure is printed into each individual layer. We introduce here the new method of local laser drying, which is a suspension-based method specially developed for the manufacture of large voluminous ceramic parts. The structure information is directly written into the freshly deposited layer of suspension by local drying. Initially the technology was developed for ceramic suspensions, however, first experiments with geopolymers reveal a high potential for this class of materials. Metakaolin, fly ash as well as silica and lithium aluminate based geopolymers were used in first experiments. The local annealing of the geopolymer slurry results in a drying and crosslinking reaction and, thus, in a local consolidation of the material. First parts made will be presented and their properties will be discussed.

Synthesis, Processing and Microstructure II

Room: Ponce de Leon

Session Chair: Paolo Colombo, University of Padova

10:20 AM

(ICACC-S16-004-2018) 3D Printing of Alumina-Platelet – Reinforced Geopolymer Composites (Invited)

B. Munoz[1]; P. F. Keane[1]; W. M. Kriven*[1]

1. University of Illinois at Urbana-Champaign, USA

A 3D Arduino Mega printer was bought and built, where the fluid to be printed was deposited by a pulsating air compressor. K-based, geopolymer composite reinforced with up to 67 wt % alumina platelets of average diameter 50 mm. The composition of the geopolymer was $K_2OAl_2O_3 \bullet 4SiO_2 \bullet 11H_2O$ and the alumina platelets had a predominant particle size of 50 mm. The extrudate had a diameter of 1-2 mm. Various shapes were continuously extruded and allowed to set at ambient temperatures under high relative humidity of water. This work demonstrated the feasibility of continuous, additive manufacturing of reinforced, stoichiometric geopolymer composites of high viscosity and setting under ambient conditions in a moist relative humidity.

10:40 AM

(ICACC-S16-005-2018) Basalt Chopped Fiber Reinforced, Amorphous Self-Sealed Geopolymers (ASS-G) and Amorphous Self-Sealed Ceramics (ASS-C) (Invited)

P. F. Keane*[2]; C. P. Marsh[2]; W. M. Kriven[1]

1. University of Illinois at Urbana-Champaign, USA
2. ERDC, Construction Engineering Research Laboratory, USA

Potassium-based geopolymer of composition composition $K_2O \bullet Al_2O_3 \bullet 4SiO_2 \bullet 11H_2O$ of low viscosity was fabricated by the standard procedure in a high shear IKA mixer. Basalt and glass frit were then dispersed in KGP using a gravitational Thinky mixer and the samples allowed to set under applied pressure for at ambient temperatures for 1 day followed by 1 day at 50°C to complete the reaction. 1"x1"x6" Samples were heat treated up to 1200°C for 1 hour before 3-point bend tests were conducted. Density and shrinkage were also recorded after treatments. SEM/EDS data suggests melting

and bonding of the glass dispersed phase into the surrounding KGP matrix, with a self sealing effect on the dehydrated and cracked matrix. Low melting temperature glass was selected to produce self-sealing/ crack filling in an a dehydrated but unfired geopolymer composite (900-1000°C). Kamenivec basalt from Kamenivec Company in Moscow, melted after KGP transformed into leucite, providing a network/glass filling system in a ceramic (1200°C). At intermediate temperatures the geopolymer was converted to a ceramic, but the basalt fibers remained intact.

11:00 AM

(ICACC-S16-006-2018) One-part geopolymers and geopolymer-zeolite composites based on silica: Factors influencing microstructure and engineering properties (Invited)

G. J. Gluth*[1]; P. Sturm[1]; S. Greiser[1]; C. Jäger[1]; H. Kühne[1]

1. Bundesanstalt für Materialforschung und -prüfung (BAM), Germany

Mixing and curing of geopolymers and related alkali-activated materials without storage and handling of highly alkaline solutions possesses advantages regarding safety and economic viability; one possible approach is to produce these materials from solid silica feedstocks and sodium aluminate by mixing with water. We present a comparison between geopolymers and geopolymer-zeolite composites synthesized by this route from different silica feedstocks (by-product silica from chlorosilane production, microsilica, rice husk ash) and with different SiO_2/Al_2O_3 ratios. Results obtained from NMR, XRD, SEM, thermal analysis, mechanical and durability testing are used to study the effects of these factors. The use of rice husk ash favors formation of a fully amorphous geopolymer with good mechanical properties. Use of the two other silica sources leads to formation of geopolymer-zeolite composites, the amount and kind of zeolites depending on the feedstock and the SiO_2/Al_2O_3 ratio. These materials show superior dehydration behavior (no distinct dehydration step of thermal strain) with a controllable phase assemblage on heating to 1000°C. Mortars produced from both, the geopolymers as well as the composites, exhibit a high resistance against sulfuric acid attack, making them promising materials for the repair of industrial and sewage structures.

11:30 AM

(ICACC-S16-007-2018) Geopolymerizaition Process and Mechanism of the Lithium-substituted Cesium-based Geopolymers (Invited)

J. Yuan*[1]; P. He[1]; D. Jia[1]

1. Harbin Institute of Technology, School of Materials Science and Engineering, China

The geopolymerization process and mechanism of $Cs_{(1-x)}Li_xGP$ and the effects of Li^+ substitution on the mechanism of polymerization was to investigate in this paper. The whole reaction process was divided into two stages in this paper, namely, reacting stage (Stage I) and curing stage (Stage II), and the tested specimens were extracted from whole geopolymerization process at specific times. A series of test characterization techniques, including FI-IR, XRD, NMR and SEM, were employed to characterize the valence bond structure, phase composition, coordination configuration and morphology evolution during geopolymerization process. The results indicated that the dissolution and diffusion process were crucial for the whole geopolymerization in Stage I, while Stage II was dominated by condensation, polymerization and reorganization process. A transformation from Al^V and Al^{VI} atom into Al^{IV} atom occurred in the whole geopolymerization process, and the conversion rate was significantly accelerated from Stage I transferred into Stage II. In addition, the geopolymerization process was evidently accelerated after Cs^+ ion substituted by Li^+ ion, and the acceleration effect was more obvious with the increase of Li^+ ion content, which could be ascribed to a higher charge/mass ratio or charge density of Li^+ ion than that of Cs^+ ion.

S17: Advanced Ceramic Materials and Processing for Photonics and Energy

Photonics I

Room: Halifax A/B
Session Chairs: Fabio Cicoira, Polytechnique Montreal; Gilles Lerondel, University of Technology of Troyes

8:30 AM

(ICACC-S17-021-2018) Fluoresent Carbon Dots in Sensing and Imaging Applications (Invited)

J. Manioudakis[1]; J. Macairan[1]; F. Noun[1]; F. Victoria[1]; R. Naccache*[1]

1. Concordia University, Chemistry and Biochemistry, Canada

Carbon dots have garnered significant interest as fluorescent materials with a vast potential in sensing and imaging applications, in optoelectronics, as well as energy conversion. Their ultra-compact size, low cytotoxicity, low photo-bleaching/blinking, tunable photoluminescence, combined with simple, environmentally-friendly and low-cost synthesis, makes them ideal candidates for study. We synthesize carbon dots via a bottom-up microwave method, with simple organic precursors (e.g. citric acid) as the carbon source. We passivate the surface of our carbon dots to achieve high fluorescence quantum yields. Moreover, our work focuses on trying to elucidate the fluorescence mechanisms in carbon dots, which remain a subject of debate. Finally, we exploit their optical properties in order to design chiral, pH or temperature sensors, as well as fluorescent probes that are geared towards heavy metal detection in solution.

9:00 AM

(ICACC-S17-022-2018) Light Emitting Nanoplatforms Based on Rare Earth Doped Nanoparticles (Invited)

F. Vetrone*[1]

1. Institut National de la Recherche Scientifique, Cente Énergie, Matériaux et Télécommunications, Canada

Nanoparticles excited in the near-infrared (NIR), in particular rare earth doped nanoparticles, are quickly emerging as useful tools in diagnostic and therapeutic medicine. In particular, the usefulness of these nanoparticles for applications in biology stems primarily from the fact that NIR light is silent to tissues thus minimizing autofluorescence, possesses greater tissue penetration capabilities, reduced scattering, and does not cause photodamage to the specimen under investigation. Moreover, tailoring of the nanoparticles' absorption and emission wavelengths allow them to operate within the so-called "biological windows", regions of the spectrum in which tissues are partly transparent. Here, we present the synthesis and surface functionalization of various rare earth doped nanoparticles and show how they can be used as building blocks in the development of multifunctional hybrid nanoplatforms through intelligent combination with other optically active nanostructures, for the potential diagnostics and therapeutics of disease.

9:30 AM

(ICACC-S17-023-2018) Degenerately doped zinc oxide nanocrystals as plasmonic and chemoresistive gas sensors (Invited)

M. Sturaro[1]; E. Della Gaspera[2]; C. Cantalini[3]; M. Guglielmi[1]; A. Martucci*[1]

1. University of Padova, Industrial Engineering, Italy
2. RMIT University, Australia
3. University of L'Aquila, Italy

Highly doped wide band gap metal oxides nanocrystals have recently been proposed as building blocks for applications as transparent electrodes, electrochromics, plasmonics and optoelectronics in general. Here we demonstrate the application of ZnO doped with gallium (GZO), aluminum (AZO) and silicon (SZO)

nanocrystals as novel plasmonic and chemiresistive sensors for the detection of hazardous gases including hydrogen (H_2) and nitrogen dioxide (NO_2). GZO, AZO and SZO nanocrystals are obtained by non-aqueous colloidal heat-up synthesis with high transparency in the visible range and strong localized surface plasmon resonance (LSPR) in the near IR range, tunable with dopant concentration (up to 20% mol nominal). Thanks to the strong sensitivity of the LSPR to chemical and electrical changes occurring at the surface of the nanocrystals, such optical features can be used to detect the presence of toxic gases. By monitoring the changes in the dopant-induced plasmon resonance in the near infrared, we demonstrate that GZO, AZO and SZO thin films prepared depositing an assembly of highly doped ZnO colloids are able to optically detect both oxidizing and reducing gases at mild (< 100 °C) operating temperatures. Combined optical and electrical measurements show that the dopants within ZnO nanocrystals enhance the gas sensing response compared to undoped ZnO.

10:20 AM

(ICACC-S17-024-2018) Energy-Related and Optical Phenomena in Hybrid Nanostructures and Bio-assemblies (Invited)

A. Govorov*[1]

1. Ohio University, Physics, USA

Metal nanocrystals and semiconductor quantum dots have the ability to absorb and scatter light very efficiently. This study concerns special designs of hybrid nanostructures with electromagnetic hot spots, where the electromagnetic field becomes strongly enhanced. Overall plasmonic nanostructures with hot spots demonstrate strongly amplified optical and energy-related effects. (1) Using nanoparticle arrays made of different metals, one can transfer plasmonic signals coherently and with small losses. (2) Plasmonic hot spots efficiently generate energetic electrons, which can be used for photochemistry and photodetection. (3) Using nanostructures with hot spots, one can strongly enhance optical generation of heat and also confine high photo-temperatures in small volumes. (4) Colloidal nanocrystal assemblies with plasmon resonances allow us to strongly enhance chiral optical responses (circular dichroism) of biomolecules and drugs.

10:50 AM

(ICACC-S17-025-2018) Silicon Carbonitride Nanostructured Thin Films for Photonic Applications (Invited)

Z. Khatami[1]; J. Wojcik[1]; P. Mascher*[1]

1. McMaster University, Engineering Physics and CEDT, Canada

Silicon carbonitrides (SiCxNy) have attracted interest for the manufacturing of materials with robust mechanical properties and promising optical features. This is a consequence of their unique properties inherited from the combined properties of binary substructures, silicon carbide (SiC), silicon nitride (SiN), and carbonitride (CN). In this presentation we will show that the visible photoluminescence (PL) emission from SiCxNy films fabricated by PECVD is stronger than that of SiC and SiN materials and all optical properties (band gap, transmittance, index of refraction, and light emission) can be controlled by adjusting the carbon content. We will also discuss the influence of the deposition temperature on the visible luminescence from a-SiCxNy:Hz thin films by linking changes in the film properties, including hydrogen concentration, film microstructure, and composition to the observed changes of visible light emission. Finally, we will review the influence of Ar dilution during the growth and its consequences on the film composition and luminescent properties.

11:20 AM

(ICACC-S17-026-2018) Optical Properties of 3D CsPbBr3, 2D CsPb2Br5 and 0D Cs2PbBr6 (Invited)

J. Bao*[1]

1. University of Houston, USA

Organic-inorganic perovskites have found applications in many optoelectronics devices such as LED, lasers, photodetectors and high-energy radiation detectors. All-inorganic perovskites were later developed to improve device stability. Although nanocrystals of inorganic perovskites represented by 3D CsPbBr3 were quickly found to exhibit extremely large quantum efficiency, aggregated or micropower CsPbBr3 shows quantum yield as small as 0.1%. In the meantime, 2D CsPbBr5 and 0D Cs2PbBr6 were reported to show even higher photoluminescence quantum yield, but other people found no photoluminescence at all. In this talk, I will summarize their optical properties and discuss how these controvercies have happened.

11:50 AM

(ICACC-S17-027-2018) Low temperature process glass for lighting packaging

C. Chan*[1]; F. Wu[1]

1. National United University, Materials Science and Engineering, Taiwan

The reduction of energy consumption is one of the important issues in the modern process technology. The addition of functional elements such as Li, Na and K in the aluminophosphate glass system decreases glass characteristic temperatures and process temperature as well. With adequate Li, Na and K incorporate in the aluminophosphate glass transition temperature decreases down to 302 °C. When the elements mixed, they will reduce the free energy of system. The developed characteristic temperature glass is thus a potential material to replace the frequently polymers in lighting packages. In the developed aluminophosphate system, ZnO is further added to improve the optical properties of the glass. The Yttrium Aluminum Garnet, YAG, powders are then embedded in aluminophosphate glass to form, Glass-Ceramic-Phosphor, GCP, which the light conversion layer in the LED package. The GCP microstructure and performance are manipulate through the temperature and time control to achieve the conversion requirements. The GCP layer, about tens to hundred micrometers, is integrated and demonstrated in lighting device.

Honorary Sympoisum: Advancing Frontiers of Ceramics for Sustainable Society Development - International Symposium in Honor of Dr. Mrityunjay Singh

Advancing Frontiers of Ceramics IV -Energy Technologies

Room: Coquina Salon E
Session Chairs: Palani Balaya, National University of Singapore; Hua-Tay Lin, Guangdong University of Technology

8:30 AM

(ICACC-HON-027-2018) Can we Use Silicon as Stable and High Capacity Anode Material in Lithium Ion Batteries? Yes we can! (Invited)

R. Riedel*[1]; M. Graczyk-Zajac[1]; D. Vrankovic[1]

1. TU Darmstadt, Materials Science, Germany

In this presentation, we demonstrate a cost-effective synthesis route for coated, highly porous Si that is simple to implement and provides Si-based anode materials with capacities between 2000 and 3000 $mAhg_{Si}^{-1}$, Coulombic efficiencies above 99.5 %, and almost

100 % capacity retention over more than 100 cycles. The Si-based composite is prepared from porous silicon (obtained by reduction of silica) by encapsulation in an organic carbon and polymer-derived siliconoxycarbide (C/SiOC) matrix. Molecular-dynamics (MD) simulations show that the highly porous silicon morphology delivers free volume for the accommodation of strain leading to no macroscopic changes during initial Li-Si alloying. In addition, a carbon layer provides an electrical contact whereas the SiOC matrix significantly diminishes the interface between the electrolyte and the electrode material and thus suppresses the formation of a solid electrolyte interphase (SEI) on Si. Electrochemical tests of the micrometer-sized, glass-fibres derived silicon demonstrate the up-scaling potential of the presented approach.

9:00 AM

(ICACC-HON-028-2018) Non-flammable Sodium-ion Batteries for Large Scale Storage Systems (Invited)

P. Balaya*[1]

1. National University of Singapore, Department of Mechanical Engineering, Singapore

Deployment of smart grids using renewable energy (solar/wind) requires large scale electrical energy storage systems (EESS). Currently lithium-ion batteries (LIBs) are preferred for EESS. High power density LIBs addressing intermittency of renewables uses lithium titanate as anode but is expensive (USD800-1000/kWh). Besides, lithium is a scarcity. Sodium, on the other hand, is the sixth most abundant element. Sodium-ion batteries (NIBs) are expected to be durable, safe and inexpensive (about USD250/kWh). Regardless of the relatively lower energy density of NIBs, they can be effectively employed in smart grid applications, where the weight and footprint requirement are not severe. Non-flammable sodium-ion cells (18650) fabricated using sodium vanadium phosphate as cathode and hard carbon as anode exhibit energy density close to 55Wh/kg (kg refers to the full cell weight) with impressive 4C rate performance retaining close to 80% of the capacity observed at low rates. This is the only ultra-safe and inexpensive commercial type sodium-ion cells known so far with relatively higher energy density than the aqueous (non-flammable) NIBs available commercially. We will further present safety parameters such as heat losses and internal resistance of the reported 18650 cell which help future directions to develop thermal management systems for NIB packs for smart grids (100-500kWh).

9:20 AM

(ICACC-HON-029-2018) Materials for Energy Storage – Key Enabler for Low Carbon Energy Future (Invited)

R. Bordia*[1]; J. Nanda[2]

1. Clemson University, Materials Science and Engineering, USA
2. Oak Ridge National Lab, Materials Science and Technology Division, USA

Energy storage is the key enabler for renewable energy technologies such as wind and solar which by nature are intermittent. These technologies are critical in our progress towards a zero or ultra-low carbon footprint economy. The primary challenges are (i) low cost and (ii) methods and approaches that are sustainable. The presentation will provide an overview and roadmap for various high energy density electrochemical storage systems applicable for both automotive and grid applications. Critical technical challenges and barriers towards achieving high performance as well as cost will be discussed. The second part of the talk will cover a few specific examples of battery electrodes and materials from the point of view of optimization of ion-transport and microstructural control to enable high energy density that eventually reduces the cost at gravimetric and/or volumetric bases. Energy efficient approaches towards battery materials synthesis and processing including recycling methods will be presented. JN acknowledges support from the Vehicle Technologies Office, under Energy Efficiency and Renewable Energy (EERE) Office and the Office of Electricity Delivery and Energy Reliability (OE) under the Department of Energy, United States of America.

9:40 AM

(ICACC-HON-030-2018) Development of Oxide Ceramics for Thermoelectric Power Generation Applications (Invited)

M. Ohtaki*[1]

1. Kyushu University, Interdisciplinary Graduate School of Engineering Sciences, Japan

Development of metal oxides for thermoelectric energy conversion is surveyed along with their relatively steep progress in their history in thermoelectrics. As commonly recognized, refractory oxides have a broad advantage in mid-to-high temperature thermoelectric power generation, in which the electrical power is proportional to the square of the temperature difference thermoelectric devices are exposed. High durability of metal oxides at high temperature under aerobic conditions as well as their low cost and minimal environmental impact seem to be highly promising toward a global sustainable society with improved energy utilization. However, metal oxides also have some inherent disadvantages, including the relatively strong iconicity in their interatomic bonding. Several key issues of oxide materials are addressed, including the performance stability of metal oxides against oxygen non-stoichiometry at high temperature. A competitive thermodynamics-kinetics relation between the oxygen partial pressure and temperature can be crucial for some materials such as Ti-based oxides. Systematic consideration of electronic and structural (phonon) properties of metal oxides in terms of their interatomic bonding and crystal structure will be presented.

10:20 AM

(ICACC-HON-031-2018) Electroceramics for High Temperature Energy Systems (Invited)

P. Singh*[1]

1. University of Connecticut, MSE, USA

A number of ionic, electronic and mixed ionic-electronic oxides offer excellent structural, chemical and electrochemical properties for application as dense electrolyte membrane or porous electrodes in high temperature electrochemical systems ranging from fuel cells to electrolyzers and electrochemical reactors. Materials chemistry, defect structure, oxygen stoichiometry and their role on electrical properties and structural stability in oxidizing and reducing atmospheres will be examined and approaches for developing surface and interfacial stability will be presented. Experimental findings pertaining to dopant exsolution, interactions with solid fluorite (e.g. YSZ) and intrinsic and extrinsic gas phase impurities (e.g. CrOHx, SOx, and halides) will be presented. Mechanisms for electrochemical performance degradation with reference to electrochemical poisoning, surface coverage and compound formation will be discussed.

10:50 AM

(ICACC-HON-032-2018) Glass Sealing of Solid Oxide Fuel Cells: Processing and Mechanical Characterization (Invited)

M. J. Hoffmann*[1]; E. Reitz[1]; B. Ehreiser[1]; G. Schell[1]; E. C. Bucharsky[1]

1. Karlsruhe Institute of Technology, Institute for Applied Materials (IAM-KWT), Germany

Solid Oxide Fuel Cell (SOFC) stacks can be sealed by a high temperature resistant and electrical insulating glass ceramics. In our present studies we focus on the fundamentals during joining of two steel plates. The glass solder was deposited by screen printing using an aqueous suspension with a high solids loading of glass powder. It will be demonstrated how the rheological behaviour of the glass paste, adjusted by adding disperser and network former, will affect the geometry of the seal in terms of thickness, cross section and planarity in the as-printed and dried state. The subsequent joining

process of steel plates was studied by optical dilatometry under various mechanical loads. SEM and XRD investigations gave information about the changes in cross-section and the crystallization behaviour of the glass during heat treatment. The resulting strength of the seal was analyzed by various fracture mechanical experiments showing fundamental correlations between the process parameters for screen printing, brazing conditions and the microstructure of the glass ceramic.

11:20 AM

(ICACC-HON-033-2018) Silicon-based Ceramics for Sustainable Clean and Efficient Energy Technologies (Invited)

H. Lin*[1]

1. Guangdong University of Technology, School of Electronic and Mechanical Engineering, China

It is forecasted that the total global energy consumption will increase up to 50% from 2007 to 2035 based on the data analyzed and published by the U.S. DOE Information Administration. The driving forces behind the substantial increase in worldwide energy demand include: 1) industrialization and strong economic growth in emerging markets, 2) globalization, and 3) concerns over national energy security. It is well recognized that the use of fossil energy will continue to increase and dominate the global energy market, but that causes an increased concerns about greenhouse gas emissions and global warming, which have negative impact to the long-term use of fossil fuels. Therefore, there is an imminent need for alternative energy resources to meet the worldwide fast-pace growing demand, which now create tremendous new emerging markets for these alternative and nuclear energy technologies. This lecture will review how advanced silicon-based ceramic technologies would help to improve manufacturing and energy generation efficiency and bring renewable energy production closer to reality. The design and implementation of those silicon-based ceramic technologies to clean and efficient gas turbines and next-generation nuclear energy generation will be discussed as well. *Research supported by the Project of Innovative and Entrepreneurial Research Team Introduction Program of Guangdong Province (No.2013G061)

11:40 AM

(ICACC-HON-034-2018) Challenges and Opportunities in Energy and Environmental Remediation, including Hybrid Microwave Technology (Invited)

G. Wicks*[1]

1. Applied Research Center, USA

In our society today, energy generation, storage and distribution, along with potential impacts to the environment, provide critical challenges not only on a national scale, but also in the global arena. Existing technologies and capabilities are believed to be insufficient and a variety of alternatives are being examined. This includes energy generation via solar, wind, nuclear, biomass, geothermal, marine and hydrokinetic hydropower, along with new and improved innovations in the electric grid, hydrogen storage materials and nano-composites. A new type of interdisciplinary, international meeting called MCARE was developed and lead by ACerS and is designed to provide a unique forum to address common materials challenges throughout the energy community. The MCARE symposia continue to address our global energy materials needs and will be overviewed. One of the most unique and interesting energy related concepts, involves microwave technology. Hybridization of this technology has been developed and is now being applied to a variety of waste remediation needs. This includes disposition of hazardous and nuclear wastes, treatment of infectious medical wastes, recycling of rubber, destruction of e-wastes and reclamation of the precious metals within, decontamination of C&B agents, and treatment of effluents. This and some of its many potential applications, will be discussed.

S1: Mechanical Behavior and Performance of Ceramics & Composites

Interlamainar and Interfacial Properties

Room: Coquina Salon D
Session Chairs: Rabih Mansour, Teledyne Scientific Company; Emmanuel Maillet, GE Global Research

1:30 PM

(ICACC-S1-038-2018) Microstructure-based modeling of the interlaminar tensile strength of ceramic matrix composites

M. Moscinski*[1]; A. Cerrone[1]; P. Meyer[1]; E. Maillet[1]; D. Dunn[1]

1. GE Global Research, USA

Interlaminar strength is one of the critical properties to understand when designing components with a layered composite material such as ceramic matrix composites (CMCs). Inherently, layered materials tend to be susceptible to interlaminar damage caused by forces trying to peal apart the various layers of material. The actual strength is a strong function of the microstructural composition which includes the properties of the constituents as well as their relative spatial locations. The presentation will describe a damage based FEA approach for calculating the influence microstructural variations have on the resulting interlaminar strength properties. These variations include fiber size, coating thickness, fiber spacing along with the properties of the fibers, coatings and matrix material.

1:50 PM

(ICACC-S1-039-2018) A Scaled Four Point Flexure Test Method for Interlaminar Tensile Strength Measurement in Ceramic Matrix Composites

S. C. Zunjarrao[2]; N. Janakiraman[2]; D. Patro[2]; M. Mathivanan[2]; S. Subramanian*[1]; D. Dunn[1]; Y. Zhou[3]; D. Carper[3]

1. GE Global Research, USA
2. GE JFWTC, India
3. GE Aviation, USA

The interlaminar tensile strength (ILTS) of 2D laminated ceramic matrix composites (CMCs) is an important design parameter since these materials are vulnerable to failure in the through thickness direction. However, measurement of ILTS is challenging, particularly due to challenges in specimen fabrication and the low strength/strain capability of these materials. A scaled four-point flexure test method is evaluated and proposed here to address the industrial need for a test method with simplified specimen geometry, less machining and minimal material wastage. Volume scaling effects are evaluated by testing specimen of different sizes under flexure and performing a Weibull strength-size scaling analysis. Finite element analysis is employed to obtain the specimen stress state in the coupon under 3-point and 4-point bending conditions and to predict the failure load. The proposed test method is also amenable to fatigue loading and high temperature testing.

2:10 PM

(ICACC-S1-040-2018) Interlaminar Fracture Properties of 2D Woven Ceramic Matrix Composites at Room and Elevated Temperatures

R. Mansour*[1]; Y. P. Singh[2]; G. N. Morscher[2]

1. Teledyne Scientific Company, Composite Materials, USA
2. University of Akron, Mechanical Engineering, USA

Interlaminar fracture properties play an important role in predicting failure of structural components for CMC materials. Elevated temperatures induce more severe conditions for interlaminar properties resulting in a weaker interlaminar toughness. The main challenges associated with measuring interlaminar fracture toughness are the ability to measure crack growth without visual

observation and to develop an experimental setup that can be used at both room and elevated temperatures. Hence, a non-visual crack monitoring technique has been successfully developed to estimate crack lengths in CMC composites using electrical resistance. For mechanical testing, a wedge-loaded double cantilever beam method has been introduced and utilized to compare interlaminar fracture properties for different CMC systems at room and elevated temperatures (815 °C and 1200 °C).

2:30 PM

(ICACC-S1-041-2018) Asymmetric Four Point Bending Test Method for Interlaminar Shear Strength in Ceramic Matrix Composites

S. C. Zunjarrao[1]; M. Kashfuddoja[1]; D. Patro[1]; S. Subramanian*[3]; Y. Zhou[2]; D. Carper[2]

1. GE JFWTC, India
2. GE Aviation, USA
3. GE GRC, USA

Asymmetrical Four Point Bend test method is proposed for measurement of interlaminar shear strength (ILS) of ceramic composites. The current standard ASTM test method (ASTM C1425) for ILS strength of composites uses a double edge notched compression coupon. Large variation in measured strength is observed with this method, possibly due to machining variability and damage at the notches. The proposed test method for ILSS is adapted from ASTM C1469 Standard Test Method for Shear Strength of Joints of Advanced Ceramics. This test method does not require any machining of notches and the sample size requirement is much smaller than the ASTM test method. The shear loading in this method is similar to the standard short beam shear test (ASTM D2344) with higher shear to tensile ratio compared to SBS with AFBP. Using finite element analysis, coupon geometry and the distance between the loading and support pins was optimized to maximize shear and minimize tensile and compressive stresses on the specimen. It was found that the variability in the measured ILSS strength was lower with this method compared to the ASTM standard method using the DNC specimen. In addition, the value of ILSS measured using AFPB method was found to be consistently higher than that measured using DNC coupons. It was also found that specimen preparation did not have significant effect on the measured strength.

2:50 PM

(ICACC-S1-042-2018) In-situ study on SiC/Si interfacial strength of reaction bonded SiC/Si composites

C. Hsu*[1]; Y. Zhang[1]; P. Karandikar[1]; F. Deng[1]; C. Ni[1]

1. University of Delaware, USA

Reaction bonded SiC/Si composite (RBSC) has been utilized in aerospace and military industries due to its superior thermal and mechanical properties, particularly in extreme environment. The microstructure of RBSC composite was found to be 6H-SiC, 3C-SiC, and residual Si phases. Near the SiC/Si interface, amorphous Si was detected. The SiC/Si interface is an essential region of the RBSC composites and has attracted considerable attention in research. However, obtaining the intrinsic mechanical properties of the interface at nanoscale remains to be an experimental challenge. To investigate the mechanical properties of SiC/Si interface at the nanoscale, a method of crafting adequate tensile specimen of the interface was successfully established. A home-built nanomanipulation tensile testing system was able to perform within a field emission scanning electron microscope (FE-SEM). This testing system is currently the only method to probe the mechanical properties of nano-size dog-bone fabricated from the bulk final product. The average SiC/Si interfacial strength was measured to be 1588.4 MPa and the fractured surface showed non-crystalline form of Si by high-resolution transmission electron microscope (HRTEM). The results are in good agreement with the tensile strength data in the literature and also represent the first successful in-situ measurement of SiC/Si interfacial strength.

3:30 PM

(ICACC-S1-043-2018) Microcrack evaluation of an orthogonal 3-D woven SiC/SiC composite using a digital image correlation method with microscopic speckle patterns

M. Sato*[1]; T. Ogasawara[1]; T. Aoki[2]

1. Tokyo University of Agriculture and Technology, Mechanical Systems Engineering, Japan
2. Japan Aerospace Exploration Agency, Advanced Composite Research Center, Institute of Aeronautical Technology, Japan

It is known that microcrack propagation occurs in SiC fiber reinforced / SiC matrix composites (SiC/SiC) under tensile load. As a result, fiber/matrix interphase are exposed to air in service condition. The matrix cracks derive the oxidation of the interphase at elevated temperatures, resulting in considerable strength degradation. In addition, microcrack cumulation reduces the stiffness of SiC/SiC composites. Therefore, understanding of micro-cracking behavior is important for structural designing and lifetime estimation of actual parts. The objective of this study is to demonstrate the evaluation of microcrack progression of an orthogonal 3-D woven SiC/SiC composite under tensile load using a digital image correlation (DIC) method. Micro-order speckle patterns were printed on the surface of SiC/SiC specimens using a super fine inkjet printer. The representative speckle pattern was several tens of micron, which made it possible to obtain the full field strain using DIC method in a layer of about 0.2 mm of thickness. The matrix cracks were successfully observed as larger displacement gradient, namely higher strain regime because of crack opening displacement (COD). The stress of crack initiation was determined comparing COD with an appropriate threshold. The experimental results were directly compared with the numerical results.

3:50 PM

(ICACC-S1-044-2018) Combining in-situ synchrotron X-ray microtomography and acoustic emission to characterize damage evolution in ceramic matrix composites

E. Maillet*[1]; A. Singhal[1]; A. Hilmas[2]; Y. Gao[1]; Y. Zhou[1]; G. Henson[1]

1. GE Global Research, USA
2. University of Michigan, Department of Materials Science & Engineering, USA

This paper reports the first combined use of in-situ synchrotron X-ray microtomography and acoustic emission (AE) during room-temperature mechanical testing of ceramic matrix composites. A detailed characterization of damage initiation and progression is obtained from microtomography, and the relationship between damage and AE is directly observed. A graphical representation of AE data, which has potential for real-time use, is employed to reveal differences in damage progression due to fiber architecture or loading mode. In addition, strong empirical relationships are observed between matrix crack area and AE energy, as well as between fiber breaks and number of AE events. This paper is based upon work supported by ARFL under Contract F865011C5227. Any opinions, findings and conclusions or recommendations expressed in this material are those of the author(s) and do not necessarily reflect the views of the USAF/AFMC Air Force Research Laboratory.

4:10 PM

(ICACC-S1-045-2018) Evaluation of SiC/SiC CMCs with yttrium disilicate fiber coatings

E. E. Boakye*[1]; P. Mogilevsky[1]; T. Key[1]; T. A. Parthasarathy[1]; R. Hay[2]; M. Cinibulk[2]; S. S. Opeka[1]

1. UES Inc., Materials Science, USA
2. AFRL, Materials and Manufacturing Directorate, USA

Yttrium disilicate ($Y_2Si_2O_7$) coatings are potential oxidation-resistant alternatives to carbon or BN fiber coatings for SiC/SiC CMCs. Compared to our previous coatings, the coating uniformity was optimized and its effect on the composite strength was evaluated. Hi-Nicalon-S/$Y_2Si_2O_7$/SiC minicomposites with improved $Y_2Si_2O_7$ (2nd generation) coatings were formed by polymer infiltration pyrolysis (PIP) and characterized by tensile tests, fiber push-in tests, TEM, and SEM fractography. Composites formed with improved Y_2Si_2O7 fiber coatings were effective in crack deflection and fiber pullout and had higher strengths than minicomposites with the 1st generation fiber coatings.

4:30 PM

(ICACC-S1-046-2018) Hardness and stiffness of long fiber reinforced C/C-SiC composites

D. Koch*[1]; Y. Shi[1]

1. Institute of Structures and Design, Ceramic Composites and Structures, Germany

The mechanical properties of long fiber reinforced ceramic matrix composites are generally investigated in in-plane orientation where the fibers can help to enhance the mechanical properties. In out of plane however, the fibers are loaded perpendicular to their longitudinal axis and therefore they cannot contribute on load bearing in a significant manner. Nevertheless, in various applications the mechanical properties in out of plane orientation are of interest e.g. if parts are screwed together or if the material is used in bearings. The hardness and the Young's modulus in out of plane orientation are investigated on liquid silicon infiltrated C/C-SiC composites. For hardness tests a hemispherical indenter is used in order to evaluate residual indentation. Additionally the load displacement curves are used for calculation of hardness and stiffness. In parallel instrumented compression tests in out of plane orientation are performed at small compression specimens. It turns out that the occurring damage during hardness indentation is not to be interpreted accurately as the indenter induces delamination and large volume failure of matrix in a large number of layers. Therefore the load displacement curves are used to calculate hardness. These curves also allow the calculation of stiffness perpendicular to the fiber orientation. The results are then compared to the stiffness values achieved from compression tests.

S3: 15th International Symposium on Solid Oxide Fuel Cells (SOFC): Materials, Science and Technology

Cathode Materials I

Room: Crystal
Session Chair: Xingbo Liu, West Virginia University

1:30 PM

(ICACC-S3-034-2018) The Effects of Composition and Operating Conditions on the Microstructure and Performance of LSM-Based SOFC Cathodes (Invited)

M. R. De Guire*[1]

1. Case Western Reserve University, Materials Science and Engineering, USA

SOFC button cells with cathodes of different lanthanum-strontium manganite (LSM, $(La_{1-x}Sr_x)_{1-y}MnO_{3\pm\delta}$) compositions were operated for 500 h or more under conventional and aggressive conditions. The LSM compositions differed primarily in the degree of manganese excess (Mn/(La+Sr)>1). The cells underwent durability testing (voltage versus time at constant current density) with intermittent linear-sweep voltammetry and electrochemical impedance spectroscopy measurements. Post-test microstructural analysis consisted of transmission electron microscopy with energy-dispersive x-ray spectroscopy, focused ion-beam scanning electron microscopy, and 3D reconstruction. An intermediate level of excess Mn gave low increase of area-specific resistance (ASR) over time, and also exhibited high microstructural stability (minimal formation of manganese oxides, little cathode densification, and lowest decrease in three-phase boundary (TPB) density). The LSM with the least excess Mn gave the lowest values of ASR in 500 h of testing, but also exhibited significant changes in ASR and in TPB density over time. Significant differences in microstructural evolution at the interfaces with the electrolyte and the cathode current collector were also observed between the cathode compositions.

2:00 PM

(ICACC-S3-035-2018) Microstructure modification in the interfaces between electrode / electrolyte to enhance performance of the anode-supported solid oxide fuel cell

M. Liao[1]; T. Lin*[1]; C. Yeh[1]; H. Kuo[1]; Y. Chen[1]; W. Kao[1]; R. Lee[1]; S. Lee[2]

1. Institute of Nuclear Energy Research, Nuclear Fuels and Materials Division, Taiwan
2. National Central University, Institute of Materials Science & Engineering, Taiwan

As SOFC perovskite cathode, Lanthanum strontium manganite (LSM) exhibits advantages such as good chemical stability, high electronic conductivity and compatible thermal expansion coefficient. Introducing LSM-YSZ composite as functional layer is essential for the limited ion conductivity of LSM. A few investigations indicate that the LSM-YSZ benefits an increase in power with operation time. In this study, long-term performance evaluation has been executed on the anode supported cell with NiO-YSZ anode, YSZ electrolyte, LSM-YSZ functional layer, and LSM current collecting cathode. The cell performance measurement results show a significant enhancement after long-term operation. The maximum power density is ~ 280 mW cm^{-2} at the initial start-up stage. It dramatically increases to ~ 520 mW cm^{-2} after the 100 hours operation at 800 °C with 400 mA cm^{-2}. The measurement results indicate that a higher constant current loading is beneficial for the improvement of cell performance. Microstructure investigations by transmission electron microscopy (TEM) has been conducted and the LSM-YSZ composite with small grain size of several tens nano-meter structure exists in a local region near interface. It is evidenced that the nano-sized LSM-YSZ which may be attributed to the high current loading can increase the triple phase boundary (TPB) density.

2:20 PM

(ICACC-S3-036-2018) Effect of Temperature and Overpotential on Degradation of LSM-YSZ and LSCF Electrodes during Reversing Current Operation

M. Y. Lu*[1]; J. G. Railsback[1]; S. Barnett[1]

1. Northwestern University, Materials Science and Engineering, USA

Reversible solid oxide cells have promising application in large scale energy storage, but a more complete understanding of their degradation behavior is needed prior to commercial development. The oxygen electrode has previously been found to be a point of failure in both electrolysis and reversible operation. Previously, current switched life-tests of two common oxygen electrodes, $La_{0.8}Sr_{0.2}MnO_{3-\delta}$-$Zr_{0.84}Y_{0.16}O_{2-\delta}$ (LSM-YSZ) on YSZ electrolytes and $La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_{3-\delta}$ (LSCF) on $Ce_{0.9}Gd_{0.1}O_{2-\delta}$ electrolytes, were conducted at a range of current densities at typical operating temperatures (800 and 700 °C respectively) using symmetric cells. While some degradation trends could be established, it was not possible to clearly separate the roles of current density and

overpotential or extrapolate behaviors to other temperatures. Here we present a series of current switched ~1000 h life tests (current direction switched every 6 h, with current densities ranging from 0.5 – 1.5 A/cm^2) on the same material sets at varying temperatures (650 - 750 °C for LSCF and 750 - 800 °C for LSM-YSZ), probing the role of temperature and overpotential. EIS life-test data are presented along with post-test microstructural analysis. Degradation of both LSM-YSZ and LSCF is accelerated at lower temperatures where the overpotentials are higher.

Cathode Materials II

Room: Crystal

Session Chair: Minfang Han, Tsinghua University

3:20 PM

(ICACC-S3-037-2018) Microstructure Degradation of LSCF Cathodes for Solid Oxide Fuel Cells

A. Zekri*[2]; G. Sourkouni[1]; N. Klaassen[2]; C. Gutsche[2]; P. Michalowski[2]; C. Argirusis[1]; J. Parisi[2]; T. Plaggenborg[2]; M. Knipper[2]

1. Clausthal Centre for Materials Technology, Germany
2. Institute of Physics, Department of Energy and Semiconductor Research, Germany

The performance of solid oxide fuel cells (SOFCs) as well as their durability are dependent on the microstructure of the electrodes. In order to understand the microstructural evolution in SOFC cermet cathodes, different cells were thermally aged at 850 °C under dry and humid conditions for different operating times (100 h, 1000 h). The effect of the humidity and aging time on the microstructure of LSCF based cathodes was analyzed. Similar cells were also electrochemically aged under the same operating conditions and with a current density of J=220 mA.cm^{-2}. In this study, Raman, cross section EDX, XRD and XPS were employed in order to detect the chemical changes in the microstructures. The results showed a clear Sr-segregation in some regions of the cathodes. The Sr-segregation was accompanied by a noticeable formation of the $SrCrO_4$ phase, mainly on the surface of the aged cathodes. This phase formation of $SrCrO_4$ becomes more significant under humid conditions. Furthermore, the results obtained from different techniques were combined in order to get a deeper insight into the distribution of the $SrCrO_4$ within the LSCF based cathode. The Sr-segregation and the presence of chrome contamination in the aged cathodes led to the formation of secondary phases such as $SrCrO_4$, which is considered as one of the main degradation mechanism in the SOFC cathode.

3:40 PM

(ICACC-S3-038-2018) The use of thermodynamic modelling for tuning microstructure and composition of $(La_{0.8}Sr_{0.2})_{0.98}Cr_xFe_{1-x}O_{3\pm\delta}$

H. Sabarou*[1]; Y. Zhong[1]

1. Florida International University, Mechanical & Materials Engineering, USA

The current research takes advantage of thermodynamic modelling results to tune the microstructure and regional compositions of $(La_{0.8}Sr_{0.2})_{0.98}Cr_xFe_{1-x}O_{3\pm\delta}$ (x=0.3, 0.5, 0.7) in the purpose of enhancing its oxygen transport membrane (OTM) properties. The samples are synthesized by sol-gel method, annealed, and heat treated under reducing atmospheres above 1000°C. The microstructure and changes in regional compositions have been characterized by scanning electron microscopy and Energy-dispersive X-ray spectroscopy. Based on the modelling results, different oxygen partial pressure and temperatures have been chosen to improve densification of the samples and dissolve detrimental secondary phases into the perovskite structure. The quality of the tuning process has been measured by thermodynamic modelling and X-ray diffraction. The results show that the tuned samples can have reliable requirements for OTM application with higher densification, reliable chemical stability, and promising electrical and thermal properties.

4:00 PM

(ICACC-S3-039-2018) Evaluation of Cathode Materials for Proton Conducting Intermediate Temperature Solid Oxide Fuel Cells

S. Sun*[1]; Z. Cheng[1]

1. Florida International University, Mechanical and Materials Engineering, USA

The cathode is often considered to be the rate-limiting factor in solid oxide fuel cells (SOFC), especially for operation at intermediate temperatures (~400-700ºC). In that temperature range, oxide electrolytes with proton conductivity such as acceptor-doped Ba(Ce, Zr)O_3 (e.g. $BaZr_{0.1}Ce_{0.7}Y_{0.1}Yb_{0.1}O_3$ or BZCYYb) are attractive due to their potential for higher ionic conductivity than oxide-ion conducting electrolytes especially when temperature gets lower. However, whether proton conductivity would also offer additional benefits in terms of cathode reaction kinetics or electrocatalytic activity versus conventional cathode reactions based on oxygen ion has not been settled so far. In this study, several promising cathodes including $Ba_{0.5}Sr_{0.5}Co_{0.8}Fe_{0.2}O_{3-\delta}$ (BSCF), BSCF-BZCYYb composite, as well as $SrCo_{0.8}Nb_{0.1}Ta_{0.1}O_{3-\delta}$ (SCNT) are evaluated for cathode reaction kinetics at intermediate temperatures down to 450ºC using electrolyte supported cell based on BZCYYb proton conducting electrolyte. The implication of the investigations will be discussed aiming at revealing the best strategy for designing and improving the cathode for proton conducting intermediate temperature SOFC.

S5: Next Generation Bioceramics and Biocomposites

Bioceramics and Biocomposites I

Room: Coquina Salon B

Session Chairs: Fiorenzo Vetrone, Institut National de la Recherche Scientifique; Alberto Vomiero, Lulea University of Technology

1:30 PM

(ICACC-S5-004-2018) Tuning light emission properties in composite ceramics for advanced applications (Invited)

A. Vomiero*[1]

1. Lulea University of Technology, Engineering Sciences & Mathematics, Sweden

Light emission from composite ceramics can be finely tuned by proper adjustment of chemical composition, size and morphology of the single components, and can be profitably used in a series of advanced applications like nano-thermometry, bio-imaging and bio-sensing. Key element for light emission is the modulation of the final electronic band structure of the composite systems. We will illustrate different strategies to obtain the desired optical properties in different complexes. One of the most interesting systems is composed of semiconducting nanocrystals exhibiting quantum confined effects (the so-called quantum dots, QDs), which act as light absorbing materials, and generate excitons as a consequence of photon absorption. In this case, the optical properties are determined by the dynamics of hole/electron dissociation and recombination, and the structure of the interface between the core and the shell regulates such dynamics. Another option is to apply core-shell systems, in which the core composition can be adjusted to offer multiple functionalities. We will illustrate an example of magnetofluorescent nanoparticles assembled from terbium and gadolinium 1,3-diketones, where two different functionalities lead to improved sensitivity.

1:50 PM

(ICACC-S5-002-2018) Ceramic Nanosensors and Single Exhale Breathalyzers for Asthma and Flu Monitoring (Invited)

P. Gouma*[1]

1. University of Texas, Arlington, MSE, USA

This work describes the novel processing of ceramic materials and nanosensors for the non-invasive detection and monitoring of diseases. Single gas sensing elements are useful in detecting certain diseases -asthma monitoring is based on NO biomarker detection by γ-WO_3-based nanosensors. A wireless prototype of a portable, hand-held breathalyzer based on this technology was used to carry out single exhale measurements of NO with success. On the other hand, monitoring of viral infections is much more useful if more than one exhaled breath marker is followed simultaneously over time. A common feature of the inflammatory response in patients who have actually contracted influenza is the generation of a number of volatile products of the alveolar and airway epithelium. These products include a number of volatile organic compounds (VOCs) and nitric oxide (NO). Unlike the signals from other instruments that have been used to profile and quantitate gaseous analytes in breath, which are interpretable only by research personnel, our resistive ceramic sensors have both specificity/selectivity and sensitivity, so that the significance of the digital data can be recognized with a high degree of confidence. The advances presented here in ceramic materials and devices based on them are enabling the development of inexpensive and affordable personalized medical diagnostic tools.

2:10 PM

(ICACC-S5-003-2018) Hybrid Nanoparticles as New Theranostics for Cancer Detection and Therapy (Invited)

M. Wang*[1]

1. The University of Hong Kong, Department of Mechanical Engineering, Hong Kong

Millions of people around the world suffer from cancer and the early detection and effective treatment of cancers are vital for their survival and recovery. Advanced diagnostic techniques such as magnetic resonance imaging are now routinely used and cancer treatments such as surgery and radiotherapy save millions of lives. However, these diagnostic and therapeutic techniques have limitations. Nanotechnology has made enormous impact in all fields, and nanoparticles have attracted great attention for their potential as diagnostic and/or therapeutic tools in oncology due to their unique properties. Combining diagnosis and therapy in multifunctional nanodevices (the so-called "theranostics") can provide huge benefits to both patients and healthcare systems. New theranostics should have the capability for early cancer detection and provide multiple functions for cancer cell targeting, sensing/imaging, and cancer therapy/therapies. This lecture will present our designs and investigations of new theranostics, which are based on gold nanoparticles, gold nanorods or gold-silver nanoparticles and include hybrid nanoparticles such as metal-polymer nanoparticles, metal-silica shell-metal satellite nanoparticles, and metal-hollow silica yolk-shell nanoparticles. Anticancer drugs or plasmids are encapsulated. The theranostics can provide chemotherapy, photothermal therapy, gene therapy, or combined therapies.

2:30 PM

(ICACC-S5-001-2018) NIR Emitting Nanoplatforms: Harnessing Light for Applications in Biology and Nanomedicine (Invited)

F. Vetrone*[1]

1. Institut National de la Recherche Scientifique, Cente Énergie, Matériaux et Télécommunications, Canada

The ability to stimulate luminescent inorganic nanoparticles with near-infrared (NIR) light has made them attractive optical bioprobes for a number of applications in biology and nanomedicine. In fact, the biggest impact of such materials would be in the field of disease diagnostics and therapeutics, now commonly referred to as theranostics. The use of NIR light for excitation mitigates some of the drawbacks associated with high-energy light (UV or blue) excitation, for example, little to no background autofluorescence from the specimen under investigation as well as no incurred photodamage. Moreover, one of the biggest limitations is of course, that of penetration. As such, NIR light can penetrate tissues much better than high-energy light especially when these wavelengths lie within the three so-called biological windows. Thus, significant strides have been made in the synthesis of inorganic nanomaterials whose excitation as well as emission bands lie within one of these three optically transparent biological windows. Here, we present the synthesis of various NIR excited (and emitting) inorganic core/shell nanostructures and demonstrate their potential use in nanomedicine. Furthermore, we will show how such nanoparticles can be used as building blocks towards developing multifunctional nanoplatforms for simultaneous detection and therapy of disease.

3:10 PM

(ICACC-S5-005-2018) Controlled Release of Growth Factor from Bijels-derived Hybrid Hydrogel Membranes

M. Wang*[1]; H. Sun[1]

1. The University of Hong Kong, Department of Mechanical Engineering, Hong Kong

Bicontinuous interfacially jammed emulsion gels (bijels) are newly discovered structures consisting of two interpenetrating continuous liquid phases. Bijels can be used as templates for fabricating bijels-derived materials, maintaining the bicontinuous microstructure in the solid form. The bicontinuous architecture makes bijels-derived materials attractive for biomedical applications. In tissue engineering, the local delivery of growth factors enhances tissue regeneration. This study investigated the fabrication of hybrid hydrogel membranes of biocompatible materials via bijels templates and the incorporation and release of vascular endothelial growth factor (VEGF) from the membranes. Bijels membranes were made using the solvent transfer-induced phase separation technique. Hexanedioldiacrylate was the oil phase in bijels and was cured by UV. After curing, bijels membranes were immersed in VEGF-containing Na-alginate solutions for forming continuous alginate phase in membranes, and alginate was then crosslinked by a $CaCl_2$ solution. Release experiments of VEGF were performed by immersing membranes in a simulated body fluid for different times. VEGF concentrations in the test liquid were measured using human VEGF ELISA kit assay and the VEGF in vitro release profiles were established. Controlled released of VEGF could be achieved with optimized processing parameters for hybrid hydrogel membranes.

3:30 PM

(ICACC-S5-006-2018) Magneto-Plasmonic nanoplatform in photothermal therapy

C. Multari*[1]; M. Miola[1]; F. Laviano[1]; R. Gerbaldo[1]; G. Pezzotti[2]; D. Debellis[3]; E. Verne'[1]

1. Politecnico di Torino, DISAT, Italy
2. Kyoto Institute of Technology, Japan
3. Italian Institute of Technology, Electron Microscopy Facility, Italy

Nanoparticles have been widely studied as new therapeutic and diagnosis tool for cancer treatment. Particularly, magneto-plasmonic nanoplatforms(MPNPs), composed by superparamagnetic iron oxide nanoparticles(SPIONs) and gold nanoparticles(GNPs), are of great interest because they possess both magnetic and plasmonic properties. A reproducible synthesis method is used to obtain MPNPs made of a magnetic core and an external gold decoration. The correct formation of MPNPs, their superparamagnetic and their plasmonic behaviors are detected by physical, magnetic and optical characterization respectively. A cytotoxicity study is performed on

healthy and cancer cells exposed to MPNPs by using a green laser source in order to evaluate the ability of GNPs to convert absorbed light into thermal energy. Cell tests confirm that MPNPs causes an important damage of cancer cells while is not resulting dangerous in normal cells. This indicate that the MPNPs allows to convert the light received into heating which can destroy cancer cells, due to their high heat sensitivity. These MPNPs are a new approach to cancer therapy because they are able to arrive directly in tumor site, to be used as drug delivery system, contrast agent and contemporaneously as photosensitizer for photothermal therapy.

3:50 PM

(ICACC-S5-007-2018) Composite nanostructures as a means for bioimaging, nanothermometry, photothermal therapy and controlled magnetic heating (Invited)

D. H. Ortgies*[2]; U. Rocha[3]; L. de la Cueva[4]; D. Cabrera[4]; G. Salas[4]; F. J. Teran[4]; A. S. Vanetsev[5]; M. Rähn[5]; V. Sammelselg[5]; Y. V. Orlosvkii[5]; D. Jaque[1]

1. Universidad Autonoma de Madrid, Física de Materiales, Spain
2. Instituto Ramón y Cajal de Investigación Sanitaria IRYCIS, Spain
3. Universidade Federal de Alagoas, Instituto de Física, Brazil
4. iMdea Nanociencia, Spain
5. University of Tartu, Institute of Physics, Estonia

The advances in the design of various nanoparticles that can generate heat locally has rekindled interest in heat-based (hyperthermia) treatments. Most commonly employed as nanoheaters are plasmonic (metal nanoparticles), rare-earth-doped nanoparticles, which are both activated through light, or magnetic nanoparticles especially those based on iron oxide, which generate heat under magnetic excitation. Nevertheless, their clinical application is still hampered by the lack of in situ thermal feedback, which would allow to measure the local temperature in the tissue or tumor, which can be drastically different from the surface temperature, usually measured with thermal cameras. In order to overcome this limitation we combined different types of nanoheaters with luminescent nanoparticles, whose luminescence is influenced by the in situ temperature and who therefore act as nanothermometer, in composite nanostructures based on encapsulation with the biocompatible polymer PLGA. We synthesized and characterized different combinations and showed that their heating capability as well as their luminescence-based imaging and temperature sensing were maintained. Furthermore, we demonstrated with experiments in biological tissues their applicability for thermal therapies with control of the intratissue heat delivery due to the included nanothermometer.

4:10 PM

(ICACC-S5-008-2018) Antifogging Diamond-like Carbon Coatings for Laparoscope Lenses (Invited)

A. Evans[1]; R. L. Leonard*[1]; J. A. Johnson[1]

1. University of Tennessee Space Institute, Mechanical, Aerospace, and Biomedical Engineering, USA

As surgical technology has advanced, the use of the laparoscope has become more widespread. Laparoscopes allow for surgeries to be completed with smaller incisions, resulting in decreased recovery time for patients. Although the laparoscope has improved many surgical procedures, a drawback is that laparoscopic lenses can become obscured by fogging or mucus adhesions, reducing the surgeon's vision. Diamond-like carbon (DLC) thin films have many favorable properties including chemical inertness, exceptional scratch resistance, and optical transparency. By doping with the appropriate elements, DLC may become highly hydrophilic, which prevents fogging and discourages protein adhesion. Through pulsed laser deposition, using multi-component targets, we have prepared novel silicon and oxygen-doped DLC coatings for laparoscope lenses. We report the properties of these films relating to their suitability as anti-fogging optical coatings, including water contact angle and surface energy, optical transmission, film adhesion, and scratch resistance. The biocompatibility of these films will also be discussed.

S6: Advanced Materials and Technologies for Direct Thermal Energy Conversion and Rechargeable Energy Storage

Beyond Lithium Batteries I

Room: Tomoka A

Session Chair: Xiangxin Guo, Shanghai Institute of Ceramics, Chinese Academy of Sciences

1:30 PM

(ICACC-S6-031-2018) Amorphous Li_2O_2: Chemical Synthesis and Charge Transport Properties (Invited)

Z. Peng*[1]

1. Changchun Institute of Applied Chemistry, China

When aprotic Li-O_2 batteries discharge, the product phase forms in the cathode that often contains two different morphologies, i.e., crystalline and amorphous Li_2O_2. The morphology of Li_2O_2 has been recognized to impact strongly on the electrochemical performance of Li-O_2 cells in terms of energy efficiency and rate capability. Crystalline Li_2O_2 is readily available and its properties have been studied in depth under the context of Li-O_2 batteries. However, little is known about the amorphous Li_2O_2 because of the rarity of the compounds with high purity. Here, amorphous Li_2O_2 has been synthesised by a rapid reaction of tetramethylammonium superoxide and $LiClO_4$ in solution, and its amorphous nature has been confirmed by a range of complementary techniques. Compared with its crystalline siblings, amorphous Li_2O_2 demonstrates enhanced charge transport properties and increased electro-oxidation kinetics, manifesting itself a desirable discharge phase for high-performance Li-O_2 batteries.

2:00 PM

(ICACC-S6-032-2018) Towards better Na-O_2 batteries: Factors controlling the electrochemical behavior (Invited)

A. Grimaud*[1]

1. College de France - CNRS, France

Increasing the energy storage demands motivate the search for lower-cost, higher-capacity and sustainable batteries. Metal-air batteries are of interest because of their large theoretical capacity, which relies on the redox reaction of oxygen rather than intercalation reactions such as those that occur in Li-ion and Na-ion batteries. Although the capacity is lower than for the well-known Li-O_2 system, Na-O_2 battery has recently attracted attention because of its higher-energy efficiency and chemical reversibility due, in part, to the lack of side-reactions when compared to Li-O_2 batteries. Nevertheless, even though promises were demonstrated, a lack of understanding of the underpinning science hampers the development of this system which remains in its infant state. Hence, critical differences can be observed between the Na-O_2 and the Li-O_2 systems, from different discharge products formed in reduction (Li_2O_2 vs NaO_2), to different solubility and solvation properties of the intermediates of the reaction as well as different interactions with the surface of the electrode. In this talk, we will unravel the different mechanisms at play during the formation of the discharge product as well as its oxidation. From this mechanistic study, we will then summarize the critical parameters that control the capacity, the cyclability and the stability of this system in order to further optimize cell components.

2:30 PM

(ICACC-S6-033-2018) Functional and smart electrolytes for rechargeable Zn batteries (Invited)

G. Cui*[1]; J. Zhao[1]

1. Qingdao Institute of Bioenergy and Bioprocess Technology, Chinese Academy of Sciences, China

Zn batteries constitute an important area of electrochemical energy systems, particularly for low cost and low power applications. However, the rechargeable Zn batteries suffer from capacity loss and internal-shorts, due to zinc redistribution and the solubility of discharge products. One method for overcoming these issues is novel electrolyte design. We developed strategies to optimize the recharging process by using "water-in-salt"-based and molten solvent-based electrolytes. Compared with traditional aqueous electrolytes, our electrolytes impart Zn electrodes with highly improved plating/stripping property and cycling stability. A series of high-voltage cathodes can be successfully coupled with the Zn-based anode for developing high-performance rechargeable Zn batteries. Besides the investigation on the reversible Zn chemistry, we further presented a flexible Zn battery system with a smart cooling-recovery function by using a thermoreversible polymer electrolyte. In contrast to conventional flexible batteries, when this battery system was exposed to extreme deformations, a simple cooling process can repair the fractured electrode-electrolyte interface, thus enabling in situ restoration of the electrochemical performance. This cooling-recovery function will enable new applications in advanced electronic systems with a satisfactory level of architectural durability.

Beyond Lithium Batteries II / Solid Electrolytes and All-solid-state-batteries III

Room: Tomoka A

Session Chair: Naoaki Yabuuchi, Tokyo Denki University

3:20 PM

(ICACC-S6-034-2018) NASICO-typed $Na_3V_2(PO_4)_2F_3$ as a Promising Cathode for Aqueous Zinc Ions Batteries (Invited)

W. Li[1]; K. Wang[1]; K. Jiang*[1]

1. Huazhong University of Science and Technology, China

Aqueous batteries possess the distinctive advantages of low-cost, high safety, and assemble flexibility. In the family of aqueous batteries, bivalent zinc-based batteries are attractive due to their low cost, high capacity, and environmental benignity, compared with the lead-acid and nickel-metal hydride batteries. At present, the cathodes for aqueous Zn-ion batteries are dominated by MnO_2 and Prussian blue analogues due to their open-framework structures. As a potential candidate, NASICO-typed $Na_3V_2(PO_4)_2F_3$ possesses large channel and rapidly ionic diffusion capability, which is appealing for Zn^{2+} ions storage. During the CV scan, Na^+ ions extract from the host structure in the initial cathodic scan and Zn^{2+} ions insert in the following anodic scan, and then reversible uptake and removal of Zn^{2+} ions occur in the subsequent scans. There is no obvious shift of insertion/extraction potentials for Zn^{2+} ions compared with those of Na^+ ions, and the comparative peak areas and reproducible curves of the repeated cycling are indicative of good reversibility of Zn-ions batteries. These preliminary results reveal that $Na_3V_2(PO_4)_2F_3$ can be served as a promising cathode for Zn-ions batteries.

3:50 PM

(ICACC-S6-035-2018) Garnet Electrolytes for Rechargeable Solid State Lithium Batteries

X. Guo*[1]

1. Shanghai Institute of Ceramics, Chinese Academy of Sciences, China

The solid electrolytes based on the powders with high ionic conductivity, chemical as well as electrochemical stability are crucial for development of solid state lithium batteries (SSLB), which may offer higher energy density, longer cycle life, and better safety than the conventional Li-ion batteries. Recently, we have succeeded on fabrication of highly conducting garnet $Li_7La_3Zr_2O_{12}$ (LLZO)-based powders, by element doping and parameter optimization. Using these powders, the high-dense ceramic electrolytes and the flexible membrane electrolytes have been fabricated, which are powerful for construction of high-performance SSLB.

4:10 PM

(ICACC-S6-036-2018) Electrode Fabrication for All-solid-state Rechargeable Lithium Batteries using $Li_{6.25}Al_{0.25}La_3Zr_2O_{12}$ Solid Electrolyte (Invited)

H. Munakata*[1]; J. Wakasugi[1]; K. Kozuka[1]; T. Kimura[1]; M. Shoji[1]; K. Kanamura[1]

1. Tokyo Metropolitan University, Japan

$Li_{6.25}Al_{0.25}La_3Zr_2O_{12}$ (Al-LLZ) has been focused on as a promising ceramic solid electrolyte for all-solid-state rechargeable lithium batteries since it has a relatively high Li^+-ion conductivity of ~10^{-4} S cm^{-1} at room temperature and a wide electrochemical window enabling to use both lithium-metal anode and high potential cathodes. In this study, we tried to optimize the electrode fabrication for all-solid-state rechargeable lithium batteries using Al-LLZ and lithium metal anode. For the anode side, a gold interlayer was introduced into the interface between Al-LLZ and lithium metal anode to decrease their interfacial resistance. The interfacial resistance was decreased to 150 ohm from 1200 ohm by the introduction of gold interlayer with a heat treatment at 150 °C, in which Li-Au alloy formation occurred. The specific capacity of cathode is generally smaller than that of lithium-metal anode, so that the cathode should be thick to reduce the capacity difference between cathode and lithium-metal anode. Aerosol deposition (AD) method is effective to make thick cathode for all-solid-state rechargeable lithium batteries. The details of this method will be also discussed.

4:40 PM

(ICACC-S6-037-2018) Field Assisted Sintering of Garnet-type $Li_7La_3Zr_2O_{12}$ Solid Electrolytes with Enhanced Ionic Conductivity for All-solid-state Batteries (Invited)

F. Chen*[1]; J. Li[1]; X. Xiang[1]; W. Zha[1]; D. Yang[1]; Y. Zhang[1]; Q. Shen[1]; L. Zhang[1]

1. Wuhan University of Technology, China

In the research of solid electrolytes, cubic $Li_7La_3Zr_2O_{12}$ (LLZO) gains widespread attention because of its high room temperature ionic conductivity, good chemical and electrochemical stability. However, cubic LLZO is not stable in room temperature and is hard to synthesize due to Li evaporation during high temperature sintering. In our research, phase transition, cubic phase stabilization with high valence element doping and ionic conduction of LLZO were firstly investigated via molecular dynamic simulation. Then using field assisted sintering technology (FAST) and high valence Al doping to reduce Li loss and stabilize cubic phase, the cubic Al-doped LLZO with ionic conductivity up to 5.7×10^{-4}Scm^{-1} and relative density >99.8% was obtained. To further improve ionic conductivity, the effect of two main factors, framework structure and lithium concentration, on the ionic conduction were investigated via both molecular dynamic simulation and FAST. The framework structure and lithium concentration are both regulated through Ta doping. As a result, the ionic conductivity of Ta-doped LLZO could be improved to >10^{-3}S/cm. Finally, solid-solid interfaces between electrodes and solid electrolyte were optimized by using PEO/$LiCoO_2$ composite cathode and Ta-doped LLZO to assemble all-solid-state battery.

S7: 12th International Symposium on Functional Nanomaterials and Thin Films for Sustainable Energy Harvesting, Environmental, and Health Applications

Inorganic Materials and Composites for Energy Harvesting and CO2 Conversion

Room: Coquina Salon C

Session Chairs: Qi Li, Institute of Metal Research, Chinese Academy of Sciences; Yujie Xiong, University of Science and Technology of China

1:30 PM

(ICACC-S7-033-2018) Tailoring Bimetallic Nanocatalysts toward Solar-Chemical Energy Conversion (Invited)

C. Kuo*[1]; Y. Chuang[2]; D. Cullen[3]; B. Sneed[4]

1. Acadmeia Sinica, Institute of Chemistry, Taiwan
2. National Synchrotron Radiation Research Center, Taiwan
3. Oak Ridge National Laboratory, Materials Science and Techology Division, USA
4. Oak Ridge National Laboratory, Center for Nanophase Materials Sciences, USA

Integration of plasmonics and catalysis in bimetallic nanocrystals provides substantial benefits for the creation of novel catalyst systems. This kind of function-added nanocrystals possess the superiority in delivering light energy to chemical production as well as sensing. In this talk, a synthetic concept for the design of nanocrystals with bi-functions of plasmon and catalysis will be presented. The one-step synthesis of Au-Pd icosahedral nanocatalysts is the first example, including the precise control on their surface structures, compositions and thickness (lattice strain) of shells. By turning the ionic switch of [Cl^-]/[Br^-], the bimetallic nanocatalysts selectively form in alloy and core-shell structures. Their superior catalytic performances in electrochemical fuel oxidation and photoreduction reveal the success in the design of such active and stable icosahedral nanocatalysts. A facile strategy for wearing the noble metal Pt on the Cu nanocubes will be introduced, too. The coating of Pt is site-selective and thus leads to the rhombic dodecahedral core-frame structures. Turning the amount of Pt atoms, the frames would evolve at the 100 corners to become the spiny ones. After treatment with acid, the regular and spiny nanoframes were obtained. The results for oxygen reduction reaction (ORR) and photoreduction eventually show the regular CuPt nanoframes the most cost-efficient catalysts.

2:00 PM

(ICACC-S7-034-2018) Photo-driven activation of CO_2 molecule

R. Solarska*[1]; K. Bienkowski[1]; M. Arasimowicz[1]

1. University of Warsaw, Centre of New Technologies, Poland

Investigations of the photo-induced phenomena enabling to improve and understand fundamental processes involved in CO_2 reduction includes co-assembly of plasmonic and catalytic metal nanoparticles to the build-up of a semiconductor photocathode. Combination of the nanostructures of plasmonic metals, such as silver or gold with other metal electrocatalysts will substantially decrease the energy barriers of the CO_2 reduction process through interaction of photo-excited surface plasmon states with adsorbed reaction intermediates. The significance to investigate reduction of CO_2 at gold, silver and copper surfaces enhanced by the extra activation of the process provided by the illuminated, incorporated plasmonic metallic nanostructures is supported by the occurrence of the photo-emission process associated with the decay of photo-excited surface plasmons. Actually, two metals Ag and Au, the nanostructures of which exhibit strong excitation of surface plasmon resonance through interaction with near UV and visible photons are also catalytically active toward electro-reduction of CO_2. Besides the photo-emission, another property of plasmonic NPs – heat generation resulting from the absorption of incident photons and their influence on the reduction of CO_2 at composite cathodes will be the objectives of this presentation.

2:20 PM

(ICACC-S7-036-2018) Manipulating hematite nanostructure design for efficient water oxidation reaction under sunlight irradiation (Invited)

F. L. de Souza*[1]; W. M. Carvalho-Jr[1]; D. Ferreira Muche[2]; R. Castro[2]

1. Federal University of ABC, Center of Natural Science and Humanity, Brazil
2. University of California, Davis, USA

Nanostructure design faster becomes a key factor in the race for enhancing the photoelectrochemical device performance, because it may lead to a reduction of the charge recombination rate by ensuring an efficient photogenerated charge separation. This work shows that chlorine species originated from commonly used iron precursors annihilate hematite nanorods photocurrent by providing recombination pathways. The annealing at 750 °C per 30 min led to observable chlorine species elimination enabling hematite nanorod to achieve a photocurrent response around 1.3 $mA.cm^{-2}$ at 1.23 V vs RHE, which is comparable to the best results found in the literature. Although hematite nanorod films could be obtained by thermal decomposition of iron oxyhydroxide phase (β-FeOOH) at low temperature (390 °C), indistinguishable photocurrent responses under dark and sunlight irradiation conditions were observed until the nanorods were annealed (activated) at 750 °C. Our results, suggest that residual chlorine species from the synthesis can act as electron trap and recombination sites for photogenerated holes.

Nanomaterials for Photocatalysis, Solar Hydrogen and Thermoelectrics III

Room: Coquina Salon C

Session Chairs: Renata Solarska, University of Warsaw; Chun-Hong Kuo, Acadmeia Sinica

3:20 PM

(ICACC-S7-037-2018) Highly Efficient, Visible-Light-Activated Photocatalysts with Post-illumination "Memory" Effect (Invited)

Q. Li*[1]

1. Institute of Metal Research, Chinese Academy of Sciences, Shenyang National Laboratory for Materials Science, China

Highly efficient, visible-light-activated photocatalysts with post-illumination "memory" effect were recently developed in our work. This interesting post-illumination "memory" effect was firstly found by us on palladium oxide-modified, nitrogen-doped TiO_2 (TiON/PdO). Clear evidences of charge flow to/from PdO nanoparticles on the visible light photocatalytic TiON matrix were obtained under visible light illumination and after the illumination was shut off, respectively. The photoelectron flow to PdO nanoparticles enhanced photocatalytic disinfection efficiency under visible light illumination by the separation of electron and hole pair, while the discharging of PdO nanoparticles following the turn-off of the illumination created the interesting photocatalytic "memory" effect as demonstrated by its bacterial disinfection in dark. Further work demonstrated that the photocatalytic "memory" effect was not limited to noble metal modification. It may be possessed by any photocatalyst system, if in which photo-excited electrons could transfer from the light absorber component to the decoration component while the decoration component could trap and release them. Thus, the material cost could be largely reduced by removing noble metal components, beneficial for their potential applications.

3:50 PM

(ICACC-S7-038-2018) Interface Engineering in Inorganic Hybrid Structures towards Improved Photocatalysis (Invited)

Y. Xiong*[1]

1. University of Science and Technology of China, Department of Chemistry, China

Designing new photocatalytic materials for improving photoconversion efficiency is a promising route to alleviate the steadily worsening environmental issues and energy crisis. Despite the invention of a large number of catalytic materials with well-defined structures, their overall efficiency in photocatalysis is still quite limited as the three key steps - light harvesting, charge generation and separation, and charge transfer to surface for redox reactions - have not been substantially improved. To improve each step in the complex process, there is a major trend to develop materials based on inorganic hybrid structures. In this case, interface engineering holds the promise for boosting the overall efficiency, given the key roles of interface structures in charge and energy transfer. In this talk, I will demonstrate several different approaches to designing inorganic hybrid structures with improved photocatalytic performance via interface engineering. The typical demonstrations include semiconductor-plasmonics systems for broad-spectrum light harvesting, metal-semiconductor interfaces for improved charge separation, semiconductor-MOF (metal-organic framework) configurations for activated surface reactions. It is anticipated that this series of works open a new window to rationally designing inorganic hybrid materials for photo-induced applications.

4:20 PM

(ICACC-S7-039-2018) Flame Inspired Nanostructuring: Flexible Ceramics to Advanced Biomaterials (Invited)

J. Gröttrup[1]; F. Schütt[1]; D. Smazna[1]; S. Shree[1]; I. Paulowicz[1]; F. Ceynowa[1]; L. Siebert[1]; S. Kaps[1]; O. Lupan[1]; R. Adelung[1]; Y. K. Mishra*[1]

1. Kiel University, Institute for Materials Science, Germany

The introduced flame synthesis method offers unique nanostructuring avenues for different metal oxides ranging from quasi 1D wires to porous 3D interconnected networks.[1] This strategy allows direct integration of ZnO nanostructures and their 3D networks on the desired substrates for various applications, e.g., optics, photocatalysis or nanosensing, and piezotronics, etc. The 3D shape of the ZnO tetrapods facilitates them to be used as efficient fillers for advanced composites. The 3D soft ceramic networks are technologically very important and the developed method offers desired synthesis of various 3D networks. The ZnO tetrapods are least cytotoxic and have shown strong potentials towards biomedical applications. The porous networks can be used to design hybrids and can also be used to grow new nanomaterials, 3D Aerographite, CNTT etc.

4:40 PM

(ICACC-S7-040-2018) Citrate Precursor Synthesis, Structural Characterization and Dielectric Properties of $Ba_{1-x}Ca_xZrO_3$ ($0.05 \leq x \geq 0.20$) Nanoparticles

M. Ubaidullah*[1]

1. The Glocal University, Natural and Applied Science, India

Nanoparticles of barium calcium zirconate (BCZ) of general formula $Ba_{1-x}Ca_xZrO_3$ (x = 0.0, 0.05, 0.10, 0.15 and 0.20) have been successfully synthesized via polymeric citrate precursor method for the first time. The nanoparticles were investigated by means of X-ray diffraction (XRD), TEM, SEM, energy dispersive X-ray spectroscopy (EDX), BET surface area studies and dielectric properties. Monophasic nanocrystalline formation of Ca-doped $BaZrO_3$ solid solutions have been confirmed by X-ray diffraction studies. Particles size comes out to be in the range of 42-75 nm by using TEM studies. SEM studies exhibit the surface morphology of ceramics. The incorporation of calcium on $BaZrO_3$ matrix and element detection has been established by energy dispersive X-ray spectroscopy. The surface area of as prepared nanoparticles was found to be in the range of 233 –271 m^2g^{-1} and corroborated with TEM particle size and pore (BJH and DA) size studies. Room temperature dielectric studies were carried out in the frequency range of 20 kHz to 1 MHz to explore the electrical properties of Ca-doped $BaZrO_3$. The dielectric constant was found to increase (48.1 to 284.7) on increasing the Ca^{2+} ion concentration. High dielectric constant, low dielectric loss and high surface area make these nanoceramics as the potential candidates for electro ceramic industries.

5:00 PM

(ICACC-S7-041-2018) Efficiency Enhancement of Low-Cost Upgraded Metallurgical-Grade Si Solar cells

D. Lee*[1]; H. Kim[1]; E. Kim[2]

1. Korea Institute of Industrial Technology, Green Materials & Processes Group, Republic of Korea
2. Korea Institute of Industrial Technology, Ulsan Regional Division, Republic of Korea

Recent reports on solar modules based on ultrathin microcells, have shown that unique properties such as flexibility and transparency can be obtained from inorganic solar cells. Here, we present ultrathin solar modules that were fabricated using low-cost metallurgical-grade silicon wafers. In order to compensate the diminished cell performance resulting from high impurity level of the starting material and from small physical thicknesses of the microcells, metallic (Ag) nanoparticles that serve as light-concentrating centers were incorporated to the device by block-copolymer lithography. These plasmonic crystals (nanoparticles) have brought improvements in light absorption and quantum efficiency of the device, and we further enhanced these properties by forming nanopillars via reactive ion etching (RIE). Detailed studies of optical properties and performance of the device with varying the nanostructures' size, together with investigation of stability of the ultrathin metallurgical-grade silicon microcells over time were carried out.

S8: 12th International Symposium on Advanced Processing and Manufacturing Technologies for Structural and Multifunctional Materials and Systems (APMT12)

Advanced Composite Manufacturing

Room: Coquina Salon A
Session Chairs: Young-Wook Kim, University of Seoul; Hisashi Serizawa, Osaka University

1:30 PM

(ICACC-S8-032-2018) Continuous Carbon Nanotube Yarn Reinforced Ceramic Nanocomposites by PIP Process (Invited)

Y. Li*[1]

1. Tianjin University, China

Continuous carbon nanotube (CNT) yarns, with length over thousands of meters long, are now available by direct spinning of CNTs from CVD synthesis, making it possible to develop light weight, strong and high temperature CNT reinforced ceramic composites. These CNT yarns, consisting of millions of interconnected double walled or single walled CNTs, are also highly electrically conductive, thermally conductive, and have multifunctional properties such as field emission and actuation behaviors, promising to be used for making functional composites. They can be manipulated, twisted, packed, aligned and woven into desired objects to be used as preforms for designing structural and functional nanocomposites with defined orientations, alignments and distributions of CNTs

in ceramic matrices with monolithic or multiscale structures. After introducing the CNT yarns, including their fabrications, properties and processing behaviors, we demonstrate the fabrication of CNTs reinforced ceramic composites using the CNT yarn preforms by the precursor-infiltration-pyrolysis (PIP) process. The fabricated CNT-ceramic composites include SiC/CNTs, SiOC/CNTs, SiCN/CNTs and SiBCN/CNTs obtained from their respective precursors of polycarbosilanes, polysiloxanes, polysilazanes and polyborosilizanes. The processing, structures and high temperature and electrical properties of these nanocomposites are discussed.

2:00 PM

(ICACC-S8-033-2018) Carbon Nano-Phase Directionaly Reinforced Alumina-Zirconia (ZTA) Composites with superior dispersion and increased mechanical properties

C. L. Falticeanu*[1]

1. McGeoch Technology-Precision Ceramics, Ceramic Materials, United Kingdom

This paper presents a compoarative study on commercial Carbon Nano Phase(CNP-multilayer Grahene platelets and multiwall Carbon nano tubes)-reinforced ZTA ceramic composites and the effect of different reiforcing phase amounts on the final mechanical properties. Commercial ZTA powders were mixed for with different loadings of both CNP types ranging from 0.5 to 1.5 vol.%. Increased dispersion and directionality of the reinforcement phase has been achieved using a novel high intensity viscous polymer mixing methodology followed by directional forming (e.g. calendaring/rolling). The critical temperature corresponding to the onset of the carbothermal reduction and subsequent CNP degradation has been ascertained through a combination of of TGA/DTA. A combination of low temperature pre-sintering followed by Hot Isostatic Pressing ensured complete densification with limited CNP degraddation. High resolution imaging investigation showed that the viscous polymer mixing/forming method is very effective in dispersing and aligning the CNP with the rolling direction. Addition of approximately 0.5vol% CNP led to a significant increase in bending strength and fracture thoughness up to 60%. The excellent mechanical properties of the CNP/ZTA composites and the relatively ease of processing may enable them to be applied to a wide range of demanding applications.

2:20 PM

(ICACC-S8-034-2018) High-Temperature Ceramic Matrix Composites using Microwave Enhanced Chemical Vapour Infiltration

M. Porter*[1]; A. D'Angio[1]; J. Binner[1]; P. Mogilevsky[2]; M. Cinibulk[2]

1. University of Birmingham, Metallurgy and Materials, United Kingdom
2. Air Force Research Lab, USA

High-temperature ceramic matrix composites (HT-CMCs), specifically SiC_f/SiC, have been identified as candidates to operate in the hostile aero-thermo-chemical environments experienced in service without compromising structural integrity, whilst keeping mass at a premium. Chemical vapour infiltration (CVI), is an effective manufacturing route capable of creating near fully dense CMCs with a refined microstructure with no preform degradation and minimal residual stresses. CVI's challenges, however, are three fold; i) processing is typically 2 – 3 months; ii) premature surface pore closure occurs preventing great than 90% densification; iii) associated costs are very high and the product expensive. Microwave energy (MCVI) has been proposed as a potential solution to heat the SiC fibre preform for CVI; it produces an inverse temperature profile, which initiates densification at the centre of the sample, thus avoiding porosity closure. It is expected that the use of MCVI could yield near fully dense products in as little as 72 – 96 hours. This paper presents an update on the forming and characterising of the SiC matrix produced using the MCVI technique. Kinetics, composition, densification profile, morphology and growth mechanism of the SiC matrix have all been observed using a suite of characterisation techniques to see the effect of changing the processing variables.

2:40 PM

(ICACC-S8-035-2018) Influences of Laser Condition and Slit Shape on Joinability of Zircaloy-SiC/SiC Composite Tube Joint

H. Serizawa*[1]; H. Motoki[2]; Y. Asakura[3]; Y. Sato[1]; N. Nakazato[4]; M. Tsukamoto[1]; J. Park[3]; H. Kishimoto[4]; A. Kohyama[3]

1. Osaka University, Joining and Welding Research Institute, Japan
2. Osaka University, Graduate School of Engineering, Japan
3. Muroran Institute of Technology, OASIS, Japan
4. Muroran Institute of Technology, Japan

As for the fuel cladding in the light-water reactor, silicon carbide fiber reinforced silicon carbide composite (SiC/SiC composite) is one of the promising candidates as a replacement of Zircaloy due to many superiorities, where it is necessary to develop the end-cap seal of SiC/SiC composite cladding. According to our previous researches, as one possible design, SiC/SiC composite and Zircaloy tubes were joined by employing the laser irradiation based on the caulking method. Where the inner surface of Zircaloy tube and the outer surface of SiC/SiC composite tube were simple machined so that Zircaloy tube partially overlaps with SiC/SiC composite tube. In addition, the outer surface of Zircaloy tube was circumferentially silted and titanium powder was inserted in the slit. Then, the laser irradiation was circumferentially applied on the outer surface of Zircaloy tube. In this research, in order to examine the joinability of Zircaloy and SiC/SiC composite, the laser irradiation time and the shape of lit were varied. As the result of laser irradiation, two tubes were mechanically joined. Moreover, the good adhesion between Zircaloy and SiC/SiC composite was partially produced as the result of the generation of all proportional solid solution between titanium and zirconium. The microstructural observations will be discussed in order to examine an airtightness of this joint.

3:20 PM

(ICACC-S8-036-2018) Effect of Additive Composition on Mechanical and Thermal Properties of Pressureless Sintered Silicon Carbide Ceramics

Y. Kim*[1]; Y. Seo[1]; J. Eom[2]

1. University of Seoul, Dept. of Materials Science & Engineering, Republic of Korea
2. Dandan Materials, Republic of Korea

Mechanical and thermal properties of three different pressureless sintered liquid-phase sintered SiC ceramics (LPS-SiC) were characterized: SiC ceramic sintered with 6.5 vol% Y_2O_3-Sc_2O_3–AlN (YScN); SiC ceramic sintered with 9.0 wt% Al_2O_3-Y_2O_3-CaO (AYC); and SiC ceramic sintered with 3 vol% Al_2O_3-Y_2O_3-AlN (AYN). All SiC ceramics could be sintered to >96% theoretical density at temperatures between 1850°C and 1950°C. Toughened microstructures have been obtained when sintered at 1850°C for the AYC specimen and at 1900°C for the AYN specimen. In contrast, relatively equiaxed microstructure consisting of SiC grains with smaller aspect ratio than the others was obtained in the YScN specimen even after sintering at 1950°C for 6 h. Typical flexural strength and toughness values of the YScN, AYC, and AYN at RT were 520 MPa and 5.1 $MPam^{1/2}$, 528 MPa and 5.3 $MPam^{1/2}$, and 425 MPa and 6.9 $MPam^{1/2}$, respectively. Typical thermal conductivity values of the YScN and AYC at RT were 110 $Wm^{-1}K^{-1}$ and 80 $Wm^{-1}K^{-1}$, respectively. Generally, the thermal conductivity of the SiC ceramics increased with increasing sintering temperature because of the decrease in the lattice oxygen content of the SiC grains. The present results suggest that mechanical and thermal properties of LPS-SiC could be adjusted by judiciously selecting sintering additive composition and processing conditions.

3:40 PM

(ICACC-S8-037-2018) Effect of Alloy Composition on Reactive Melt Infiltration for the Production of SiC/SiC Composites

R. B. Reitz*[1]; F. W. Zok[1]; C. G. Levi[1]

1. University of California, Santa Barbara, Materials, USA

Reactive melt infiltration can be used to produce dense matrices for SiC/SiC composites, but always leaves behind residual phases which can limit the operating temperature or mechanical performance of the composite. In a robust composite, these phases would be expected to have high melting points, desirable oxidation properties, and thermal expansion coefficients that are compatible with SiC. The current work seeks to explore the role that alloy composition plays in the reactive melt infiltration process itself as well as in determining the properties of the final composite. DTA data and microstructures resulting from infiltrations with different alloying systems will be discussed. Criteria for selecting a desirable alloy and producing desired residual phases will be emphasized.

4:00 PM

(ICACC-S8-038-2018) Elecroconductive oxide ceramics with graphene-encapsulated fillers

I. Hussainova*[1]; I. Jasiuk[2]; M. Drozdova[1]; S. Kale[2]

1. Tallinn University of Technology, Estonia
2. University of Illinois at Urbana-Champaign, USA

Miniaturization of ceramic components with complex shapes is a great challenge in many applications. The main limiting factors are mechanical unreliability and poor electrical conductivity, which make the materials difficult to be processed. Electro-discharge machining allows producing complex-shaped parts, but requires the use of materials with sufficient electrical conductivity. In this study, an ex situ strategy for fabrication of electrically conductive and cost-effective metal oxide ceramics by incorporation of hybrid graphene-wrapped ceramic nanofibers is presented. Fully dense alumina and zirconia composites with homogeneously distributed hybrid nanofillers of length ranging from 0.1 to 10 micrometres and a single fibre diameter of 10 nanometres is produced by spark plasma sintering. Three-dimensional Monte Carlo simulation is performed using a tunneling-percolation model based on critical path approximation to compliment the experiments. The fillers are modeled as randomly oriented and polydisperse hard-core soft-shell prolate ellipsoids. A percolation threshold as low as 0.2 wt.% is achieved and the electrical conductivity surpasses 10^3 Sm^{-1} when hybrid filler content is only 2 wt.% in the composites. An increase in conductivity does not result in deterioration of the mechanical properties. Hardness and fracture toughness increase up to 10% and 40%, respectively, as benchmarked against plain ceramics.

4:20 PM

(ICACC-S8-039-2018) TEM Analysis of Interfaces in Diffusion-Bonded SiC Fiber-Bonded Ceramics Using Ti/Cu Interlayers

T. Ozaki*[1]; Y. Hasegawa[1]; H. Tsuda[2]; S. Mori[2]; M. C. Halbig[3]; R. Asthana[4]; M. Singh[5]

1. Osaka Research Institute of Industrial Science and Technology, Japan
2. Osaka Prefecture University, Graduate School of Engineering, Japan
3. NASA Glenn Research Center, USA
4. University of Wisconsin-Stout, USA
5. Ohio Aerospace Institute, USA

Silicon Carbide-based fiber bonded ceramics are promising materials for a wide variety of aerospace and ground based thermo-structural and thermal management applications. Robust joining and integration technologies are indispensable for this material system in order to fabricate large size and complex shape components with desired functionalities. Active metal brazing and diffusion bonding techniques using metallic interlayers have been developed for integration of fiber bonded ceramics for intermediate temperature applications. Recently, good strength was exhibited in SiC fiber-bonded ceramics (SA-Tyrannohex™: SA-THX) joints fabricated using diffusion bonding of Ti/Cu metal interlayers. However, interfacial microstructure was not investigated and the reason for high strength has not been elucidated. For transmission electron microscopy (TEM), we prepared thin samples from the joint of SA-THX diffusion bonded with Ti/Cu metal interlayers by Focused Ion Beam (FIB) system and carefully investigated the interfacial microstructure and composition. In this presentation, microstructure of diffusion bonded SA-THX using Ti/Cu interlayers mainly obtained by TEM observation will be presented. The formation process of the interfacial microstructure will be discussed.

S9: Porous Ceramics: Novel Developments and Applications

Innovations in Processing Methods and Synthesis of Porous Ceramics III

Room: Coquina Salon G

Session Chair: Gideon Grader, Technion - Israel Institute of Technology

1:30 PM

(ICACC-S9-009-2018) Additive manufacturing of periodic ceramic substrates for catalyst supports

A. Ortona*[1]

1. SUPSI, MEMTi, Switzerland

Additive manufacturing (AM) has the potential to revolutionize engineering because of its advantages in the product development phase. The revolution consists in the new approach of components' design by function and no longer by manufacturability. This is the motivation driving authors to design and realize by additive manufacturing a series of new catalyst supports which are no longer constrained by the standard industrial manufacturing methods and thus allows an optimized gas flow. This work presents a new class of periodic cellular ceramic substrates designed for their function and produced with different AM techniques. They were finally characterized via microscopic analysis (SEM, CT) and compression tests revealing the best printing configuration.

1:50 PM

(ICACC-S9-010-2018) Low Density Reticulated Polymer Derived SiC, SiCN and SiOC foams

P. Jana[1]; E. Zera[1]; B. Santhosh[1]; G. D. Sorarù*[1]

1. University of Trento, Industrial Engineering, Italy

Polymer derived ceramic foams can be processed through the replica method starting from a polyurethane foam impregnated with a suitable pre-ceramic polymer. Unlike the conventional replica method, which uses a ceramic slurry, in the present case, due to the swelling of the polyurethane foam by the preceramic polymer, the resulting ceramic struts are dense. The method is highly flexible and SiC, SiOC and SiCN foams with density in the range 35 – 350 kg/m3 and cell dimension in the range 300 – 1000 μm can be easily prepared. Also, partially closed cell foams can be obtained. Physical properties such as the thermal conductivity and the compressive strength have been measured and they follow the Gibson–Ashby model for open cell foams. Potential applications as high temperature insulating materials or scaffolds for bone regeneration will also be presented.

2:10 PM

(ICACC-S9-011-2018) Ceria coating of carbide macroporous supports by electrophoretic deposition

A. Ortona*[2]; Y. Dang-Hyok[1]

1. Yeungnam University, Republic of Korea
2. SUPSI, MEMTI, Switzerland

This work shows the first results of a reserach line where we aim at combining different well-known ceramics in order realize layered structures and exploit their functional properties. In this first trial we coated commercial Si-SiC foams with Ceria. This material couples the outstanding thermo-mechanical behavior of SiC foams with the redox properies of Ceria. Alternate current EPD in water medium was used to deposit the CeO2 powders. The best EPD parameters were selected emplying a Design of Experiment (DoE) approach. Coated samples were thermally treated and characterized.

2:30 PM

(ICACC-S9-012-2018) Chemistry of Functional Boron-Modified Silicon Carbide Precursors behind the design of 3D Porous Si-B-C Ceramics

M. Schmidt[2]; C. Durif[2]; P. Colombo[3]; S. Bernard*[1]

1. CNRS, Ceramic Research Institute, France
2. European Membrane Institute, France
3. Università di Padova, Dipartimento di Ingegneria Industriale, Italy

Inherent difficulties to the traditional techniques for the formulation of high performance ceramics can be overcome by the development of synthetic paths where chemistry, processing and material science are combined rationally. The Polymer-Derived Ceramics (PDCs) concept, which is based on the shaping then pyrolysis of inorganic (= preceramic) polymers into advanced ceramics, mostly of non oxide-type, is a chemical route which can offer original and new preparation opportunities in ceramic science. Here, a series of boron-modified polyorganosilanes was synthesized by reaction between allylhydridopolycarbosilane and controlled amounts of borane dimethyl sulfide, then characterized in details. Then, the relationship between the chemical behavior and the processability of the polymers was examined. Polymers with low boron contents display appropriate requirements for facile processing in solution such as impregnation of host carbon foams resulting in the design of monoliths with hierarchical porosity, significant pore volume and high specific surface area after pyrolysis. Polymers with high boron contents are more appropriate for solid-state processing. Macroporous ceramics can thus be obtained by direct warm-pressing of polymer-PMMA mixture and pyrolysis. The chemical and mechanical stability of the microcellular ceramics has been investigated by ageing tests.

2:50 PM

(ICACC-S9-013-2018) Tailoring Pore Structure and Properties of Yttria-Stabilized Zirconia Aerogels for High Temperature Applications

F. Hurwitz*[1]; H. Guo[2]; N. Olson[1]; R. B. Rogers[1]

1. NASA Glenn Research Center, USA
2. Ohio Aerospace Institute, USA

There is a need for lightweight, mesoporous materials that maintain their pore structure at temperatures of 600 to 1200°C for use in applications requiring thermal insulation. Synthesis methods for fabricating yttria stabilized zirconia (YSZ) aerogels have been explored, with the objective of controlling pore structures which can persist to elevated temperatures. Tailoring of pore structure, as well as shrinkage and phase transformation were studied for compositions ranging from 0-20 mole percent yttria. Thermal stability of the microstructure was characterized by scanning electron microscopy and X-ray diffraction and surface area analysis techniques, and the results compared with microstructural stability of aerogels in the aluminosilicate and yttria-alumina systems, with the goal of developing aerogel systems optimized for high temperature applications.

Structure and Modeling of Porous Ceramics

Room: Coquina Salon G
Session Chair: Sawao Honda, Nagoya Institute of Technology

3:30 PM

(ICACC-S9-014-2018) Three-dimensional microstructural modeling and homogenization analysis of highly porous ceramics prepared by gelation freezing route

M. Fukushima*[1]; H. Hyuga[1]; C. Matsunaga[1]; T. Ohji[1]; Y. Yoshizawa[1]

1. National Institute of Advanced Industrial Science and Technology (AIST), Japan

Process and properties of cellular ceramics via gelation freezing route have been investigated, in terms of processing factors, microstructure, three-dimensional microstructural modeling and homogenization analysis, in which compressive stress and thermal conductivity of porous ceramics with various porosities and pore configurations were analyzed by homogenization technique. The homogenization method was conducted with microscopic models created from three dimensional images, global stress distributions and temperature variations in macroscopic models were analyzed by finite element method. The simulation results were relatively consistent with the experimental results. The relationship between processing factors, experimental and simulation results, and microstructures will be also discussed.

3:50 PM

(ICACC-S9-015-2018) Towards development of a robust 3D numerical method to interpret thermograms of cellular ceramics acquired by the Laser Flash method

S. Lal[1]; M. Badri[1]; Y. Favennec[1]; B. Rousseau*[1]

1. LTeN UMR CNRS 6607, France

So far, understanding influence of radiative transfer on the effective thermal conductivity of cellular ceramics, especially at high temperatures (~1600°C), remains unclear. Cellular ceramics being highly porous and carrying a complex 3D arrangement based on their interconnected network of struts, can be potentially modelled as semitransparent media. For such cases thermal radiation can be emitted, absorbed and scattered. Thereby, for precise modelling of thermograms obtained by the Laser Flash method, it is required to solve the heat diffusion equation with considerable influence of radiative transfer (solved with the Radiative Transfer Equation, RTE). Previously, most of the works conducted in this area have assumed the problem to be one-dimensional in nature while considering degraded radiative properties. Such interpretations of thermograms may be imprecise and could amplify the doubt on exact contribution of thermal radiation. For the present work, a fast and robust 3D numerical method based on the finite element method, is used to compute thermograms without restrictive assumptions. The exact radiative properties are provided by genMat software for a set of silicon carbide-based cellular materials with prescribed textural features. The influences of both the texture and used coatings on the themograms curves will be finally discussed.

4:10 PM

(ICACC-S9-016-2018) Microstructural Variations and Compaction Characteristics of Spray-Dried Alumina

I. Maher*[1]; R. A. Haber[1]

1. Rutgers University, Materials Science and Engineering, USA

Microstructural variability in ceramic green bodies has been a known issue within the ceramic processing world. In this study, alumina was the system analyzed and will be processed by spray drying alumina slurries. Two slurry characteristics, specific gravity

and viscosity, were altered and studied to understand the affect slurry characteristics have on the formation of the granules prior to atomization within the spray dryer. Image analysis was conducted to understand the extent of core size and formation within the alumina granules by use of a field emission scanning electron microscope. The plasticity of the spray dried granules on a compaction scale is used as a tool to determine the effects of slurry characteristics on the formation of the granules. A relationship between their compaction characteristics and density variations to slurry characteristics prior to spray drying will be investigated.

4:30 PM

(ICACC-S9-017-2018) Effect of sintering temperature on porosification behavior of fired LTCC substrates

A. Hajian*[1]; M. Stöger-Pollach[2]; M. Schneider[1]; H. Homolka[1]; D. Müftüoglu[1]; U. Schmid[1]

1. Vienna University of Technology, Institute of Sensor and Actuator Systems, Austria
2. Vienna University of Technology, University Service Centre for Transmission Electron Microscopy, Austria

Besides its outstanding material properties, low temperature co-fired ceramic (LTCC) is the technology of choice when targeting the realization of highly integrated substrates or packaging solutions for micromachined devices, especially for high-frequency applications [1]. However, LTCC materials are generally suffering from high permittivity and we have recently shown that the wet-chemical porosification under acidic condition allows a permittivity reduction in the as-fired state [2,3]. In the present study, we have employed an alternative etching condition for porosification of the LTCC with a better surface quality which features a suitable bearing plane for the subsequent realization of metallization lines. In this approach an aqueous solution of KOH at temperatures lower than 90 °C was applied as etching solution for porosification of the Ceramtec GC LTCC samples fired at different sintering peak temperatures of 800, 850, 900, and 950 °C. The surface of the as-fired samples was porosified in the aforementioned solution and thereby the effect of sintering peak temperature on the porosification behavior was studied. Different characterization techniques, including SEM, TEM, XRD, EELS, and EDX were used for investigation of the morphology and chemical composition of the fired samples, before and after porosification which will be presented in the final conference paper.

4:50 PM

(ICACC-S9-018-2018) Characterization of Catalyst Support Porosity Using a Visualization and Segmentation Approach

M. K. Alazzawi*[1]; R. A. Haber[1]

1. Rutgers University, Materials Science and Engineering, USA

Species diffusivity in porous materials such as catalyst supports and membranes are governed by pore distribution and pore interconnectivity. Extrusion process used to fabricate porous materials. This process poses an issue in the spatial variation of porosity and inhomogeneity in the final product. The inhomogeneity and spatial variation of porosity at the die wall compared to other regions of extrudate can be related to the frictional die wall shear and die entry deformation. The frictional die wall shear and die entry deformation are a function of extrusion velocity and die geometry. In this work, a titania- binder system was used. A range of extrusion velocities and die geometries were analyzed using a capillary rheometer. In assisting of determining the quality of extrudate, a rotation rheometer was used to quantify the green strength. A quantitative approach using visualization and segmentation was developed to study the spatial variation of porosity at the die wall (sheared region) compared to unsheared regions (center of cross section) of extrudate. The effect of extrusion velocity and die geometry on the porosity distribution and green strength will be discussed.

S10: Virtual Materials (Computational) Design and Ceramic Genome

Modeling of Structure and Property I

Room: Coquina Salon F
Session Chair: Wai-Yim Ching, University of Missouri-Kansas City

1:30 PM

(ICACC-S10-001-2018) Computing Grain Boundary 'Phase' Diagrams: Recent Progresses and Future Directions (Invited)

J. Luo*[1]

1. University of California, San Diego, USA

This talk will review our recent progresses to compute grain boundary (GB) 'phase' (complexion) diagrams via several different methods. Earlier studies are reviewed [J. Am. Ceram. Soc. 95: 2358 (2012); Curr. Opin. Solid State Mater. Sci. 20:268 (2016)]. Two more recent studies computed GB diagrams to forecast (1) the formation and stability of sub-eutectic, quasi-liquid, intergranular films (IGFs) in TiO_2-CuO [Acta Mater. 130: 329 (2017)] and (2) bilayer complexions in Ni-Bi [Scripta Mater. 130:165 (2017)]; both computed GB diagrams have been validated by experiments. Using the symmetrical Σ 5 [210] tilt GB in Mo-Ni as a start point, we have further developed a method to combine a modified genetic algorithm with hybrid molecular dynamics and Monte Carlo simulations in semi-grand canonical ensembles to construct more realistic GB diagrams with atomistic details. Specifically, we have revealed a first-order GB phase-like transformation line, ending at a GB critical point. The GB diagrams constructed from atomistic simulations can effectively represent both low-T adsorption transitions predicted by an Ising type lattice model and the effects of high-T interfacial disordering forecasted by a phenomenological premelting/prewetting model. Ongoing work is being conducted to use a similar atomistic simulation method to model the behaviors of more general GBs as well as more complex ceramic materials.

2:00 PM

(ICACC-S10-002-2018) Classical potentials from ab initio molecular dynamics via force matching: Application to amorphous and disordered materials modeling (Invited)

P. Rulis*[1]; P. Khanal[1]; N. Dari[1]

1. University of Missouri - Kansas City, Physics and Astronomy, USA

Multi-element amorphous and disordered solids represent a unique modeling challenge because of the demands they place on common classical- and ab initio molecular dynamics methods. Classical potentials for multi-element materials are difficult to produce because of the large number of interaction coefficients and the range of environments in which each element may find itself. Using purely ab initio methods is prohibitive because of the large number of atoms that are needed to model non-periodic solids and because of the long simulation time that is required to apply cook-and-quench or condensation methods for model generation. Thus, multi-element amorphous and disordered solids sit at an unfortunate crossroad between the two most effective and popular atomic-scale modeling methods. This presentation will discuss recent efforts to use VASP and Potfit based ab initio force-matching methods to develop effective classical potentials for use in the common molecular dynamics package, LAMMPS. Presently, two target materials are under consideration: amorphous hydrogenated boron carbide (a-BC:H) and amorphous tetrahedral carbon (a-C). The accomplishments and challenges associated with those two materials will be discussed in the context of the unique attributes of each.

2:30 PM

(ICACC-S10-003-2018) Theoretical Investigation of mechanical and Thermal Properties of ABO_3 (A=Sr, Ba; B= Ti, Zr, Hf) perovskites (Invited)

B. Liu*[1]

1. Shanghai University, School of Materials Science and Engineering, China

Developing new generation of TBCs with improved high temperature performance is an urgent topic for gas turbine engine development. With various options of 'A' and 'B' cations, perovskite oxide is suggested a promising TBC material candidate. Mechanical and thermal properties of the ABO_3 (A=Sr, Ba; B=Ti, Zr, Hf) perovskites are investigated using Density Functional Theory (DFT). Calculated mechanical properties (bulk modulus, shear modulus, Young's modulus, Vickers hardness and ductility) of polycrystalline perovskites predict the stability of ABO_3 concerned. Orientation-dependent mechanical and thermal properties are also identified. In this paper, the mechanism of the anisotropic performance and the inspirations from the directional differences of perovskites are discussed, which provide new perspectives for TBC materials searching.

Modeling of Structure and Property II

Room: Coquina Salon F

Session Chair: Paul Rulis, University of Missouri - Kansas City

3:20 PM

(ICACC-S10-004-2018) First-principles calculation of the electronic structure and bonding in an inter-granular glassy film model of silicon nitride (Invited)

W. Ching*[1]; P. Adhikari[1]; P. Rulis[1]

1. University of Missouri-Kansas City, USA, USA

The electronic structure and bonding of a large inter-granular glassy film (IGF) model [1] of 3864 atoms containing two crystal grains of different orientations are calculated after the model is fully relaxed using VASP. The relaxed structure is used to calculate the electronic structure, density of states, interatomic bonding, partial charge distribution, and electron localization using the OLCAO method [2]. Analysis of the data focuses on the interfacial regions between bulk β-Si_3N_4 and the Si-O-N glass layer. The total bond order density (TBOD) and its partial components (PBOD) are evaluated in different interfacial and bulk regions. They are interpreted in the context of the presence of "defective" atoms with dangling bonds. The overall effective charges in bulk crystal grains and in the glassy regions are positive and balanced by the negative charges in the interfacial regions. The implications obtained from this quantum mechanical analysis of a realistic IGF model on the strength, fracture toughness, and processing methods are discussed.

3:50 PM

(ICACC-S10-005-2018) Atomistic modeling of thermal transport in cations doped CeO_2 (Invited)

X. Bai*[1]

1. Virginia Tech, Materials Science and Engineering, USA

CeO_2 is an important ceramic material for many technological applications. It also can be used as a model material for many fluorite-based oxides. The thermal conductivity in these oxides is an important physical property. Impurities in these oxides can affect the phonon-based thermal transport behavior significantly. Here five different trivalent cations are doped in CeO_2 to study the roles of ionic radius and ionic mass of doped cations on the thermal transport. Using non-equilibrium molecular dynamics simulations, the thermal conductivities of these doped systems are calculated for a wide range of concentrations of doped ions. It is found that the bulk thermal conductivity decreases with increasing ionic radius while such a correlation does not exist for ionic mass. When these doped cations segregate to grain boundaries, they increase the grain boundary thermal resistance. Finally, an analytical model is developed to study the effect of segregation of doped cations to grain boundaries on the effective thermal conductivity in nanocrystals and polycrystals. The results show that segregation of doped cations to grain boundaries improve the effective thermal conductivity of nanocrystals significantly.

4:20 PM

(ICACC-S10-006-2018) Virtual Characterization of Ceramic Defects and Interfaces

S. P. Coleman*[1]

1. US Army Research Laboratory, USA

Simulated characterization techniques enable a synergy between large-scale atomistic models and experiments to help further understand the role of defects and interfaces in ceramics. Large-scale atomistic models are generated with specified distributions of defects in order to systemically explore the resulting changes to various simulated characterization methods based on diffraction. Virtual x-ray, electron and Kikuchi diffraction patterns created from million-atom models and analyzed using quantitatively using similarity metrics reveal that subtle changes to the structure reveal distinct changes. Trends in these changes are used to help analyze experimental characterization in order to bring further insights into their underlying nanoscale structure.

4:40 PM

(ICACC-S10-007-2018) Theoretical Prediction of Composition-Dependent Structure and Properties of Alumina-Rich Spinel

B. Tu*[1]; H. Wang[1]

1. Wuhan University of Technology, State Key Lab of Advanced Technology for Materials Synthesis and Processing, China

Spinel transparent ceramics have outstanding thermal-mechanical properties for technological applications. The isotropic crystal, large grain size, and highly dense structure make it possible to predict the optical and mechanical properties of transparent ceramics by first-principles simulations. Five redefined supercell models of alumina-rich spinel ($Mg_{1-x}Al_{2(1+x/3)}O_4$, x = 0, 0.25, 0.5, 0.75, and 1) were constructed to investigate the composition-dependence of structural, electronic, optical and mechanical properties. The first-principles calculations were developed with high accuracy to evaluate the lattice constant, electronic structure, dielectric function and elastic constants. The detailed structural information was presented by analyzing the variations in lattice constant, interstitial volume and anion parameter with x. The composition-dependent bulk modulus, shear modulus and electronic structure of alumina-rich spinel were well discussed. As increased the content of Al, the bulk modulus and shear modulus showed increasing trends, while the optical absorption in the UV region presented a blue-shift of ~1.2 eV. These theoretical investigations of crystal structure and intrinsic properties supply useful information for the design of alumina-rich spinel transparent ceramics.

S12: Advanced MAX/mxene Phases and UHTC Materials for Extreme and High Temperature Environment

Synthesis, Processing, and Densification

Room: Tomoka B

Session Chair: Bai Cui, University of Nebraska, Lincoln

1:30 PM

(ICACC-S12-036-2018) Phase Control during Synthesis of Nanocrystalline Ultrahigh Temperature $Ta_xHf_{1-x}B_2$ Solution Powders

P. Foroughi*[1]; Z. Cheng[1]

1. Florida International University, Mechanical & Materials Engineering, USA

TaB_2 and HfB_2 can form continuous solid solutions. However, synthesis of single-phase $Ta_xHf_{1-x}B_2$ solid solution nanopowders via commonly used carbothermal reduction (CTR) is complicated due to difference in reactivity of parent oxides with carbon leading to phase separation, i.e. formation of two individual borides instead of a single-phase solid solution. Nanocrystalline $Ta_xHf_{1-x}B_2$ nanocomposite and solid solution powders were synthesized by CTR of intimately mixed tantalum-hafnium-boron oxide(s) and carbon obtained from aqueous solution processing of $TaCl_5$, $HfCl_4$, B_2O_3 and sucrose. Particular emphasis was given to investigate the influences of starting materials, processing conditions, and catalysts used on phase separation during the formation of boride phase(s). It was found that due to the immiscibility of Ta-Hf oxides, individual HfB_2 and TaB_2 phases form and then go through slow inter-diffusion forming boride solid solution phase. However, presence of a liquid catalyst such as copper or NaCl accelerates the solid solution formation during the CTR process by increasing the inter-diffusion rate. It was also observed that direct reduction of $TaCl_5$ and $HfCl_4$ using a strong reducing agent such as $NaBH_4$ yields a single phase $TaHfB_2$ solid solution at noticeably lower temperatures (~700°C) thanks to avoidance of the oxides formation and the associated CTR route.

1:50 PM

(ICACC-S12-037-2018) Microstructures and Properties of Spark Plasma Sintered MoAlB Ceramics

B. Cui*[1]; T. Lou[1]; X. Yan[1]

1. University of Nebraska, Lincoln, Mechanical & Materials Engineering, USA

Molybdenum aluminum boride (MoAlB) is a new ternary transition metal boride which has promising high-temperature applications. It inherent excellent properties of binary transitional metal borides (e.g., MoB, ZrB_2) such as high melting temperature, high hardness, and thermal conductivity. However, MoAlB is superior to MoB because the Al element can provide oxidation resistance in high-temperature environments, and its nanolaminated structure results in a unique damage tolerance. In this research, polycrystalline MoAlB have been successfully synthesized and simultaneously sintered using spark plasma sintering from MoB and Al powders. The sintering conditions have been optimized to obtain bulk MoAlB samples with a high purity (>98 vol.%) and a high relative density (>97%). The microstructures have been characterized by X-ray diffraction and scanning electron microscopy. The Vickers hardness value of MoAlB is 10.5 GPa, and there are no dominate crack along the edge of Vickers indentations. At or below 1400 Celsius degree, an Al_2O_3-rich oxide scale can form on the surface of MoAlB.

2:10 PM

(ICACC-S12-038-2018) High entropy transition metal carbides: Uncovering a vast new compositional space to explore in the development of new UHTCs

E. Castle*[1]; T. Csanadi[2]; S. Grasso[1]; J. Dusza[2]; M. Reece[1]

1. Queen Mary University of London, School of Engineering and Materials Science, United Kingdom
2. Slovak Academy of Sciences, Institute of Materials Research, Slovakia

Since their discovery in 2004, High Entropy Alloys (HEAs) have become a major research area in the field of metallurgy. These materials are typically single-phase mixtures of several (>4) different alloying elements in equi- or near-equiatomic proportions. The result is a material which has structural order, yet chemical disorder; an arrangement which has been reported to lead to enhanced mechanical, physical and chemical properties. This discovery that single phase solid solutions can be stabilised by their high configurational entropy has therefore opened up a wide new range of useful compositional space for metallurgists to explore. The 'entropy-stabilised materials' concept has recently been successfully applied to metal oxide and transition metal diboride systems, sparking significant interest in the ceramics community. Here, we report on the fabrication of high entropy refractory metal carbides. It is shown that it is possible to produce bulk homogeneous high entropy carbides. Our findings include processing optimisation, advanced diffraction studies, multi-scale microstructural investigations and mechanical and physical properties characterisation. The significance of the work will be discussed in relation to the opportunities created for the development of new Ultra High Temperature Ceramics (UHTCs).

2:30 PM

(ICACC-S12-039-2018) Densification and Oxidation Behaviors of Hafnium Diboride-Hafnium Carbide Composite System

C. M. Young*[1]; C. Zhang[1]; A. Loganathan[1]; B. Boesl[1]; A. Agarwal[1]

1. Florida International University, Mechanical and Materials Engineering, USA

The current study reports on Hafnium Diboride (HfB_2) and Hafnium Carbide (HfC) based ultra-high temperature ceramics and their oxidation behaviors. Pure Hafnium Diboride, Hafnium Carbide, and HfB_2-50 vol% HfC were consolidated using spark plasma sintering (SPS) without the use of sintering aids. HfB_2 showed a poor density of 87% of theoretical value in comparison to HfB_2-HfC composite, which had a density of 98% of theoretical value. The increased density of the HfB_2-HfC composite is attributed to the bimodal powder distribution, which allowed for the smaller HfC particle to occupy the voids between the larger HfB_2 particles resulting in improved packing efficiency and sintering. From X-ray diffraction, it was seen that no solid solution formed. Oxidation studies of each material were carried out by plasma jet in a temperature exceeding 2000^0C.

Properties, Oxidation, and Tribology I

Room: Tomoka B

Session Chairs: Jon Binner, Loughborough University; Anneliese Brenner, Purdue University

3:30 PM

(ICACC-S12-041-2018) A single, meltable precursor for zirconium carbide ceramics and composites

H. Hu*[1]; K. Xie[1]

1. National University of Defense Technology, College of Aerospace Science and Technology, China

A single, meltable ZrC precursor was prepared by condensation of zirconium acetylacetonate ($Zr(acac)_4$) at 190°C for 40-150 min. The preparation of ZrC precursor and the conversions from

precursor to ceramics were investigated by using FTIR, NMR, GPC, DSC-TGA, XRD, SEM and EDS. The precursor after process optimization has a low viscosity (~10mPa-s) and proper processing window (120min) for PIP process, a ceramic yield of 29.6% at 1650°C and a composition of $(ZrC)_{0.337}(HfC)_{0.0025}(ZrO_2)_{0.044}C_{0.1865}$. The synthetic method provides not only a simple and cheap route for precursors, but also a combined composite preparation possibility at the same time with high efficiency.

3:50 PM

(ICACC-S12-042-2018) Effect of surface roughness on ablation performance of Sm-Doped ZrB2/SiC Systems

A. Brenner*[1]; R. Trice[1]; C. Petorak[2]; B. Thompson[2]

1. Purdue University, Materials Engineering, USA
2. Praxair Surface Technologies, USA

Zirconium diboride/silicon carbide (ZBS) ceramics doped with samarium (Sm) have been shown to have high emissivity and good ablation performance for hypersonic applications. Having a dopant concentration of 5 mol.% has shown to increase the emissivity and improve the ablation performance for shrouded plasma sprayed coatings. Using this Sm concentration, the effect of gun power, gas flow rate, and spray distance have been varied during plasma spray to investigate how microstructural differences (e.g. porosity, and surface roughness) affect emissivity and ablation performance. SEM/EDS, AFM, and ablation studies are being performed to quantify the microstructures. Furthermore, TEM investigations have shown that with increasing Sm dopant concentration (3, 5, and 8 mol%) the Sm becomes less evenly distributed around the outside of ZrB2 particles. Knowing the location of the Sm dopant may assist in understanding how the dopant affects the ablation resistance and emissivity of the coatings.

4:10 PM

(ICACC-S12-043-2018) Oxidation Resistance of High Entropy Carbide and Boride UHTCs

L. Backman*[1]; E. J. Opila[1]; T. Harrington[2]; J. Gild[3]; K. Vecchio[2]; J. Luo[3]

1. University of Virginia, Materials Science and Engineering, USA
2. University of California, San Diego, Department of NanoEngineering, USA
3. University of California, San Diego, Program of Materials Science and Engineering, USA

Bulk samples of high entropy ultra-high temperature ceramics (UHTCs) of the composition (HfNbTaTiZr)C and (HfNbTaTiZr)B_2 were fabricated via high energy ball milling and spark plasma sintering. Oxidation behavior of this new class of UHTCs was tested at 1500°C and 1700°C using a resistive heating apparatus in 1 atmosphere reduced PO_2 oxygen/argon gas mixtures for times up to 5 minutes. Oxidation kinetics were determined from the variation of material consumption vs. time. Oxide composition and morphology were characterized using XRD, SEM, TEM and EDS. A nearly continuous layer of complex oxides was observed on the surface, and a subsurface layer showed evidence of selective grain boundary oxidation. Rapid oxidation rates were observed for both carbide and boride at 1500°C and 1700°C, even in 1% O_2/balance Ar. This work serves to further elucidate the oxidation behavior of a new class of ceramics that are proposed for ultra-high temperature applications where oxidation properties are of key importance.

4:30 PM

(ICACC-S12-044-2018) Effect of rare-earth co-dopant (Sm and Er) concentration on total hemispherical emissivity and ablation resistance of ZrB_2/SiC coatings

A. A. Pena*[1]; R. Trice[1]; J. Vernon[2]; C. Petorak[3]; B. Thompson[3]

1. Purdue University, Materials Science and Engineering, USA
2. AFRL, USA
3. Praxair, USA

Hypersonic aircraft need a high emissivity coating (HEC) to mitigate the aerodynamic heating effect produced by the sharp leading edges. Recent work has shown that small amounts of Sm dopant added to zirconium diboride/silicon carbide coatings can increase their emittance in the visible to near infrared wavelength range important for the temperature ranges experience during hypersonic flight. In the work reported here, the effect of an additional dopant (Er) on the emittance as a function of wavelength is being investigated. In this study, coatings with five different combinations of Sm:Er ratios were prepared and evaluated. Emittance as a function of temperature and ablation performance were measured and will be reported.

4:50 PM

(ICACC-S12-045-2018) Electrical conductivities of ceria-based electrodes for use in MHD generators

M. Johnson[2]; D. Cann*[1]; B. Wright[2]; K. Kwong[2]; C. R. Woodside[2]

1. Oregon State Univ, Materials Science, School of Mechanical, Industrial, and Manufacturing Engineering, USA
2. U.S. Department of Energy, National Energy Technology Laboratory, USA

Direct power extraction using oxy-fuel combustion and magnetohydrodynamics (MHD) has the potential to increase a thermal power plant's efficiency by adding a high temperature energy conversion process to existing power cycles. Within an MHD generator, accelerated oxy-combustion products are expected to have temperatures of about 2400 to 3000 K. Electrodes at the generator walls are used to extract the power. The development of high temperature electrodes is desirable in order to reduce wall heat losses, among other considerations. In this work, ceria-based ceramic electrodes were developed for use as MHD electrodes. Ceramic disks of undoped CeO_2 and Y_2O_3- and Gd_2O_3-doped CeO_2 were synthesized using solid state synthesis. The sintered ceramics were analyzed via x-ray diffraction, and the electrical properties were measured via impedance spectroscopy and current-voltage measurements. In addition, the electrochemical potential measurements were conducted to determine the role of ionic conductivity in these materials. Overall, the doped CeO_2 ceramics exhibited an increase in conductivity with values approaching 10 S/m at 1500 K, with electronic conductivity dominating in the doped compositions. Corrosion tests also indicated these materials are relatively inert in the presence of K_2CO_3 up to 1500°C. Overall, these tests suggest that ceria-based ceramic electrodes show promise for use in MHD generators.

5:10 PM

(ICACC-S12-046-2018) First Principles Investigation on the Mechanical and Thermal Properties of α- and β-$YAlB_4$

F. Dai*[1]; Y. Zhou[1]

1. Aerospace Research Institute of Materials and Processing Technology, Science and Technology of Advanced Functional Composite Laboratory, China

Ultra high temperature ceramics (UHTCs) exhibit a unique combination of excellent properties that makes them promising candidates for applications in extreme environments. Different types of UHTCs are needed due to different harsh conditions that UHTCs are faced with in applications. Currently, investigations on UHTCs are focused on binary compounds, e.g. ZrB_2, which makes the choice of UHTCs quite limited. To extend the database of UHTCs, we investigated the ternary boride $YAlB_4$ with layered structure similar to

that of ZrB_2 in the present work. The electronic structure, chemical bonding, mechanical and thermal properties of both α-type ($YCrB_4$ type, space group Pbam) and β-type ($ThMoB_4$ type, space group Cmmm) $YAlB_4$ were calculated by first principles based on density functional theory. The result reveals that all the properties of α-type and β-type $YAlB_4$ are very similar to each other due to the structural similarity. In addition, both Young's modulus and G/B of $YAlB_4$ are lower than that of ZrB_2, which means that $YAlB_4$ may display better thermal shock resistance and damage tolerance.

S13: Advanced Ceramics and Composites for Nuclear Fission and Fusion Energy

Corrosion and Compatibility

Room: Coquina Salon H
Session Chair: Christian Deck, General Atomics

1:30 PM

(ICACC-S13-035-2018) Effect of irradiation defects on SiC dissolution in aqueous solution (Invited)

S. Kondo*[1]; Y. Maeda[2]; K. Fukami[2]; S. Mouri[3]; T. Hinoki[1]

1. Kyoto University, Institute of Advanced Energy, Japan
2. Kyoto University, Department of Materials Science and Engineering, Guyana
3. Ritsumeikan University, Japan

The applicability of silicon carbide (SiC) and its composites to core components in LWRs is a lively discussion topic. Recently, acceleration of the hydrothermal corrosion of SiC due to self-ion irradiation was reported by authors. The underlying mechanisms, which would be largely different from that for metallic alloys, are still not clear, unfortunately. Particularly in case of SiC semiconductor, the modification of electrochemical dissolution is difficult in absence of assistance from the polarization or illumination. However, the irradiation-induced defects and their localized states could affect the carrier transportation at the water-SiC interface. This work try to associate the irradiation induced defects "or defect levels" with the electrochemical properties of the ion-irradiated single crystalline 3C-SiC in order to understand the underlying mechanisms and to find the corrosion prevention system. Our results showed the strong correlation between the cyclic voltammograms in aqueous solutions saturated with ambient oxygen and the concentrations of defect levels measured by DLTS (Deep Level Transient Spectroscopy). No significant correlation was observed in the voltammograms obtained using deoxygenated solutions, implying the anodic SiC dissolution, due likely to hole injection from the dissolved oxygen, was accelerated by introduction of defect levels within a bandgap.

2:00 PM

(ICACC-S13-036-2018) Evaluation of Environmental Barrier Coatings on SiC materials in Simulated Boiling Water Reactor (BWR) Environments

S. S. Raiman*[1]; P. J. Doyle[2]; C. Ang[3]

1. Oak Ridge National Lab, Corrosion Science, USA
2. University of Tennessee, USA
3. Oak Ridge National Lab, USA

Silicon carbide ceramic matrix composite (CMC) materials have shown promise as next generation light water reactor (LWR) fuel cladding. A major unresolved issue prohibiting the use of SiC_f/SiC in LWR cores is their compatibility with high temperature water. Both CVD SiC and SiC_f/SiC have been shown to dissolve in LWR environments and will require the use of environmental barrier coatings to prevent aqueous dissolution. This talk presents recent experimental results in which SiC materials with CrN, Cr, and TiN coatings were exposed for up to 600 h in 288°C water with either 2 wppm dissolved oxygen or 150 wppb dissolved hydrogen to simulated boiling water reactor normal water chemistry (NWC) and hydrogen water chemistry (HWC). In both conditions, Cr and CrN coated coupons exhibited significantly less mass loss than uncoated coupons. TiN coated coupons gained mass due to the formation of an adherent TiO layer. Samples were characterized with SEM, Raman, and light microscopy to show how the coatings evolved over the course of the exposures.

2:20 PM

(ICACC-S13-037-2018) Silicon carbide volitilization in ultra-high temperature hydrogen environments

K. M. Benensky*[1]; K. Terrani[2]; S. J. Zinkle[1]

1. University of Tennessee, Nuclear Engineering, USA
2. Oak Ridge National Lab, USA

Nuclear thermal rockets require fuels capable of operating in a hydrogen environment at ultra-high temperatures (2000+ K) for short lifetimes. Fuel forms must also possess nuclear and structural properties to ensure engine criticality and integrity. There exist knowledge gaps in the corrosion behavior of most materials in the presence of flowing hydrogen at temperatures anticipated at the hot end of the rocket. To assess the use of silicon carbide (SiC) as a structural matrix material for nuclear rocket applications, cylindrical SiC coupons were cycled in flowing hydrogen at temperatures of 2000, 2250, and 2500 K. Two SiC variants, the high purity chemical vapor deposition (CVD) and nano-infiltration transient eutectic (NITE) containing impurities as sintering additives (Al_2O_3 + Y_2O_3), were examined. The volatilization behavior of SiC materials was studied using mass loss measurements and microstructural characterization in order to access the anticipated lifetime and degradation behavior of SiC formed by different processes. CVD-SiC exhibits adequate compatibility with hot hydrogen up to 2500 K, whereas NITE-SiC experiences significant mass loss at temperatures ≥ 2250 K. Preliminary research on secondary effects including gas velocity and sample geometry are also presented.

2:40 PM

(ICACC-S13-038-2018) Characteristics of the Hydrothermal Corrosion SiC Under Normal Light Water Nuclear Reactor Conditions

P. J. Doyle*[1]; S. S. Raiman[2]; K. Terrani[2]

1. University of Tennessee, Nuclear Engineering, USA
2. Oak Ridge National Lab, USA

Accident-tolerant fuel (ATF) is an increasingly important research topic for the nuclear industry, and ceramics such as SiC are strong contenders for deployment as ATF cladding, for reasons stemming from low neutron absorption cross-section, excellent mechanical properties, and radiation damage resistance. The hydrothermal corrosion characteristics of chemical vapor deposited (CVD) SiC were investigated with a constantly-refreshing autoclave. Tests were performed at 288^{0}C corresponding to BWR power plants with 2ppm dissolved oxygen or 0.3ppm dissolved hydrogen, and 330^{0}C with 3.5ppm dissolved hydrogen, corresponding to PWR power plant conditions. 600 hour tests were conducted in 3 segments of 200 hours each. Mass change between the tests was coupled with pre- and post- EBSD and AFM maps to determine uniform and grain boundary corrosion characteristics, as well as detailed chemical reaction mechanisms. SEM and EDS were coupled with Raman spectroscopy to analyze any surface chemistry that was occurring. Grain boundary attack on SiC was found to be extensive under oxygenating (BWR-NWC) conditions, while this attack is dramatically reduced when oxygen is scavenged from the system. Corrosion rates of SiC are such that it is unlikely that SiC will be a viable candidate for ATF cladding unless corrosion can be reduced, such as with a mitigation coating.

3:20 PM

(ICACC-S13-039-2018) Hydrothermal corrosion behavior of ZrC-SiC composite matrix for SiC composites tube

H. Lee*[1]; S. Lee[1]; J. Han[2]; D. Kim[1]; J. Park[1]; W. Kim[1]

1. Korea Atomic Energy Research Institute, Republic of Korea
2. Seoul National University, Republic of Korea

SiC ceramics and composites are considered as structural materials for fusion and fission system due to its excellent mechanical properties at high temperature, irradiation tolerance, inherent low activation and other superior physical/chemical properties. SiC composite tubes are one of the candidate fuel cladding for Light Water Reactor (LWR) within the Accident Tolerant Fuel (ATF) design concept. However, SiC composites have the disadvantage impeding their use as an ATF cladding due to hydrothermal corrosion under normal operating condition of LWR. The motivation for evaluate the ZrC-SiC composites as a matrix of the SiC composites tube is to produce ZrO_2 or ZrO_2 mixed oxide film that has comparable or better dissolution resistance to SiC. In this study, ZrC-SiC composite coatings with various ZrC fractions were deposited by chemical vapor deposition (CVD). The microstructure and the phase of the composite coatings were observed by Raman spectroscopy, XRD, SEM, and TEM. Corrosion tests of ZrC, SiC, and ZrC-SiC composite coatings were conducted under simulated pressurized light water reactor (PWR) primary water conditions. The hydrothermal corrosion behavior of ZrC-SiC composite coatings and the influence of the including ZrC phase on hydrothermal corrosion were investigated.

3:40 PM

(ICACC-S13-040-2018) Improvement to hydrothermal corrosion resistance of SiC fibers for SiC/SiC composites

S. Suyama*[1]; M. Ukai[1]; M. Akimoto[1]; K. Kakiuchi[1]; H. Heki[1]

1. Toshiba Corporation, Japan

Recently, SiC/SiC composites have become one of the candidates for structural materials used in accident tolerant fuel systems. We have started optimizing the design of CVD/CVI-SiC/SiC composites for use in hydrothermal environments. In this study, candidate SiC fibers for CVD/CVI-SiC/SiC composites were evaluated in pure water at two operating conditions to determine which SiC fibers were best for preventing or minimizing hydrothermal corrosion of CVD/CVI-SiC/SiC composites. Uncoated SiC fibers were degraded on their own by hydrothermal corrosion in the two above conditions from very early stages. Even when SiC fibers are covered with SiC matrix, CVD/CVI-SiC/SiC composites have a porosity of about 10%. Therefore, to protect SiC fibers from hydrothermal corrosion, carbon coatings on the surface of crystallized SiC fibers were most effective.

4:00 PM

(ICACC-S13-041-2018) Tribological and oxidation behaviour of nitride coatings for protection of zirconium alloy cladding

Z. Gao*[1]; J. Kulczyk-Malecka[2]; Y. Chen[1]; P. Kelly[2]; P. Xiao[1]

1. University of Manchester, School of Materials, United Kingdom
2. Manchester Metropolitan University, United Kingdom

Increasing the oxidation resistance of zirconium alloy cladding during a loss of coolant accident (LOCA) is a key issue in enhancing the accident tolerance of nuclear fuel. A protective coating for the cladding has been developed to improve the oxidation resistance under LOCA, when compared to the bare zirconium alloy. Thin films of zirconium nitride (ZrN), titanium nitride (TiN), and other nitrides, were deposited on the zirconium alloy by reactive magnetron sputtering. Tribological behaviour of the nitride coatings were studied by scratch testing and nanoindentation. The effect of the nitride coatings on the oxidation resistance of zirconium alloys in pressurized water (350°C) or a high-temperature steam environment (600 to 1200°C) have been studied and compared. The structure and phase composition of the coatings and the associated oxide layers after oxidation tests were examined by transmission electron microscopy (TEM), focused ion beam X-ray photoelectron spectroscopy (FIB-XPS) and transmission electron backscatter diffraction (t-EBSD). The effects on the oxidation resistance of nitride coatings in high-temperature steam environment of: the thermal mismatch between nitride coatings and zirconium alloy; the thermal stability of nitride coatings; and the microstructure and phase composition of the oxides that formed on nitride coatings, have all been systematically studied.

4:20 PM

(ICACC-S13-042-2018) High-temperature High-pressure Steam Oxidation of SiC

P. Mouche*[2]; K. Terrani[1]

1. Oak Ridge National Lab, USA
2. University of Illinois at Urbana-Champaign, Nuclear, Plasma, and Radiological Engineering, USA

The decoupled effects of high-temperature steam velocity and pressure on the oxidation of CVD silicon carbide (SiC) were investigated at 1200°C. SiC, Si, and amorphous SiO_2 coupons were exposed to pressure increases from 0.101MPa to 1.37MPa at a fixed steam velocity of 0.25cm/s, and increasing steam velocities from 0.25 cm/s to 0.89 cm/s at a fixed pressure of 0.34MPa for time periods up to 32 hours. Initial sample characterization was conducted with a microbalance, jaw micrometer, and Keyence optical microscope. After steam exposure, the oxide growth and SiC recession was characterized using x-ray diffraction, electron and optical microscopy, and glow discharge optical emission spectroscopy. Oxidation kinetics as a function of pressure and steam velocity are calculated from the both the oxide and remaining SiC thicknesses. Porous oxides were observed at all pressures, however, dense nodular cristobalite SiO_2 agglomerates occurred under certain conditions. Significant oxide spalling manifested at higher steam velocities and longer exposure times. The porosity appears unique to the furnace configuration and environment. Unlike other experiments in non-SiC furnaces, most internal surfaces in the furnace used were SiC-based. This leads to more sources of CO, volatilized silicon hydroxide, and H_2, increasing their partial pressures in the steam which promotes bubble formation in the oxide film, compromising its functionality.

S14: Crystalline Materials for Electrical, Optical and Medical Applications

Optical Material II

Room: Tomoka C

Session Chairs: Romain Gaume, University of Central Florida; Yuntao Wu, University of Tennessee

1:30 PM

(ICACC-S14-030-2018) Defect engineering by codoping in $KCaI_3:Eu^{2+}$ single-crystalline scintillators (Invited)

Y. Wu*[1]; Q. Li[2]; M. Zhuravleva[1]; C. Melcher[1]

1. University of Tennessee, Materials Science and Engineering, USA
2. Physical Science Division, IBM Thomas J Watson Research Center, USA

Eu^{2+} doped alkali or alkali earth iodide scintillators with energy resolutions ≤3% at 662 keV promise the excellent discrimination ability for radioactive isotopes required for homeland security and nuclear non-proliferation applications. To extend their applications to X-ray imaging, such as computed tomography scans, the intense afterglow which delays the response time of such materials is an obstacle that needs to be overcome. However, a clear understanding of the origin of the afterglow and feasible solutions is still lacking. In this work, we present a combined experimental and theoretical combined investigation of the physical insights of codoping-based defect

engineering which can reduce the afterglow effectively in $KCaI_3:Eu^{2+}$ single crystal scintillators. We illustrate that Sc^{3+} codoping greatly suppresses the afterglow, whereas Y^{3+}, Gd^{3+}, or La^{3+} codoping enhances the afterglow. Meanwhile, a light yield of 57,000 photons/MeV and an energy resolution of 3.4% at 662 keV can be maintained with the appropriate concentration of Sc^{3+} codoping, which makes the material promising for medical imaging applications. Through our thermoluminescence techniques and density functional theory calculations, we are able to identify the defect structures and understand the mechanism by which codoping affects the scintillation performance of $KCaI_3:Eu^{2+}$ crystals.

2:00 PM

(ICACC-S14-031-2018) Observation of positive hysteresis in scintillators (Invited)

T. Yanagida*[1]; G. Okada[1]; N. Kawano[1]; N. Kawaguchi[1]

1. Nara Institute of Science and Technology, Japan

Scintillator materials is one of the luminescent materials which convert invisible ionizing radiations to visible photons immediately. It is known that some scintillators show the positive hysteresis phenomenon. The positive hysteresis is an enhancement of the scintillation light yield after huge radiation exposure, and this phenomenon is also called the radiation burn or radiation drift. In this talk, I will show the positive hysteresis of Ce-doped Ce/Zr co-doped GSO and some garnet scintillators activated with Ce^{3+} or Pr^{3+}. In addition, we will propose one interpretation of the origin of this phenomenon by using the Synchrotron data.

2:30 PM

(ICACC-S14-032-2018) Non-hygroscopic scintillators for thermal neutron detection (Invited)

N. Kawaguchi*[1]; N. Kawano[1]; G. Okada[1]; T. Yanagida[1]

1. Nara Institute of Science and Technology, Graduate School of Materials Science, Japan

The ^3He gas proportional counter is widely used for detecting neutrons; however, alternative neutron detectors equipped solid scintillators are required due to the lack of ^3He gas in recent years. Solid neutron scintillators, which contain ^6Li, ^{10}B or Gd elements, emit luminescence by the excitation energy generated from nuclear reactions between these elements and thermal neutrons. The ^6Li containing materials are mainly used for solid neutron scintillators because the generated energy from ^6Li is the highest (Q=4.8 MeV) among these elements. Most of lithium compounds show hygroscopicity, and conventional neutron scintillators with high light yields are also strongly hygroscopic (e.g., LiI:Eu and Cs_2LiYCl_6:Ce). In the past few years, we have investigated scintillation properties of various fluoride-based non-hygroscopic phosphors for thermal neutron detections (e.g., LiF:W, LiF/CaF_2:Eu eutectic, $LiCaAlF_6$:Ce and $LiCaAlF_6$:Eu). Among these materials, $LiCaAlF_6$:Eu shows the highest light yield (typically ~20,000 photons/MeV). In this presentation, we introduce fluoride-based non-hygroscopic scintillators for thermal neutron detection studied through the course of R&D.

3:20 PM

(ICACC-S14-033-2018) Scintillation Properties of Organic–Inorganic Layered Perovskite-type Compounds under Gamma-ray Radiation (Invited)

N. Kawano*[2]; M. Koshimizu[1]; G. Okada[2]; Y. Fujimoto[1]; N. Kawaguchi[2]; T. Yanagida[2]; K. Asai[1]

1. Tohoku University, Graduate school of Engineering, Japan
2. Nara Institute of Science and Technology, Graduate school of Materials Science, Japan

Scintillation properties of organic–inorganic layered perovskite-type compounds were investigated. A crystal of the hybrid compounds with phenethyl amine was fabricated by the poor-solvent diffusion method. The scintillation light yield of the crystal was estimated to be 14000 photons per MeV. The scintillation light yield was the highest among the organic–inorganic hybrid scintillators. Moreover, scintillation light yield of the crystal was proportional with gamma-ray in the 122–662 keV energy range. In addition, the decay time of the crystal was 11 ns which was much faster than that of GSO:Ce (48 ns) under X-ray radiation. These results indicate that organic–inorganic layered perovskite-type compounds are promising scintillators for gamma-ray detection.

3:50 PM

(ICACC-S14-034-2018) Field-Assisted Sintering and Phase Transition of ZnS-$CaLa_2S_4$ Composite Ceramics (Invited)

Y. Li*[1]; L. Zhang[2]; K. Kisslinger[2]; Y. Wu[1]

1. Alfred University, Kazuo Inamori School of Engineering, New York State College of Ceramics, USA
2. Center for Functional Nanomaterials, Brookhaven National Laboratory, USA

In the present study, zinc sulfide (ZnS) and calcium lanthanum sulfide (CaLa2S4, CLS) composite ceramics were consolidated via field-assisted sintering of 0.5ZnS-0.5CLS (volume ratio) composite powders at 800-1050 degree Celsius. Through sintering curve analyses and microstructural observations, it was determined that between 800 and 1000 degree Celsius, grain boundary diffusion was the main mechanism controlling grain growth for both the ZnS and CLS phases within the composite ceramics. The composite ceramics have been determined to consist of sphalerite ZnS, wurtzite ZnS and thorium phosphate CLS, with the presence of wurtzite indicative of an early ZnS phase transition due to the small particle size of the starting ZnS powders. TEM investigation of the consolidated ceramics has further revealed that the sphalerite-wurtzite phase transition of ZnS is accompanied by the formation of stacking faults and twins. It was also found that the addition of the CLS phase improved the indentation hardness of the ceramics relative to pure ZnS by homogeneous dispersion of ZnS and CLS small grains.

4:20 PM

(ICACC-S14-035-2018) Monitoring the Fabrication of Optical Ceramics by LIBS (Invited)

R. M. Gaume*[1]; S. J. Pandey[1]; M. Julian[1]; M. Martinez[3]; J. Hostasa[2]; M. Baudelet[3]

1. University of Central Florida, CREOL, USA
2. CNR ISTEC, Italy
3. University of Central Florida, National Center for Forensic Science, USA

Transparent optical ceramics (TOCs) are important optical materials with applications in street lightning, high-strength windows, electro- and magneto-optical isolators, high-power laser gain media and radiation detectors. Their fabrication relies, in most cases, on powder densification techniques carried out at high temperatures and often promoted by dopants, so-called 'sintering additives'. Achieving sufficient consistency in the fabrication process to guarantee high optical quality in the final product has proven to be a not-so-easy task however. In particular, a very precise control of stoichiometry, beyond the precision of current analytical techniques, is often required. This talk will present our most recent results on Laser-Induced Breakdown Spectroscopy (LIBS) for assessing stoichiometry shifts in $Y_3Al_5O_{12}$ (YAG) and $MgAl_2O_4$ (spinel) ceramics with a precision well within the existence domain of these phases. We have also used this technique to monitor the concentration of sintering additives and unintentional impurities throughout the fabrication process of TOCs.

S16: Geopolymers, Inorganic Polymers and Sustainable Materials

Processing, Microstructure and Properties

Room: Ponce de Leon
Session Chair: Dong-Kyun Seo, Arizona State University

1:30 PM

(ICACC-S16-008-2018) Understanding the Relationship Between Micro and Macro-Scale Properties in Sodium Silicate Activated Slag-Fly-ash Binders (Invited)

K. Sankar*[1]; X. Chen[3]; G. Al-Chaar[2]; W. M. Kriven[1]

1. University of Illinois at Urbana-Champaign, Material Science and Engineering, USA
2. Construction Engineering Research Laboratory, USA
3. University of Illinois at Urbana-Champaign, Department of Civil and Environmental Engineering, USA

Sodium silicate activated slag-fly-ash binders are room temperature hardening binders that have excellent mechanical properties and significantly lower carbon footprint than OPC. In this study, the amorphous phases present in hardened binder mixtures were analyzed as a function of variables namely, cure time, cure temperature and slag/fly ash ratio using ^{27}Al and ^{29}Si magic angle spinning - nuclear magnetic resonance (MAS-NMR) spectroscopy. The results from MAS-NMR were used to explain the observed trend in compressive strength, as a function of the same variables listed above.

2:00 PM

(ICACC-S16-009-2018) Zeolite-based ceramic components through hydrothermal dry synthesis (Invited)

A. Conte*[1]; P. Colombo[1]

1. University of Padova, Industrial Engineering, Italy

Zeolites are three-dimensional, microporous, crystalline solids with well-defined structures that contain aluminum, silicon and oxygen in their regular framework. Zeolites are generally formed in strong alkali solution (Na, K) and in hydrothermal conditions. In this work, inorganic compacts were produced using an innovative approach, where kaolinite was directly converted into a zeolite structure through an hydrothermal synthesis without the addition of any water, and therefore in dry conditions. Zeolite-based components reinforced with metallic fibers and other fillers were also produced. FTIR and XRD analyses were conducted to confirm the formation of the desired phase. Strength and other mechanical properties were evaluated to optimize the composition of the composites.

2:30 PM

(ICACC-S16-010-2018) Use of Geopolymeric Leucite as a Feldspathic Replacement in Dental Ceramics (Invited)

C. Bagci*[1]; S. Yildirim[1]; K. Sevinc[1]; W. M. Kriven[2]

1. Hitit University, Department of Metallurgical and Materials Engineering, Turkey
2. University of Illinois at Urbana-Champaign, Department of Material Science and Engineering, USA

Geopolymers are increasingly being considered as a precursor path to the formation of ceramics. In this context, potassium geopolymers crystallize into leucite (K_2O-Al_2O_3-$4SiO_2$), on heating to 900-1200 °C. This study deals with the using of geopolymeric leucite as a feldspathic replacement in the production of dental ceramics. For this purpose potassium geopolymer was heated at 1200 °C for 3 h in an open air furnace to crystallize. Leucite was used as a replacement in portions of 0 %, 25 %, 50 %, 100 % in all three parts (opaque, dentin, transparent) of the ceramic slurry by weight. The slurries were then poured into $1cm^3$ molds and annealed at 1300 °C for 270 min. Vickers hardness, compressive strength, and microstructural investigations were made on all the dental ceramics prepared. Finally, the feasibility of geopolymeric leucite as a feldspar replacement in production of dental ceramics was discussed in light of the mechanical and microstructural results.

Mechanical Properties

Room: Ponce de Leon
Session Chair: Flavio Silva, Pontificia Universidade Católica do Rio de Janeiro (PUC-Rio)

3:20 PM

(ICACC-S16-011-2018) Thermal resistant alkali-activated materials based on the COx argillite

C. Dupuy*[2]; M. Elie[1]; A. Gharzouni[1]; N. Texier-Mandoki[2]; X. Bourbon[2]; S. Rossignol[1]

1. SPCTS, France
2. Andra, France

The reference solution for the long-lived radioactive wastes management is, at present in France, a deep geological waste disposal. The geological medium chosen is the Callovo-Oxfordian argillite (or COx argillite), laying at a depth of 500 m in the east of the Parisian basin. With regards to the present concepts, building such a facility will lead to the excavation of millions of cubic meters of COx argillite. Different solutions exist to take into account this significant volume of by-products from the disposal digging. Among, potential way of re-use/revalorization, the aim of this study is focused on thermal resistant alkali-activated materials based on the COx argillite. To activate the argillite, different calcination processes are used to compare flash and furnace calcinations. Moreover, different alkaline silicate sources in different forms (solution and powder) have been used and allow to obtain alkali-activated materials. In order to increase the thermal resistance, the addition of different reactants such as metakaolin, calcined-clay or sand, are used. The thermal resistance using a thermal loop with an increase up to 850 °C or a thermal shock demonstrated the possibility to formulate thermal resistant argillite based samples. To optimize the thermal shock resistance the use of mineral additive permit to limit the apparition of viscous flow (SEM) and improve the mechanical properties.

3:40 PM

(ICACC-S16-012-2018) Effects of Applied Electric Potential on the Adhesion of Geopolymer to Steel

T. A. Carlson*[1]; D. Hernandez[1]; M. Ziemann[1]; P. Stynoski[1]; C. P. Marsh[1]; G. Kutyla[2]; W. M. Kriven[2]

1. USACE, USA
2. University of Illinois at Urbana-Champaign, USA

Geopolymers have widespread potential for application in infrastructure and facilities. One application is the use of geopolymer coatings on metallic surfaces to increase corrosion protection over typical polymeric based coatings. This requires a greater understanding of geopolymer to metal adhesion. To investigate the factors contributing to the adhesion of geopolymers to metals, an applied electric potential was employed to investigate effects on adhesive strength, ion migration, reaction kinetics, and any segregation effects. A metakaolin-based geopolymer (Na_2O:Al_2O_3:$4SiO_2$:$11H_2O$) was used to bond two rectangular steel samples together with a 1 in^2 overlap. A variable voltage was applied to the samples throughout the curing process, and the samples were then tested in lap shear to determine the effect of voltage on cured adhesion strength. The results showed a threshold-based preferential adhesion to the cathode. This necessitated further adhesion strength testing of the geopolymer to cathode interface using tensile pull-off methods. The cathode and anode interfaces were characterized using electron microscopy and X-ray spectroscopy to quantify variations in chemical composition. This work demonstrates that the electrochemical response of the geopolymer during curing plays a role in adhesion to steel.

4:00 PM

(ICACC-S16-013-2018) Influence of Nanoporosity on Strength of Inorganic Polysialates: A Molecular Dynamics Study (Invited)

Y. Cui[1]; E. Guleryuz[2]; S. Koric[2]; W. M. Kriven[3]; A. Akono*[1]

1. University of Illinois at Urbana-Champaign, Civil and Environmental Engineering, USA
2. National Center for Supercomputing Applications, USA
3. University of Illinois at Urbana-Champaign, Materials Science and Engineering, USA

In this study, we investigate the influence of nanopores on the mechanical behavior of inorganic polysialates using atomistic simulations. Previous investigations in the scientific literature have demonstrated the nanoporous nature of geopolymer. However, very little emphasis has been devoted to understanding the origin of the nanoporosity and the interplay between nanoporosity, chemistry and mechanical response. Herein, we rely on molecular dynamic simulations which consist of numerical integration of Newton's equations of motion to track instantaneous positions of atoms in an ensemble under certain initial and boundary conditions. The molecular dynamics (MD) method has become a common computational tool for analyzing mechanical properties and structure of crystalline and non-crystalline solids at the nanometer length scale. MD is a critical enabling simulation technology and can provide an atomic-resolution structural detail not accessible through experiments. We simulate diverse amorphous polysialates with different alkali cations and Si:Al ratios. The nanoporosity is found to vary according to the chemistry. Furthermore, the nanoporosity has a strong impact on the stiffness and strength characteristics and the failure mechanisms. The computational framework articulated opens new venues for the study of geopolymer composites.

4:30 PM

(ICACC-S16-014-2018) Strength Properties of Geopolymer Composites Using a Theoretical and Numerical Approach (Invited)

A. Kataruka[1]; E. Guleryuz[3]; S. Koric[3]; W. M. Kriven[2]; A. Akono*[1]

1. University of Illinois at Urbana-Champaign, Civil and Environmental Engineering, USA
2. University of Illinois at Urbana-Champaign, Materials Science and Engineering, USA
3. National Center For Supercomputing Applications, USA

Geopolymer is an inorganic material with high strength and good adhesive, thermal and chemical properties. It has manifold applications like - fire resistant materials, binders, construction material, radioactive waste containment, etc. Different kinds of organic and inorganic fillers are commonly added to geopolymers for enhanced mechanical properties. Due to a wide range of possibility, many experimental studies have been conducted. Although, these studies have brought significant breakthroughs into the science and technology of geopolymer composites, the determinants of strength and toughness are still not fully understood. Thus, the research objective is to shed light on the elasto-plastic behavior of geopolymer composites using theoretical and computational nonlinear micro-mechanics. An analytical upscaling model is developed to predict the homogenized strength response of geopolymer composites as a function of volume fraction of reinforcement and porosity. Additionally, finite element analyses are carried out to account for details like matrix-inclusion interaction and shape of inclusions. Results were validated against experimental studies carried out on 30 different geopolymer composite systems. Hence, this study opens new avenues in geopolymer research by substantially reducing the experimental time and cost, meanwhile enabling us to study a wider range of filler materials.

S17: Advanced Ceramic Materials and Processing for Photonics and Energy

Multifunctional II

Room: Halifax A/B

Session Chairs: Emanuele Orgiu, Institut National de la Recherche Scientifique; Giovanni Fanchini, University of Western Ontario

1:30 PM

(ICACC-S17-028-2018) Heterojunctions of Abundant Materials for Solar Fuel Generation (Invited)

O. K. Varghese*[1]; R. Neupane[1]; M. Paulose[1]

1. University of Houston, Department of Physics, USA

Solar photocatalysis offers a sustainable pathway for generating fuels. The reactions can be performed in gas or liquid phase at room temperature using environmentally friendly ways. During the past four decades a number of materials have been investigated as photocatalysts for processes such as hydrogen generation via water splitting and hydrocarbon production via carbon dioxide recycling. Among these materials, oxide ceramic semiconductors have been recognized as stable photocatalysts. Nonetheless, such ceramics generally have wide band gaps and hence, not capable of converting visible light photons to fuels. We were successful in enhancing the visible light activity of earth abundant wideband gap semiconductors by creating heterojunctions with other oxides and non-oxides. This presentation will give details of this work.

2:00 PM

(ICACC-S17-029-2018) Processing and Properties of Hierarchical Porous Ceramics for Clean Energy (Invited)

F. Akhtar*[1]

1. Division of Mateirals Science, Department of Engineering Sciences and Mathematics, Sweden

Production of carbon neutral fuels is important challenge to reduce greenhouse gas emissions from energy sector. In this regard, separation of carbon dioxide from power-plant fluegas and raw biogas to upgrade it to biomethane economically is a pressing challenge. Microporous ceramics such as Zeolites, aluminophosphates, silicoaluminophosphates and activated carbons holds great potential for decarbonization of gas streams. These microporous ceramics requires tailoring of porosity at various length scales to optimize their properties for economical decarbonization of gas streams. The recent developments on tailoring of porous structure and evolution of properties required for CO_2 separation from power-plant fluegas and raw biogas including CO_2 uptake capacity, high CO_2 over CH_4 and CO_2 over N_2 selectivity, rapid uptake and release kinetics and long term durability will be discussed. We will demonstrate that novel processing approaches such as binderless processing, nanostructurization and electrospinning can produce novel structured materials for production of biomethane economically for small scale and large scale raw biogas plants.

2:30 PM

(ICACC-S17-030-2018) Design of Materials for Advanced Energy Storage (Invited)

C. S. Ozkan*[1]

1. University of California Riverside, Mechanical Engineering, USA

I will describe innovative approaches for the design and synthesis of hierarchical three dimensional graphene hybrid materials which possess characteristics including ultra large surface area, tunability, mechanical durability and high conductivity which are appealing to diverse energy storage systems. Integration of nanostructured pseudocapacitive metal oxides received a lot of attention recently due to their superior electrochemical performance. In order to realize high energy density supercapacitors, we developed a scalable

method to fabricate MGM (graphene/MWNT/MnO_2) and RGM (graphene/MWNT/RuO_2) hybrid systems. The high specific/areal capacitance and extended operational voltage window of 1.5 V lead to an exceptionally high energy density of 39.28 $Whkg^{-1}$ and power density of 128.01 $kWkg^{-1}$. Next, I will talk about three-dimensional cone-shape carbon nanotube clusters decorated with amorphous silicon for lithium ion battery anodes. An innovative silicon decorated cone-shape CNT clusters (SCCC) is prepared by depositing amorphous silicon onto CCC via magnetron sputtering. The seamless connection between silicon decorated CNT cones and graphene facilitates the charge transfer in the system and provides a binder-free technique for fabricating lithium ion batteries. Lithium ion batteries based on this novel 3D SCCC architecture demonstrated a high reversible capacity of 1954 $mAhg^{-1}$ and excellent cycling stability.

3:20 PM

(ICACC-S17-031-2018) Sulfur Cathode Materials for Lithium–sulfur Batteries (Invited)

M. Ozkan*[1]

1. University of California Riverside, Electrical and Computer Engineering Department, USA

In this study, silica-coated sulfur particles (SCSPs) were synthesized and characterized as a cathode material for Li–S batteries. The novel core–shell structure was fabricated in a facile 2-step wet chemical synthesis. The SCSP cathode showed superior cycling stability when coupled with mrGO (mildly reduced Graphene Oxide) as an additive, improving the capacity retention after 50 cycles from 440.8 mAh/g without mrGO to 763.2 mAh/g with mrGO. The electrochemical data also shows reduced capacity fading over 50 cycles, from 12.2 mAh/g per cycle without mrGO to 8.6 mAh/g per cycle with mrGO. During cycling, SCSPs are understood to fracture and release active material (S8), and mrGO helps to contain the ruptured particles, thereby improving cycling stability. By the 50th cycle, SCSPs experienced a 318.8 mAh/g boost in specific discharge capacity with the addition of mrGO. These improvements are attributed to the polysulfide inhibiting effects of SiO2 as well as the host of benefits provided by mrGO, similar to other work. Thus, SCSPs with the addition of mrGO show great promise in the application of low-cost, high energy density battery systems for portable electronics and electric vehicles.

3:50 PM

(ICACC-S17-032-2018) Surfaces in graphene-ceramic nanoparticle composites: Examples from Li ion battery anodes and photocatalysts (Invited)

M. Cerruti*[1]

1. McGill University, Canada

Graphene-nanoparticle composites combine the extraordinary electrical conductivity and stiffness of graphene with specific properties of the ceramic nanoparticles. Their full potential can be exploited if the particle nucleation and bond with graphene are well controlled. Hence, graphene surface properties are crucial in these composites. Here we will discuss first graphene-Sn nanoparticle composites as materials for Li ion battery anodes. We will show how graphene surface functionalization controls nanoparticle nucleation, distribution, and bonding, as well as solid electrolyte interphase stability. We will then move to photocatalytic applications and show that graphene functionalization influences the morphology and facets of TiO_2 nanoparticles synthesized in situ on graphene. This in turn strongly affects the photocatalytic performance of the composites.

4:20 PM

(ICACC-S17-033-2018) Leveraging heterostructural alloying to design metastable nitrides with improved piezoelectric properties

S. Millican*[1]; K. Talley[2]; A. W. Weimer[1]; A. Zakutayev[3]; C. B. Musgrave[1]; G. L. Brennecka[2]; A. Holder[1]

1. University of Colorado, Department of Chemical and Biological Engineering, USA
2. Colorado School of Mines, Department of Metallurgical and Materials Engineering, USA
3. National Renewable Energy Laboratory, USA

Despite success in isostructural alloying, heterostructural alloying remains an unexplored area. In heterostructural alloys, the crossover between crystal structures enables control of the atomic structure by variation of the composition. The deliberate manipulation of local atomic coordination symmetry introduces an additional material design parameter. We present a theoretical and experimental investigation of piezoelectric nitrides to develop design principles for utilizing heterostructural alloying as a material design strategy. We use ab initio methods to predict the structural and electronic properties of $Al_{1-x}Sc_xN$ alloys for the energetically competitive polymorphs and compute the corresponding equilibrium phase diagram and material properties. Combinatorial sputtering is employed as a non-equilibrium growth technique to produce metastable thin-films spanning the composition range. The experimentally validated predictions and theory guided synthesis of piezoelectric heterostructural alloys exemplify how our integrated research strategy is used to design and realize functional metastable materials. We apply these design principles to prototype new metastable nitride alloys with improved piezoelectric properties. Our approach establishes a new route for the control of structure-property and composition-structure relationships by accessing non-equilibrium phase space.

4:40 PM

(ICACC-S17-034-2018) Critical issues and future prospects of particulate magnetoelectric composites

P. Galizia*[1]; C. Capiani[1]; C. Galassi[1]

1. CNR-ISTEC, Italy

Within ten years magnetoelectric multiferroics could be implemented into the emerging technologies such as wireless power, mesh network, etc. Remarkable efforts have been done to develop laminated multilayer multiferroic composites. These structures lead to remarkable magneto-electric coupling coefficients of a few $V\ cm^{-1}\ Oe^{-1}$ because the ferroic multilayer is a "full dielectric" which can be completely poled in the conventional way. On the other hand in the particulate ceramic composites the requirement for "full dielectric" is no longer applicable, since the ferroic phases are fully separated within the composite. The main strength of particulate ceramic composites is the higher strain mediated magneto-electric coupling since electric order phase/magnetic phase interface density can be higher. In this work the process' developments and the functional characterizations of particulate PZT–$CoFe_2O_4$ composites are presented. Up to date, by setting a quite-fast sintering, full densification (up to 99%) and prevention of unwanted reactions were achieved, but reaching the electric saturation of the PZT matrix is still a challenge.

Honorary Sympoisum: Advancing Frontiers of Ceramics for Sustainable Society Development - International Symposium in Honor of Dr. Mrityunjay Singh

Advancing Frontiers of Ceramics V -Characterization

Room: Coquina Salon E

Session Chairs: Junichi Tatami, Yokohama National University; Yanchun Zhou, Aerospace Research Institute of Materials & Processing Technology

1:30 PM

(ICACC-HON-035-2018) Role of Defect Chemistry on Phase Transformation Kinetics, Phase Stability and Phase Diagram Determination in Oxide Ceramics (Invited)

A. V. Virkar*[1]

1. University of Utah, Materials Science & Engineering, USA

Most oxide ceramics of interest are refractory with very low diffusion coefficients on one or more sublattices at low temperatures. High temperature phases are thus metastably retained at low temperatures. As a result, it is generally difficult to determine low/intermediate temperature phase diagrams in a reasonable period of time.. The kinetics of phase transformation can be changed orders of magnitude by doping with oxides of cations of different valence than the host. This allows one to investigate mechanisms and kinetics of phase transformations by either slowing down or expediting transport. Many phase transformation models have been developed on metals. Often these models are however difficult to study in metals since transport cannot be readily changed substantially by adding small amounts of other metals, as can be done in oxides. Also, by suitable doping stability of desired phases in oxides can also be increased. This talk will be based on many years of work in this area, which has importance in many situations in which non-equilibrium processes are used to make materials. Phase transformation mechanisms discussed will include nucleation and growth, spinodal decomposition, massive transformation and order-disorder transformation. The systems to be discussed will include TiO_2-SnO_2, Bi_2O_3-containing materials, $LiAl_5O_8$-$LiFe_5O_8$ to name a few.

2:00 PM

(ICACC-HON-036-2018) Evaluation of crystallographic matching in ZrO_2-CeO_2 shape-memory ceramics (Invited)

E. L. Peng[1]; A. Lai[1]; S. Patala[2]; C. A. Schuh*[1]

1. Massachusetts Institute of Technology, Department of Materials Science and Engineering, USA
2. North Carolina State University, Materials Science and Engineering, USA

ZrO_2-based shape-memory ceramics (SMCs) offer higher transformation stresses, work output, transformation temperatures, and possibly environmental resistance as compared to metallic shape-memory alloys (SMAs). To date, superelastic oligocrystalline SMC particles have demonstrated reversible transformations up to several hundred cycles. However, the fatigue life must be further improved before widespread application becomes feasible. To improve the cyclic repeatability, we examined the recently proposed cofactor conditions, which, if met, ensure excellent crystallographic matching between the austenite and martensite phases and have been used to design some highly reversible low hysteresis metallic SMAs. We applied this framework to calculate the cofactor conditions for a range of compositions in the ZrO_2-CeO_2 SMC system, which predicted reduced hysteresis and improved cyclic performance for some preferred compositions. To validate the theoretical predictions, we have experimentally explored a range of compositions near the theoretical optimum. DSC thermal cycling experiments on polycrystalline samples have been conducted to investigate thermal hysteresis. Data is also presented for superelastic mechanical cycling experiments on single-/oligo-crystalline micropillars to evaluate cyclic stability in this composition range.

2:30 PM

(ICACC-HON-073-2018) Advanced Characterizations of Ceramic and Composite Materials Using High-Energy X-Rays (Invited)

D. Singh*[1]

1. Argonne National Lab, USA

High Energy (>50 KeV) synchrotron x-rays, such as those at Argonne National Lab's Advanced Photon Source (APS), provide myriad of characterization strategies for structure-property investigations for ceramic and composite materials. High energy x-rays allow for significant penetration (several millimeters) of the x-rays into the material, allowing for sub-surface material evaluations. Further, characterizations can be done in an in situ manner allowing for collecting valuable information in a time resolved manner. As part of this presentation, techniques to measure residual stresses, flaws and phase structures, 3D tomographic visualizations, and grain mapping will be discussed. Applicability of the techniques to various ceramics and composite materials will be presented. Part of this research used resources of the Advanced Photon Source, a U.S. Department of Energy (DOE) Office of Science User Facility operated for the DOE Office of Science by Argonne National Laboratory under Contract No. DE-AC02-06CH11357.

2:50 PM

(ICACC-HON-009-2018) Current Progress in Advanced Materials Research

S. Gupta*[1]

1. University of North Dakota, Mechanical Engineering, USA

In this presentation, I will present research findings on three different areas of materials research in Advanced Materials Research Group in University of North Dakota: (a) advanced manufacturing, (b) MAX/MAB phases (novel natural laminates) and their composites, and (c) novel sustainable materials. During Part A, a novel testing methodology for studying advanced manufacturing, which includes the evaluation of free sintering strain, stress-induced dimensional changes, and weight changes, will be presented. This methodology was developed to determine the critical dimensional changes and thermomechanical response during presintering (i.e., before densification). During Part B, recent results on MAX phases and their composites will be presented. During Part C, current progress in materials development from sustainable perspective will be presented.

3:30 PM

(ICACC-HON-038-2018) In-Situ Nanomechanics of One-Dimensional Ceramic Structures: Challenges, Progress and Prospects (Invited)

S. Bhowmick*[1]; B. Ozdol[2]; S. Asif[1]; O. L. Warren[1]; T. Wyrobek[1]

1. Bruker Nano Surfaces (Hysitron), USA
2. Lawrence Berkeley National Laboratory, USA

One-dimensional ceramic structures such as nanowires and nanotubes are potential materials for future nanoelectronics, optoelectronics, piezoelectric devices, sensors, and actuators. Due to length scale effects and higher surface-to-volume ratios, such nanostructures exhibit superior mechanical and electrical, as well as other length-scale dependent properties. To utilize these fundamental advantages, it is essential to investigate and understand their unique characteristics as a function of the material parameters. In spite of the great technological progress that has been made during the last decade to characterize nanostructured materials, comprehensive electromechanical characterization of single individual nanowire is still a difficult task because of their extremely small-scale

dimensions. In this presentation, the challenges and progress on the developments of small-scale devices and nanomechanical testing methodology that can characterize ceramic nanowires will be discussed. A few case studies of understanding strain-induced electronic mobility, mechanical, and piezoelectrical behavior of ZnO, InAs, and InAs-GaAs nanowires will be presented.

3:50 PM

(ICACC-HON-039-2018) Effect of in-situ ZrB_2 on the mechanical properties and ablation resistance of SiBCN ceramics (Invited)

D. Jia*[1]; Y. Miao[1]; Z. Yang[1]; Y. Cheng[2]; Y. Zhou[1]

1. Harbin Institute of Technology, China
2. Monash University, Australia

Here we used two methods to introduce ZrB_2 into SiBCN ceramics prepared by SPS (spark plasma sintering), and effect of the in-situ ZrB_2 on mechanical properties and ablation resistance the SiBCN matrix ceramics was reported. In one approach Zirconium n-propoxide (ZNP), the precursor of zirconia was introduced then reacted with amorphous BN(C) from the matrix, forming ZrB_2 phase, labeled as SZ. In the other method, zirconium n-propoxide (ZNP), boric acid and furfuryl alcohol ($C_5H_6O_2$) (FA), the precursor of zirconia, boron oxide and carbon were introduced into SiBCN matrix by sol-gel method to form ZrB_2 through carbon/borothermal reduction, labeled as SZB. Results show that after high temperature sintering, the main phases were SiC, BN (C) and ZrB_2 for both of the two routes by sol-gel method. The mechanical properties of the SiBCN ceramics were improved with the addition of in-situ ZrB_2. The fracture mode for both ceramics are mainly transcrystalline fracture for the introduction of the ZrB_2 phases. The ablation resistance corresponding to the oxyacetylene flame ablation were also assessed by mass and liner ablation rate respectively for SZ and SZB samples with 15wt%ZrB_2 ceramics. Analysis of the material after thermal ablation testing showed that ablation products mainly consisted of the $ZrSiO_4$, SiO_2 and ZrO_2 phases. Main ablation mechanisms of the ceramics were also assessed.

4:10 PM

(ICACC-HON-040-2018) Turning the properties of ultrahigh temperature ceramics through doping strategies (Invited)

Y. Zhou*[1]; F. Dai[1]

1. Aerospace Research Institute of Materials & Processing Technology, China

Transition metal borides have contributed to the success of ultra-high-temperature materials (UHTM) development. Current ZrB_2 and HfB_2-based UHTMs exhibit high-temperature stability, high strength and modulus, but inevitable brittleness due to strong covalent bonding. It is well established that both chemical composition and bonding play important roles in dictating the structure and properties of a material. Therefore, modifying of the composition and nature of bonding is efficient in turning properties and overcoming intrinsic brittleness of ZrB_2. In this presentation, we will demonstrate that through the doping strategies tailoring the properties of ZrB_2 becomes feasible. In our first doping strategy, we select elements with d electrons tending to be full-filled such as Pd and Pt to prevent electron transferring from alloying element to the electron deficient B-B π orbits, which will reduce the local stability surrounding the alloying element and ideal shear strength. In our second doping strategy, high valence electron elements such as Mo, W, Ta, Nb are selected to strengthen the grain boundary of ZrB_2. These strategies are expected beneficial to toughening and enhancing high temperature strength of ZrB_2. Finally, the effect of doping elements on the surface properties and on grain morphologies of ZrB_2 will be demonstrated.

4:30 PM

(ICACC-HON-041-2018) Measurement of grain boundary strength of the neck in porous SiC ceramics using microcantilever beam specimens

J. Tatami*[2]; Y. Imoto[2]; M. Iijima[2]; T. Yahagi[1]; T. Takahashi[1]

1. Kanagawa Institute of Industrial Science and Technology, Japan
2. Yokohama National University, Graduate School of Environment and Information Sciences, Japan

Porous SiC ceramics are used for diesel particulate filter. Their higher strength is desired to improve the thermal shock resistance. Because they are usually broken from the grain boundary of a neck, the grain boundary strength should be a dominant factor of the strength of the porous ceramics. In this study, we directly measured the grain boundary strength of a neck in porous SiC ceramics using microcantilever beam specimens. Porous SiC having particle size of about 10 mm was used in this study. Microcantilever beam specimens were made by the focused ion beam technique. Their width, thickness and length were about 1, 3 and 10 mm, respectively, and the section profile was pentagonal. A grain boundary existed at the end of the microcantilever beam specimen. Bending test was carried out to measure the fracture load using a nanoindentor. The fractography showed that the fracture in the microcantilever beam specimens occurred in the grain boundary, which means that the measured strength should be the grain boundary strength. The measured bending strength was 18.8 GPa, which is much higher strength than the bending strength of the dense and porous bulk SiC ceramics. The strength of the porous SiC ceramics was quantitively explained by the grain boundary strength of a neck, pore size, neck radius and its size effect in the Weibull distribution.

4:50 PM

(ICACC-HON-042-2018) Critical Role of Interfacial Characterization in Integration of Silicon Carbide Ceramics for Advanced Energy and Aerospace Systems (Invited)

H. Tsuda*[1]; T. Ozaki[2]; Y. Hasegawa[2]; S. Mori[1]; M. C. Halbig[3]; R. Asthana[4]; M. Singh[5]

1. Osaka Prefectue University, Graduate School of Engineering, Japan
2. Osaka Research Institute of Industrial Science and Technology, Japan
3. NASA Glenn Research Center, USA
4. University of Wisconsin-Stout, USA
5. Ohio Aerospace Institute, USA

Silicon carbide ceramics are enabling materials for a number of advanced energy and aerospace applications due to their excellent high temperature mechanical properties, oxidation resistance and thermal stability. However, manufacturing of large and complex shape components is often expensive and time consuming. In order to produce large and complex shaped components, a variety of bonding and integration approaches have to be developed. Previously diffusion bonding has been used as a joining method for SiC-based materials. However, in many cases, detailed microstructural observation of the bonded region by transmission electron microscopy (TEM) has not been conducted due to difficulty in preparing TEM samples despite its critical importance. Recently, we succeeded in preparing good TEM samples from the diffusion bonded region of CVD-SiC and fiber bonded ceramic (SA-Tyrannohex) by FIB (Focused Ion Beam) system. TEM observation of diffusion bonded interfaces has been conducted in PVD-Ti as well as thin Ti, Mo-B, Ti-Mo, and Ti-Cu as joint interlayers. In this presentation, experimental results and critical role of interfacial characterization in the integration of silicon carbide ceramics for advanced energy and aerospace systems will be discussed.

5:10 PM

(ICACC-HON-043-2018) Minkowski Hull and Ceramics Intergranular Phenomena

V. Mitic*[1]; L. Kocic[2]; V. Paunovic[2]; S. Tidrow[3]; B. Vlahovic[4]; H. Fecht[5]

1. Serbian Academy of Sciences, Institute of Technical Sciences, Serbia
2. University of Nis, Serbia
3. Alfred University, USA
4. North Carolina Central University, USA
5. University of Ulm, Germany

For many characteristics of this material from mechanical to electric or optical are responsible intergranular contacts in materials obtained by powder processing technologies. In sintered ceramics, special importance of inter-grain contacts is emphasized due to characteristics of the liquid sintering phase. The electric properties, heavily depend on the microstructure of the close grain's vicinity that is known as intergranular region. To gain manipulability over such regions it is helpful to introduce Minkowski hull (MH). Some properties of MH are studied and some applications are suggested, such as energy applications and grains coating issues. The whole construction leads to a natural generalization which makes possibility of introducing fractal forms, which, in fact are in capacity to realistically describe the surface of the grains. Using generalized Minkowski hull, the extreme situations that then explain some details connected to micro-capacities, thermodynamics, ferroelectric etc. This concept is very useful in defining the measure of closeness w_{ij} between two or more grains from disjoint hulls to stable three-point grains' contact. In combination with space configuration of grains' network such closeness measure the intergranular contacts fractal configuration of different thickness, is modeled. It causes many versatile microelectronic situations that implies corresponding material behavior.

FS1: Bio-inspired Processing of Advanced Materials

Bio-inspired Processing I

Room: St. John

Session Chair: Zhengyi Fu, Wuhan University of Technology

1:30 PM

(ICACC-FS1-001-2018) Current Research on Bioinspired Materials in State Key Lab of Metal Matrix Composite (Invited)

D. Zhang*[1]; J. Gu[1]; Z. Li[1]; W. Zhang[1]; Q. Liu[1]; Y. Li[1]

1. Shanghai Jiao Tong University, China

Biological materials naturally display an astonishing variety of sophisticated nanostructures that are difficult to obtain even with the most technologically advanced synthetic methodologies. Inspired from nature materials with hierarchical structures, many structural and functional materials are developed based on templating. We focused on creating bioinspired structure materials and replicating the morphological characteristics and the functionality of a biological species. To improve Al composites, the structure and toughened mechanisms of nacreous layer were introduced. Biomimetic laminated CNT(carbon nano tube)/Al composites were designed and fabricated to obtain both strength and toughness. While we changed original components of nature structures into desired materials, original morphologies were faithfully kept. Based on these results, we discuss the possibility of using these materials in photonic control, solar energy harvest, electromagnetic shielding, energy harvesting, and gas sensitive devices, et al. Related research will be presented by members of our research group. These bioinspired structure and functional materials with improved performance characteristics are becoming increasing important, and will have great impact on the development on structural function materials in the near future.

1:50 PM

(ICACC-FS1-002-2018) Construction of photonic crystals with opal and inverse opal structures and their applications (Invited)

J. Zhao*[1]

1. Harbin Institute of Technology, School of Chemistry and Chemical Engineering, China

We present the recent experimental investigation of 3D ordered structures, or photonic crystals (PCs) based on the functional materials, which are inhomogeneous materials whose dielectric properties vary periodically in space on a macroscopic scale. These ordered materials have novel and interesting properties concerning both basic physics and technological applications. For three dimensional photonic crystals, various techniques have been used including photolithography and etching techniques. Some of these techniques are already commercially available. To circumvent nano technological methods with their complex machinery, alternate approaches have been followed to grow photonic crystals as self-assembled structures from colloidal crystal. After a brief description of the main properties of photonic crystals, we present the self-assembly methods for the construction of photonic crystals with inverse opal structures by using colloidal crystal templates. The 3D PCs can be built with different building blocks and different materials, including semiconductors, alloys, metals, metal oxides and polymers. All these materials are very attractive for practical applications because it is possible to govern of their optical properties with the unique structure.

2:10 PM

(ICACC-FS1-003-2018) Advanced processing of ceramics: Taking cues from natural materials (Invited)

F. Bouville*[1]; H. Le Ferrand[1]; T. Niebel[1]; A. Studart[1]

1. ETH Zürich, Complex Materials, Switzerland

Heterogeneous composites with intricate microstructures are widely spread in the natural world where they are needed to fulfil the specific functional demands imposed by their environment. Understanding the principles of the relationship between microstructure and properties has led to new conceptual designs for multifunctional composites. Additionally, strong mineral composites in living organisms or even dense rocks can sometimes be obtained under much milder conditions than what is done industrially. However, applying the multiplicity of nature's strategies to man-made materials is yet a challenge due to the lack of suitable and easily available processing tools. During this presentation, we will go through two newly developed processes: a cold sintering method inspired by geological phenomena and the magnetically assisted slip casting of anisotropic particles. These techniques allow us to reproduce the structure or mechanisms found in natural materials while adding flexibility in terms of compositions and/or fabrication time.

2:30 PM

(ICACC-FS1-004-2018) Architecture and Interface Design for Highly Conductive Graphene/Copper Composite (Invited)

D. Xiong*[1]; M. Cao[1]; D. Zhang[1]; Z. Li[1]

1. Shanghai Jiao Tong University, China

Recently, tailoring properties by architecture design that changes the spatial distribution of reinforcement in matrix at micro-/nano-scale without changing constituents has attracted intensive attention in the community of composite. Metals can be strengthened by adding hard reinforcements, but such strategy usually compromises ductility and toughness as well as electrical/thermal conductivity. In past few years, a bioinspired strategy has been applied to surmount the dilemma in our research. By assembling copper nanoflakes cladded with graphene, graphene/copper matrix composites with a natural nacre inspired nanolaminated architecture have been prepared. Owing to a combined effect from the bioinspired nanolaminated architecture and improved interface bonding, a synergism

has been made between mechanical strength and ductility as well as electrical/thermal conductivity in graphene/copper matrix composites. The bioinspired nanolaminated architecture enhances the mechanical strengthening and electrical/thermal conducting efficiencies of two-dimensional graphene by alignment of graphene that orient to maximize performance for required loading and carrier transporting conditions, and toughening by crack deflection. The strategy sheds light on the development of structural-multifunctional integrated composites.

2:50 PM

(ICACC-FS1-005-2018) Bio-inspired chromic composite Materials (Invited)

Y. Li*[1]

1. Harbin Institute of Technology, China

The reversible persistent electrochromic change in color or transparency controlled by a temporarily applied electrical potential is attractive because of its low energy consumption. Conventional flat film devices suffer from limited ion intercalation and therefore from a poor electrochromic performance. A simple solution to this problem is the construction of orderded porous network structure inspired by ordered structure from nature. The formation of the ordered porous structure in electrochromic materials not only enables the expansion and contraction the host materials during guest insertioin and extraction, but also increases the surface-to-volume ratio. Our study demonstrates that electrochromic materials constructed in a three-dimensional highly ordered porous structure enhances the electrchromic performance. Our fabrication strategy employs the colloidal crystal templates (CCT) to build the 3D porous structure. Typically, the interstitial spaces of CCT are filled by the desired materials and the templates are removed, creating the ordered porous structure or inverse opal. The method can be applied to most inorganic and polymeric materials, such as WO_3, V_2O_5, polyaniline, etc. Such structure presents a high surface-area-to-volume ratio with nanometer-sized walls for short diffusion lengths and high active surface area for large number of intercalation sites, resulting in high optical contrast and fast switching response.

3:30 PM

(ICACC-FS1-006-2018) Transparent Anti-fogging Nanocomposite Films with Multi-functionalities (Invited)

A. Hozumi*[1]; T. Sato[1]; C. Urata[1]; N. Shing[1]

1. National Institute of Advanced Industrial Science and Technology (AIST), Japan

In this study, we report transparent anti-fogging nanocomposite thin films with multi-functionalities. Our composite films are made from a mixture of positively-charged nano-scale clay platelets (AMP) and the partially-charged polymer polyvinylpyrrolidone (PVP), which tightly adhere to a substrate using surface modification, cross-linking, and non-covalent gelation. Our process consists of three steps, which improve adhesion properties. Firstly, a UV/ozone-cleaned surface was modified with aminopropyl functionalities via vapor phase deposition, then an AMP/PVP layer cross-linked with glutaraldehyde (GA) was spin-coated over the top. Finally, a thicker PVP/AMP layer was immediately deposited on this surface, which formed a non-covalent, but insoluble gel surface. The resulting films were highly transparent and exhibited excellent anti-fogging properties due to their high hydrophilicity. When cooled to ~3°C and exposed to humid air, they showed repeatable anti-fogging properties by preventing water droplets formation, and these properties remained intact for several months after sample preparation. In addition, thanks to the presence of GA as a cross-linker, our composite films adhered tightly to the surface, as well as excellent anti-bacterial properties. We thus expect such remarkable properties to be readily applicable to a wide variety of advanced applications.

3:50 PM

(ICACC-FS1-007-2018) Confined-space synthesis of nanostructured anatase, directed by modified living organisms for energy storage

H. Ping*[1]; H. Xie[1]; Z. Fu[1]

1. Wuhan University of Technology, China

The natural formation processes of biominerals are temporally and spatially regulated under the functions of biomolecules in a confined space. It is potentially very productive to rationally design a mineralized system by taking into account confinement as well as biomolecules. Bacterial cell surface display is an ideal platform to host catalytically active proteins in a three-dimensionally confined space. Aiming to regulate the synthesis of nanostructured TiO_2 anatase, repeating segments of silaffin were displayed on Escherichia coli surfaces through genetic manipulation. The displayed protein electrostatically interacted with a titanium source and catalyzed the hydrolysis of titanium dioxide precursors through hydrogen bonding interactions on the cell surface. The genetically modified cells not only served as a framework for producing rod-shaped TiO_2 assembled by nanoparticles, but also provided a carbon source in situ. The as prepared TiO_2 anatase exhibited unique characteristics including nanosized anatase crystals, mesoporous structure and carbon coating. As the anode electrode of a lithium-ion battery, the carbon coated anatase shows a excellent energy storage performances. This bioprocess-inspired approach may help broaden the scope and impact of nanosized biominerals.

4:10 PM

(ICACC-FS1-008-2018) A Phrynocephalus helioscopusP-inspired flexible electrochromic skin with variable color and infrared emissivity based on conducting polymer

L. Zhang*[1]; Y. Li[1]; S. Dou[1]

1. Harbin Institute of Technology, School of Aeronautics, China

Phrynocephalus helioscopusP have the remarkable capability to change its skin color and infrared emissivity to survive under the extreme conditions in desert. This unique characteristic has long inspired researchers to develop materials and devices to mimic the functions. However, it requires the excellent electrochromic materials and ingenious structure design. Here we show an all-solution processed Phrynocephalus helioscopusP-inspired flexible electrochromic skin, in which flexible electrochromic skin color and infrared emissivity can easily be controlled through varying the applied potential along with the applied potential duration. This system will have wide range applications such as IR camouflage for military and thermal control for satellite.

4:30 PM

(ICACC-FS1-009-2018) In-situ controllable synthesis of continuous three-dimensional graphene network reinforced copper matrix composites

X. Zhang*[1]; C. He[1]; N. Zhao[1]

1. Tianjin University, School of Materials Science and Engineering, China

Graphene has been emerging as an attractive reinforcement for composites due to its unique mechanical and electrical properties as well as its two-dimensional flexible structure. It is a great challenge to efficiently combine the graphene with the metal matrix for achieving excellent mechanical and physical performance of the metal matrix composites (MMCs). In this work, we initially proposed an easy and scalable strategy to in situ synthesizing the continuous three-dimensional network graphene (C3DNG) in the copper matrix by chemical vapor deposition (CVD) followed by powder metallurgy method. The C3DNG/Cu bulk composites after solidification exhibited extraordinary mechanical and electrical properties. Based on the experimental results as well as finite element method calculations, we have found that the continuous three-dimensional network feature of C3DNG distinguishes it from

traditional MMCs reinforced by discontinuous zero-dimensional nanoparticles and one-dimensional fibers on the aspect of both strengthening and toughening mechanisms.

Poster Session B

Room: Ocean Center Arena

5:00 PM

(ICACC-S1-P069-2018) The Microstructure, Mechanical Properties and Ablative Mechanism of the SiBN ceramics by Mechanical Alloying and Hot Pressing

Z. Yang*[1]; X. Liao[1]; D. Jia[1]; Y. Zhou[1]

1. Harbin Institute of Technology, China

The novel SiBN ceramics with excellent mechanical properties and outstanding high-temperature property are regarded as one of the best comprehensive performance materials using for high temperature application. The fabrication of dense SiBN monoliths is a processing challenge for conventional polymer precursors pyrolysis process and chemical vapor deposition. We report here successful densification of SiBN monoliths achieved by mechanical alloying and hot pressing at 1800°C and 40MPa. The dense amorphous/nano-crystal SiBN monoliths show the controllable microstructure and mechanical properties with the range of which bending strength is 74MPa to 270MPa and elastic modulus is 35GPa to 169GPa at room temperature, respectively, by designing the Si/B atomic ratio and sintering parameters. Meanwhile, the dense SiBN monoliths own outstanding anti-ablation property of which the lowest mass ablative rates is only 0.0001g/s.

(ICACC-S1-P070-2018) Damage Accumulation Behavior of C/SiC Under Compression

Y. Tobata*[1]; K. Goto[2]

1. Graduate University for Advanced Studies, Space and Astronautical Science, Japan
2. Japan Aerospace Exploration Agency, Institute of Space and Astronautical Science, Japan

Damage accumulation behavior in short fiber reinforced carbon fiber SiC composite fabricated by Si infiltration technique under compression were precisely observed in this study. Compression tests were conducted at room temperature on rectangular shaped specimens of 10 mm square cross-section with 20 mm height. For damage observation, four side surfaces of a specimen were polished with 0.25 μm diamond paste. Compression test was conducted with repeated loading-unloading cycles until total fracture. Polished surface of the specimen was observed by an optical microscope after unloading to find crack initiation and/or propagation. The stress-strain relation during compression test was recorded with strain gages adhered on surface of the compression specimens. Mechanical responses clearly show that the damage accumulation was existed in the specimen while no plastic deformation would exhibit in C/SiC at room temperature. Number of cracks increased with load especially in Si rich matrix region. All of the cracks propagated into Si rich matrix region stopped at fiber bundle region running perpendicular to the crack propagation direction. The crack propagation direction did not show strong relation with loading direction. Some crack propagates even perpendicular to the loading direction, where the applied compressive stress forced the crack to be close.

(ICACC-S1-P071-2018) Fiber/matrix interface characterization of SiC/SiC fiber bundle composite

Y. Matsumura*[1]; K. Goto[2]; Y. Kogo[1]; R. Inoue[1]; T. Matsuda[3]; S. Takahashi[3]; S. Kitaoka[3]; A. Ito[4]

1. Tokyo University of Science, Department of Materials Science and Technology, Japan
2. Japan Aerospace Exploration Agency, Japan
3. Japan Fine Ceramics Center, Japan
4. Yokohama National University, Japan

Oxide ceramics fiber/matrix interfaces for SiC/SiC ceramic matrix composite were examined in this study. First, fiber bundle composite composed by a single SiC fiber bundle (Tyrano-SA and Hi-Nicalon typeS) were evaluated. Fiber bundle composites were fabricated by both polymer infiltration and pyrolysis and Si impregnation techniques. As the evaluation method, tensile strength was measured by tensile test, fracture surface was observed with SEM to check fiber pull out length. Fabrication of SiC/SiC fiber bundle composites were succesful and the tensile test could be conducted. Fiber and matrix strongly bonded in SiC/SiC fiber bundle composite without any interface controls and no fiber pull out could be observed. After fabrication technique of the SiC/SiC fiber bundle composite were established, high water vapor oxidation resistance ceramics coating made of $Y_2Si_2O_7$ and ZrO_2 were examined by established technique of SiC/SiC fiber bundle composites. $Y_2Si_2O_7$ coating were fabricated by sol-gel wet method and ZrO_2 coating were chemical vapor deposition method. $Y_2Si_2O_7$ coating by wet process could not perform as a weak interface, however, ZrO_2 coating via CVD performed well as a weak interface in SiC/SiC. Fiber pull-out behavior were also observed by SEM observation of fractured surface of the SiC/SiC fiber bundle composites.

(ICACC-S1-P072-2018) Fabrication and properties of functionally graded materials obtained by centrifugal slip casting using a magnetic field method

J. M. Zygmuntowicz*[1]; K. Konopka[1]

1. Warsaw University of Technology, Faculty of Materials Science and Engineering, Poland

Functionally graded Al_2O_3/Ni composites were obtained by an innovative centrifugal slip casting using a magnetic field technique. This method combines the consolidation of powders by the centrifugal force with the simultaneous action of the magnetic field. The combination of the centrifugal slip casting whit magnetic field permits fabrication ceramic-metal composites with a graded distribution of metal particles in a ceramic matrix. The composites made by this method have a shape of hollow cylinders. Functionally graded composites were characterized by XRD, SEM, EDS. The hardness was measured by using a Vickers hardness-testing. Based on hardness measurements K_{IC} value were determined. Microstructural characteristics were examined from outer surface towards the inner side of the tube. The results revealed that the outer surface of Al_2O_3/Ni composites consisted of the maximum volume fraction of metal particles in contrast to the inner part of the tube which was devoid of the Ni particles. The X-ray analysis showed no reflections other than to Al_2O_3 and Ni. It was found that hardness were decreased with increasing of Ni content, as opposed to fracture toughness which increases with the increasing of metal content in composites. The project has been financially supported by National Science Centre (NCN), Poland (Agreement no. UMO-2016/23/N/ST8/00234).

(ICACC-S1-P073-2018) International Standards for Properties and Performance of Advanced Ceramics – Entering a Fourth Decade of High-Quality, Rigorous ASTM Standards

M. G. Jenkins*[1]; J. Salem[2]; G. D. Quinn[3]; J. Helfinstine[4]; S. T. Gonczy[5]

1. Bothell Engineering and Science Technologies, USA
2. NASA Glenn Research Center, USA
3. NIST, USA
4. Corning Incorporated, USA
5. Gateway Materials Technology, USA

Mechanical and physical properties/performance of advanced ceramics and glasses are challenging to measure accurately and precisely unless the proper techniques are used. Now entering a fourth decade of effort, ASTM Committee C28 on Advanced Ceramics, has developed full-consensus standards (e.g., test methods, practices, guides, terminology) to measure properties/performance of coatings as well as monolithic and composite ceramics that may be applicable to some glasses. These standards provide big and little picture details for determining many mechanical, physical, and thermal properties and performance, as well as characteristics for processing, thereby providing accurate, reliable, repeatable and complete data. Users, producers, researchers, designers, and academicians who are involved in ASTM Committee C28 write, continually update, and validate through round robin test programmes the over 50 standards under the jurisdiction of the Committee that it hss developed since its inception in 1986. Included is a pictogram of the ASTM Committee C28 standards and how to obtain them either individually or as a complete collection in one volume. Also included is a listing of other relevant ASTM committees. Finally, some examples of the tangible benefits of standards for advanced ceramics demonstrate their practical application are provided.

(ICACC-S1-P074-2018) HfB_2, ZrB_2 and $Hf_{0.5}Zr_{0.5}B_2$ Solid Solution UTHC Ceramics: Processing by SPS and Mechanical Properties

A. Carrasco-Pena*[1]

1. University of Central Florida, Mechanical and Aerospace Engineering, USA

Mechanical properties of Spark Plasma Sintered (SPS) HfB_2, ZrB_2 and their $Hf_{0.5}Zr_{0.5}B_2$ solid solution were studied both at room and high temperatures. For SPS, 300g of pure HfB_2 and 300g of pure ZrB_2 powders were milled in a planetary ball mill for 6 hours. To make 50/50 Hf-Zr-B solid solution, 150g of pure HfB_2 and 150g of pure ZrB_2 were also ball milled together for 6 hours. WC 250ml lined vial and 5-10mm in diameter SiC balls were used for milling in the ethanol as a liquid media. Pure HfB_2 and ZrB_2 samples were sintered at 2100°C, while $Hf_{0.5}Zr_{0.5}B_2$ solid solution was densified at 5 different temperatures between 1900°C to 2300°C in order to determine the optimal temperature for its densification. After sintering of 21mm in diameter and 6mm in thickness pellets, the samples were machined into 20mm in diameter and 2mm in thickness disks, which were further used for the study at room and high temperatures of the properties by Resonant Ultrasound Spectroscopy. The Young's, shear and bulk moduli, along with Poisson's ratio were measured up to 1000°C. At the same time, an attempt is being made to measure the biaxial strength of these ceramics using ring-on-ring (RoR) biaxial strength testing technique. In order to accomplish the measurement, the RoR testing jig is currently being designed nad manufactured.

(ICACC-S1-P075-2018) Effect of carbon nanotubes as fillers on the interfacial bonding in kenaf-polypropylene Composites

R. Paskaramoorthy*[1]

1. University of the Witwatersrand, Mechanical Engineering, South Africa

The use of plant-based natural fibres, such as kenaf, sisal, hemp, flax, etc., as the reinforcement in polymer composites has been growing for the past few decades. In this study, the effects of carbon nanotubes (CNT) in improving the interfacial bonding in kenaf reinforced polypropylene composite are presented. Composite laminates were made using 30% by mass of kenaf fiber and 0.25%, 0.50%, 0.75% and 1.0% by mass of functionalized CNT. Nonwoven kenaf fiber mats were first treated with NaOH solution of 6% concentration. After this, the fibres were immersed in solutions of 5% by fibre weight of 3-aminopropyltriethoxysilane in a 50% aqueous solution of methanol. Composite laminates were then made by compression moulding. Tensile and flexural strengths of composites made with CNTs were found to be better than those made without the CNTs. The laminates made with 0.5% CNT showed the highest improvement. SEM examinations of fractured surface indicated good wetting and comparably fewer instances of fiber pull-out, indicating improvement of fiber-matrix interfacial bonding. Closer examination also revealed the presence of CNTs in the interfacial region which possibly acted as a bridge between the fibers and matrix and contributed to improved mechanical properties.

(ICACC-S1-P076-2018) Effect of melt temperature and addition time on the mechanical properties of in-situ Al/TiB_2 composites

R. Paskaramoorthy*[1]; B. Prabu[2]; A. Vivekananda[2]

1. University of the Witwatersrand, Mechanical Engineering, South Africa
2. Anna University, Mechanical Engineering, India

In recent years, TiB2 reinforced Al metal matrix composite has been used in various structural and wear resistance applications. This paper reports the combined influence of melt temperature and addition time, on the formation and distribution of TiB2 particles within Al/TiB2 composites and their influence on the mechanical properties. Even though addition time is an important parameter, its effect has never been reported in the literature. The TiB2 particles were formed by the in-situ reaction of potassium hexafluoro titanate K_2TiF_6 and potassium tetrafluoroborate (KBF4) with molten aluminium. The formation of TiB2 was confirmed by XRD examination. The microstructure and size of the TiB2 particles formed are found to be strongly affected by both melt temperature and addition time. The microstructures displayed significantly different degrees of particle agglomeration. Hardness and ultimate tensile strength (UTS) are strongly affected by both melt temperature and addition time. The average hardness and UTS of the composites are both higher than those of parent aluminium. In particular, the increase in the maximum average hardness and UTS of the composite are 46% and 53%, respectively. SEM image of fracture surface of the composite shows characteristics of brittle fracture. Results clearly indicate that addition time is an important parameter.

(ICACC-S1-P077-2018) Manufacturing of Structural Regolith Parts for Lunar Colonies

K. D. Grossman*[1]; T. C. Sakthivel[1]; S. Seal[1]

1. University of Central Florida, Materials Science and Engineering, USA

In-situ resource utilization is crucial for the success of future manned space missions. Relying on local resources for biological needs as well as construction material for building colonies drastically decreases launch masses of vehicles which makes space exploration economically viable. One of the most abundant resources on the moon and Mars is the fine powder of crushed rocks known as regolith that covers the entire body. Being composed primarily of metal-oxides, the regolith on the moon has many uses beyond any un-bound water it may contain. One use is as construction material after being sintered into solid pieces. This study looks at the sintering mechanics and properties of lunar regolith simulant JSC-1A under a variety of different heating conditions. JSC-1A powder was first sieved into distinct particle size ranges and then subjected to a variety of heating profiles ranging from 1150 degrees C up to 1400 degrees. The heating profiles yielded varying phases of solidified regolith blocks which were measured for compressive strength, hardness as well as crystalline phase changes and elemental migration between particles within a sintered block.

(ICACC-S1-P078-2018) Synthesis and characterization of Al_2O_3-ZrO_2-TiC composite by spark plasma sintering

Y. Zhu*[1]

1. Institute of Modern Physics, Chinese Academy of Sciences, China

Al_2O_3–TiC composite, which possesses attractive mechanical properties and excellent wear resistance, has been widely used as cutting tools and wear resistance coating. However, as a hard brittle ceramic, Al_2O_3–TiC composite has its drawback of low fracture toughness. In this work, study have been carried out by adding ZrO_2 in order to further improve its strength and toughness. Al_2O_3-ZrO_2-TiC composite powders were prepared by wet-planetary ball milling and subsequently sintered by Spark Plasma Sintering. The effect of the fabrication parameters on the microstructure and properties of Al_2O_3-ZrO_2-TiC composites was investigated. The results showed that Al_2O_3-ZrO_2-TiC composites with fine and homogeneous microstructures, which had excellent comprehensive properties: Vickers hardness of 15.9 GPa, fracture toughness of 6.26 $MPa \bullet m^{1/2}$, and thermal conductivity of 14.36 W/mK. The relationship between microstructure and properties and the strengthening and toughening mechanisms was discussed.

(ICACC-S1-P079-2018) Antibacterial PMMA Bone Cement Composites Reinforced by Silver(Ag)-doped Hydroxyapatite Nanobelts and TiO_2 nanotubes with enhanced mechanical property

M. Qi*[1]; M. Rezazadeh Shirdar[2]; A. Phakatkar[2]; R. Shahbazian-Yassar[3]; Y. Lu[1]; T. Shokuhfar[2]

1. Shandong University, School of Materials Science and Engineering, China
2. University of Illinois at Chicago, Bioengineering Department, USA
3. University of Illinois at Chicago, Department of Mechanical & Industrial Engineering, USA

This study proposes Ag-doped Hydroxyapatite(Ag-HA) nanobelts and Titanium nanotubes (TNTs) as novel fillers with superior mechanical and antibacterial properties in poly-methyl- methacrylate (PMMA) bone cement composite. Both Ag-HA nanobelts and individual TNTs were synthesized using hydrothermal method. The obtained filler materials (Ag-HA nanobelts and TNTs) were characterized using X-ray diffraction (XRD), Transmission Electron Microscopy (TEM) and Energy-dispersive X-ray spectroscopy (EDX) and then mixed with PMMA polymer matrix to develop PMMA/Ag-HA nanobelts/TNTs composites in various proportions. The mechanical properties of the composites were evaluated using microhardness and compression tests. Antibacterial test was performed with Escherichia coli bacteria to check the influence of filler materials on inhibition zones. Additionally, MTT assay was performed to assess the cytotoxicity of filler materials. Results indicate that incorporation of Ag-HA nanobelts and TNTs filler materials has greatly enhanced the mechanical properties of PMMA composite. The presence of Ag-doped HA nanobelts has improved antibacterial properties of PMMA bone cement. These results indicate that PMMA/Ag-HA nanobelts/TNTs composite has tremendous potential to alter the state of the art bone cements.

(ICACC-S1-P080-2018) Joining of oxide/oxide (Nextel™ 610/alumina-zirconia) ceramic composites

M. Akram*[1]; V. Casalegno[1]; M. Ferraris[1]; G. Puchas[2]; S. Knohl[2]; W. Krenkel[2]

1. Politecnico di Torino, Department of Applied Science and Technology, Italy
2. University of Bayreuth, Department of Ceramic Materials Engineering, Germany

Nextel™ 610/alumina-zirconia oxide fiber composites were successfully joined to themselves and to metals by using brazing alloys and glass-ceramics. Scanning electron microscopy (SEM) showed that the joint interfaces were continuous and free of defects. Energy dispersive X-ray spectroscopy (EDS) was used to analyze the elemental composition and X-ray diffraction (XRD) was used to investigate the formation of different phases in the glass-ceramic. Lap off-set shear tests were performed at room temperature to check the mechanical strength of the joints. Thermal ageing in simulated working conditions showed the suitability of the proposed joining materials.

(ICACC-S1-P081-2018) Determination of elastic modulus of different joining materials using micro/nano-indentation directly in the joints

S. De La Pierre*[1]; C. Balagna[1]; P. Tatarko[2]; M. Fides[3]; D. Nemeth[3]; M. Ferraris[1]

1. Politecnico di Torino, DISAT, Italy
2. Institute of Inorganic Chemistry, Slovak Academy of Sciences, Slovakia
3. Institute of Materials Research, Slovak Academy of Sciences, Slovakia

The elastic modulus values of joining materials are important for the design of joined components in a broad range of applications. Measuring these values only on bulk joining material itself could be misleading because interactions with substrates are not taken into account. In this study different joining materials, such as glass-ceramics and polymeric adhesives, were selected to join ceramic, composite and metallic substrates depending on the application of the final component. The elastic modulus of these joined materials was measured using micro/nano-indentation technique; tests were performed both on the substrates and directly inside the joined area. The results for materials with brittle and elasto-plastic behavior were compared to understand if the indentation technique could be applied to all material classes. Then the indentation elastic modulus results were compared to the elastic modulus determined separately on reference bulk samples of joining materials (not joined) using a standard impulse excitation technique.

(ICACC-S1-P082-2018) "Re-wrap" method (Refractory metals Re=Mo, Nb Ta) for joining of ceramics and CMC

P. Gianchandani*[1]; V. Casalegno[1]; M. Salvo[1]; M. Ferraris[1]

1. Politecnico di Torino, DISAT-Department of Applied Science and Technology, Italy

The recently developed Mo-wrap method has been successfully extended to obtain a "Re-wrap" method (Refractory metals Re=Mo, Nb Ta) for joining of Ceramics (SiC, Mullite, etc.) and Ceramic matrix composites (CMC) (C/SiC, SiC/SiC, etc.) to be used in high temperature aerospace and energy applications. Refractory metals foils (Mo, Nb, Ta) have been used to wrap one or more silicon foils. Refractory metal silicides (Re-Si2) are promising materials for high temperature applications, due to their high thermal stability (more than 2000 °C), oxidation residence at elevated temperature, brittle-to-ductile transition and electrical conductivity. The "Re-wrap" technique is based on a composite joining material obtained by in-situ reaction of silicon and refractory metals (Mo, Nb and Ta) at 1450 °C in Ar flow, to give refractory metal silicides embedded in a Si matrix. This study presents the newly developed joints, C/SiC-"Re-Wrap"- C/SiC, their morphology, mechanical strength and oxidation resistance. Acknowledgment: The authors gratefully acknowledges and gives heartfelt thanks to the AIRBUS and MTAerospace for providing the ceramics matrix composites (CMC) for this research activity.

(ICACC-S1-P083-2018) Thermal shock performance of Sandwich structures obtained by "Mo-Wrap"

P. Gianchandani*[1]; V. Casalegno[1]; M. Salvo[1]; M. Ferraris[1]; G. Bianchi[2]; A. Ortona[2]

1. Politecnico di Torino, DISAT-Department of Applied Science and Technology, Italy
2. MEMTi Institute, The University of Applied Sciences and Arts of Southern Switzerland, Switzerland

SiC foams sandwiched between two ceramic matrix composites (CMC) skins are of interest for several high temperature applications ranging from aeronautic to energy production. A novel joining technique and material "Mo-Wrap" is used to join highly porous

SiC foams to C/SiC composites avoiding detrimental infiltration. Compression and thermal shock tests demonstrated the soundness of joint. Three cycles of thermal shock resistance tests from room temperature up to 1100 °C in air were done on the sandwich structures. It is worth noting that neither the interface Mo-Wrap/foam, nor the Mo-wrap itself changed morphology and composition (e.g. oxidation). Acknowledgment: The authors gratefully acknowledges and gives heartfelt thanks to the MTAerospace for providing the ceramics matrix composites (CMC) for this research activity.

(ICACC-S1-P084-2018) Joint Strength Improvement of C_f-SiC/Ti6Al4V System by Surface Modification

M. Bangash*[1]; A. Das[2]; V. Casalegno[1]; M. Ferraris[1]

1. Politecnico di Torino, DISAT-Department of Applied Science and Technology, Italy
2. Indian Institute of Technology (ISM), Department of Mechanical Engineering, India

Joining of dissimilar materials such as low density C_f-SiC composites to high strength Ti6Al4V to produce hybrid structures able to satisfy thermal and mechanical requirements is valuable for applications in aerospace industry. In the recent past, several joining processes have been proposed to securely join such materials. However, the improvement of joint strength by surface modification is quite new. This study focuses on the design, characterization and testing of metal-composite bonding and joint strength improvement by surface modification of the metal substrate. V-Shaped micro slots are produced on Ti6Al4V alloy surface by using in-house built Micro- Electrical Discharge Machining (EDM). C_f-SiC is joined to both plain and surface modified Ti6Al4V by pressure-less brazing process, using TiB-590 (Zr-21Ti-16Ni-1Hf wt. %) braze at 900 ºC for 10 minutes in Argon atmosphere. The joint properties are characterised by Scanning Electron Microscope (SEM) and X-Ray Diffraction (XRD). Single-Lap Offset (SLO) test results showed significant improvement in the joint strength.

(ICACC-S1-P134-2018) Grain Growth Control of Centrifugally Spun Alumina Fibers by Magnesia and Zirconia Additions

T. Natarajan*[1]; P. Bhargava[1]

1. Indian Institute of Technology Bombay, Metallurgical Engineering and Materials Science, India

In the present work we report the synthesis of 10-15µm dia. Al_2O_3 fibers using sol-gel with centrifugal spinning. Fiber index was around 96%. SEM image of fibers calcined showed the presence of vermicular grains and porosity. Sintered fibers had an avg. grain size of 4µm which is excessively large for avg. fiber dia.of around 10-15µm. Grain growth is undesirable as it degrades mechanical properties. In the current work grain growth control of Al_2O_3 fibers has been achieved by addition of MgO and 3YSZ. It was inferred from SEM image and elemental mapping respectively that these fibers are devoid of abnormal grains and they showed homogeneous distribution of MgO/3YSZ. MgO inhibited grain growth due its partial solubility and forming spinel while 3YSZ controlled by triple junction pinning effect which is in good agreement with the mechanisms reported earlier. Grain size measurements showed that without additives the avg. grain size of Al_2O_3 fibers was around 3.7+1.5µm which got reduced to 1.6+0.7,1.9+1.1µm respectively when 0.25 and 2wt. MgO% were added. Maximum reduction of ~62% was achieved with 3YSZ addition. XRD of all samples showed corundum as primary phase.2 wt.% MgO added fibers showed low intense spinel peaks whereas Al_2O_3 with 20 wt.% YSZ showed the presence of tetragonal and monoclinic phases. Besides, few hollow fibers were also seen the reasons for which are yet to be understood.

(ICACC-S5-P086-2018) Analytical evaluation of orientation behavior of β-tricalcium phosphate / poly(lactic acid) composite billets in extrusion drawing

M. Sakaguchi*[1]; S. Kobayashi[2]

1. Salesian Polytechnic, Mechanical and Electronic Engineering, Japan
2. Tokyo Metropolitan University, Japan

Bioabsorbable poly(lactic acid) (PLA) attracts much attention as a material for bone fixation device. However, the material has drawbacks, such as lower elastic modulus and no osteoconductivity. Therefore, composite material of PLA and bioactive ceramics powder with higher stiffness and osteoconductivity has been investigated. A composite device with hydroxyapatite(HA) which is included in autologous bone has been developed. However, degradation rate of HA/PLA composite is too low. It was reported that this composite remained for 5 years in vivo. In addition, the strength for composite material of polymers and ceramics is decreased by stress concentration around ceramic particles. In this study, β-tricalcium phosphate (β-TCP) was compounded to PLA to fabricate β-TCP/PLA composite and this composite was drawn to improve the strength. Past studies about self-reinforcing by drawing have been conducted on polymer single body. In this study, optimum molding conditions are investigated to improve mechanical properties of β-TCP/PLA composites. Deformation behavior of β-TCP/PLA rods during drawing was simulated through finite element calculation, and orientation of PLA molecular chains was determined using pseudo-affine model from the strain of drawn rods. Those analysis values are compared with orientation functions which are measured in drawn rods.

(ICACC-S5-P087-2018) Characterization and synthesis of silver contain silica nanocomposites particles via a facile impregnation method as an antibacterial agents against Escherichia coli and Bacillus subtilis bacteria

D. Bae*[1]

1. Changwon National University, Republic of Korea

The SiO_2@Ag nanocomposite spheres were synthesized by a facile impregnation method. The Ag NPs decoration is deposited onto the SiO_2 templates employing silver nitrate and trisodium citrate as a reducing agent, resulting in SiO_2@Ag nanocomposites. As-prepared samples were characterized by XRD, FE-SEM, FE-TEM analysis. According to the morphological study, the average particle size of deposited Ag NPs and SiO_2 spheres was approximately 5-15 and 100-130 nm, respectively. The bactericidal study was also performed using minimum inhibitory test (MIC) and disk diffusion test against Gram-negative as Escherichia coli and Gram-positive as Bacillus Subtilis bacteria. The obtained NP's have fully restricted the bacterial growth of E. coli bacteria as increasing the content of NP's up to 1.00 mg/ml. We have applied steel and polyethylene (PE) substrate as a substrate for coating of SiO_2@Ag nanocomposite to investigate antibacterial effectiveness in disk diffusion test. The SiO_2@Ag coated steel and PE had shown an outstanding result with inhibition zones among 17-20 mm either E. coli and B. subtilis.

(ICACC-S7-P088-2018) Fabrication and Characterization of Potassium Titanate Whiskers by a Hydrothermal Processing

D. Bae*[1]

1. Changwon National University, Republic of Korea

$K_2Ti_6O_{13}$ whisker were prepared under mild temperature and pressure conditions by precipitation from metal nitrates with aqueous ammonium hydroxide. $K_2Ti_6O_{13}$ whisker were obtained with reaction at the temperature range of 210°C to 250°C for various reaction time. The average length size of the synthesized $K_2Ti_6O_{13}$ whisker was in the range of 100 nm to 1500nm. The average diameter of the synthesized $K_2Ti_6O_{13}$ whisker was in the range of 10nm to 60nm. The XRD pattern showed that the synthesized $K_2Ti_6O_{13}$ whisker were crystalline. The effects of synthesis parameters, such as the flux, pH of starting solution, reaction temperature and time, are discussed.

(ICACC-S7-P089-2018) Size Control of Magnesium Submicron Particles Prepared by Pulsed Wire Discharge

H. D. Nguyen*[1]; Y. Tokoi[2]; K. Tanaka[1]; T. Sasaki[3]; T. Suzuki[4]; T. Nakayama[1]; H. Suematsu[1]; K. Niihara[4]

1. Nagaoka University of Technology, Extreme Energy-Density Research Institute, Japan
2. Nagaoka National College of Technology, Department of Electrical and Electronics Systems Engineering, Japan
3. Nagaoka University of Technology, Nagaoka, Department of Electrical, Electronics and Information Engineering, Japan
4. Nagaoka University of Technology, Nagaoka, Department of Nuclear System Safety Engineering, Japan

Pulsed wire discharge (PWD) is known to be one of many nanoparticle preparation methods as a low-cost and high production rate method. In the previous study, by the PWD method, nanoparticles of Cu were reported to be successfully prepared and particle size was controlled by gas pressure and input energy. In addition, submicron particles of Mg was also reported to be successfully prepared by PWD. However, the ability of controlling particle size of Mg submicron size particles by PWD has not been investigated. In this study, Mg submicron-sized particles were prepared in various conditions. Mean diameter of prepared particles was obtained from transmission electron microscopy observation results. The mean particle diameter was decreased with increasing the relative energy (K), which was defined as the charged energy divided by the evaporation energy of wire. Using this method, Mg particles with a geometric mean diameter of 41.9 nm were prepared. Within the author's knowledge, they are the smallest passivated Mg particles prepared by any methods. An equation for estimating mean diameter was proposed. The validity of the equation was shown by comparing estimated and measured mean diameter.

(ICACC-S7-P090-2018) Simple preparation method of Mg–Al and Mg–Al–Ti hydrotalcites as base catalyst

E. d. Magdaluyo*[1]; G. Magayanes[1]

1. University of the Philippines, Philippines

We report a simple preparation method for the Mg–Al hydrotalcite as base catalysts via the hydration of their corresponding aqueous metal nitrates. Moreover, the Mg–Al–Ti hydrotalcite was also synthesized upon addition of the TiO_2 in the Mg-Al nitrate system. The reaction pathways in the formation of the Mg–Al and Mg–Al–Ti hydrotalcites were proposed. The Mg–Al–Ti hydrotalcites were formed by the reaction of Mg^{2+}, $Al(OH)^{4-}$ and Ti^{4+}, which resulted from the hydration of the metal precursors. The prepared Mg–Al and Mg-Al-Ti retained their unique structural properties, including their layered hydroxide structure and memory effect and showed considerable catalytic activity in the conversion of vegetable oil in biodiesel.

(ICACC-S7-P091-2018) A Highly Sensitive Nonenzymatic Sensor Based on Fe_2O_3 Nanoparticles Coated ZnO Nanorods for Electrochemical Detection of Nitrite

R. Ahmad*[1]; M. Ahn[1]; Y. Hahn[1]

1. Chonbuk National University, School of Semiconductor & Chemical Engineering, Republic of Korea

Monitoring of nitrite is needed for the management of nitrite contamination and to ensure the welfare of environment and human health. Hence, it is important to develop high performance sensors that precisely measure nitrite concentration. Herein, we demonstrate fabrication of a highly sensitive nonenzymatic nitrite sensor based on Fe_2O_3 nanoparticles coated ZnO nanorods (Fe_2O_3 NPs coated ZnO NRs). The ZnO NRs were grown on seeded silver electrode by hydrothermal method and then coated under optimized conditions with Fe_2O_3 NPs using dip-coating method. Detailed material characterizations and sensor fabrication process are reported. The electrochemical properties of fabricated nonenzymatic nitrite sensor were tested with different concentrations of nitrite, which indicate that the coating of ZnO NRs with Fe_2O_3 NPs significantly enhanced the electrocatalytic activity. The sensor responded linearly with increasing concentration of nitrite from 1 μM to 1250 μM with a high sensitivity (131.2 $\mu A \mu M^{-1} cm^{-2}$) and low detection limit (0.015 μM). Moreover, excellent selectivity, stability, and reproducibility allowed using these sensors for the determination of nitrite concentration in water with satisfactory results.

(ICACC-S7-P092-2018) Synthesis of Manganese Oxide Nanostructure and Its Application for Potassium Ion Sensing in Water

M. Ahn*[1]; R. Ahmad[1]; Y. Hahn[1]

1. Chonbuk National University, School of Semiconductor & Chemical Engineering, Republic of Korea

The potassium (K^+) ion is very important mineral which controls the cellular and electrical functions in the body. The change in the concentration of potassium ion in human serum cause the risk of acute cardiac arrhythmia. Hence, it is important to monitor the potassium level in drinking water/food to control the intake and prevent its effect. Herein, we synthesized manganese oxide (MnO_2) nanostructure using low-temperature solution process and characterized in details with X-ray diffraction (XRD), field-emission scanning electron microscopy (FESEM), transmission electron microscopy (TEM), etc. We used as-synthesized MnO2 nanostructure to fabricate potassium ion sensor after modifying the glassy carbon electrode with the help of conductive binder. The fabricated sensor showed good sensitivity and selectivity during potassium ion sensing. Moreover, we also measured reproducibility, long term stability, and applicability for potassium ion detection in water samples.

(ICACC-S7-P093-2018) Adsorption and photocatalytic removal of MB dyes by WO_3 nanorods

S. Ryu[1]; C. Nam*[1]

1. Hannam University, Photonics and Sensors, Republic of Korea

Tungsten oxides have attracted much interest not only due to their fundamental scientific issues but also their various technological applications such as environment-, energy- and electronic- materials. Among them, dye-adsorption and photo-catalyst properties of WO_3 nanomaterials have been independently studied due to their large surface area and low-band gap. In this presentation, WO_3 nanorods have been synthesized by hydrothermal methods at various temperatures using sodium tungstate ($Na_2WO_4 2H_2O$) as a precursor material. In order to obtain one-dimensional nanostructures, we have added a directional capping agent of citric acid during the preparation of solution. The morphology and structure of synthesized WO_3 samples were characterized by scanning electron microscopy, x-ray diffraction technique, transmission electron microscopy, and BET methods for surface area measurements. Dye adsorption properties were investigated by using a series of MB solutions with a specific amount of WO_3 powders in a dark room, where duration time was changed to monitor adsorption capacities. In addition, pH-dependences of adsorption were studied to understand relations between the surface charges of WO_3 powders and the cationic dye. Finally, photocatalytic properties were examined by simple UV-absorption methods depending on UV exposure times after saturated adsorption time of MB dyes on the WO_3 surfaces.

(ICACC-S7-P094-2018) Silicon Nanostructures for Hydrogen Evolution Electrode of Photoeletrochemical Cells

D. Lee*[1]

1. Korea Institute of Industrial Technology, Green Materials & Processes Group, Republic of Korea

The production of fuels from solar energy has been considered one of the main challenges in the development of a sustainable energy system fulfilling both of minimal environmental impact and clean energy. Photoelectrochemical water splitting system offers the

capability of efficiently harvesting the solar energy and transferring it directly to the chemical form of hydrogen for easy storage, transport and use. The main challenge in developing Si based photocathode material is acquiring high light absorption and charge carrier collection, simultaneously. Here, we fabricated wafer-scale ultra-high aspect ratio Si nanomesh and Si nanowire array with the combination of block copolymer lithography and metal assisted etching. Block copolymer lithography is an emerging approach providing a variety of periodic nanoscale morphologies having feature sizes ranging from 5 to 50 nm. Metal assisted Si etching method gained increasing attention and various approaches derived to fabricate Si-based nanostructures. These Si nanomaterials with low feature size and high aspect ratio increase the optical absorptivity and the photocurrents compared to the bulk countpart.

(ICACC-S7-P095-2018) Mixed-Ligand Metal Arylalkenolates as New Family of Air Stable Molecular Precursors for the Atomic Layer Deposition of Iridium-based Catalytic Coatings

L. Jürgensen*[1]; M. Frank[1]; T. Fischer[1]; S. Mathur[1]

1. University of Cologne, Institute of Inorganic Chemistry, Germany

Investigation of the interplay of metal-organic chemistry will enrich the state-of-the-art of ALD technology and open new possibilities for the applications of new Ir-based materials. Therefore heteroleptic mixed-ligand complexes exhibiting manifested Janus-type reactivity are under investigation to elaborate the precursor chemistry – materials synthesis – functional property chain. Physico-chemical studies (thermal degradation behavior, volatility), the atomic layer deposition studies have been carried out to elucidate the adsorption behavior (sticking coefficient) of new molecules. Finally, the ALD-grown materials were tested towards their (electro)catalytic applications, particular in the oxygen evolution reactions. In this work synthetic preparation and structural characterization of precursors will be reported and their thermal decomposition behavior is examined. Thin films grown by ALD have been investigated towards their compositional and structural properties as well as their catalytic behavior in oxygen evolution reactions.

(ICACC-S7-P096-2018) Hollowed-out SiC nanofibers supported Pt nanoparticles as high-temperature NH_3 sensor

Y. Wang*[1]

1. National University of Defense Technology, China

Silicon carbide (SiC) is an attractive candidate as catalyst supports applied in harsh environments, owing to its chemical inertness, excellent electrical and thermal performance. β-SiC, not only possesses high electron mobility and saturation drift velocity, but also can be prepared in large scale. Three-dimensional (3D) interconnected structure and large surface area of the sensor materials contribute positively to the high sensitivity and fast response/recovery time. Electrospun nanomaterials with interconnected networks display excellent sensing performance compared with the widely used solid flat film. Herein, hollowed-out SiC nanofibers (HSiC) with high surface area and hierarchical nanostructures were fabricated through electrospinning of polycarbosilane and followed catalysis-assistance pyrolysis route. The SiC nanofibers are uniform with the average diameter of 500 nm, which are constructed of nanorods with the diameter of about 30 nm. Homogeneous platinum nanoparticles with size of 2-3 nm were deposited on the surface of SiC using an ethylene glycol reduction method. The obtained Pt/HSiC was applied as high-temperature NH_3 sensor materials, demonstrating a sensitivity of 9.2% for 500 ppm NH_3 and excellent repeatability at 500 °C.

(ICACC-S7-P097-2018) Fabrication of hybrid carbon film and their application for the oxygen reduction electrode of PEMFC

D. Lee*[1]

1. Korea Institute of Industrial Technology, Green Materials & Processes Group, Republic of Korea

Carbon nanotubes (CNTs) have been interested in various area such as electronics, photonics, energy devices, and other applications. However, it has been a long-standing challenge to establish a straightforward process for the production of uniform CNTs with desired structures and properties. Here, we have fabricated hybrid carbon films which are composed of carbon nanotube arrays grown on reduced graphene oxide films. The processes enabled the formation of diameter, wall-number, and atomic structure controlled carbon nanotube arrays. This method enabled to fabricate the Fe-porphyrinic CNTs, which are outstanding oxygen reduction catalysts via the efficient 4-electron oxygen reduction process. The cyclic and the rotating disk electrode (RDE) voltammograms confirmed the outstanding oxygen reduction reaction (ORR) properties of the Fe-porphyrinic CNT. The fabrication processes of the efficient, bio-mimetic, rigid, electron-conducting carbon nanotube catalysts can have a significant impact on the wide deployment of the current PEMFC technology.

(ICACC-S8-P098-2018) Effect of Fabrication Condition on Mechanical Properties of Hydroxyapatite

S. Kobayashi*[1]; T. Izawa[1]

1. Tokyo Metropolitan University, Mechanical Engineering, Japan

In this study, effect of fabrication condition, such as sintering temperature and time, on mechanical properties of hydroxiapatite (HA) ceramics was investigated. Relarive density and grain size increased with increasing sintering temperature and time. With cold isostatic pressing, higher relative density is obtained without grain growth. It was also confirmed that bending strength and fracture toughness follows the Hall-Petch relation.

(ICACC-S9-P099-2018) Development of fabrication procedure of porous carbon material with three dimensionally networked structure

Y. Kaneda*[1]; R. Inoue[1]; Y. Kogo[1]

1. Tokyo University of Science, Materials Science & Technology, Japan

In this study, porous carbon material was derived from phenolic resin mixture by polymerization induced phase separation based on the spinodal decomposition. We prepared phenolic-based monolith based on benzenenesulfonyl chloride, sodium carbonate, and ethylene glycol. The effect of composition of resin mixture, curing time and viscosity of phenolic resin on the pore structure of phenol and carbon material has been investigated. Phenol and carbon material were different morphologies obtained by changing composition of resin mixture, curing time and viscosity of phenolic resin. Characterization of their morphologies was also performed using scanning electron microscopy (SEM), mercury intrusion porosimetry (MIP), and X ray diffractrometry (XRD), respectively. Characterization of mechanical performance was also done. The relation between microstructure characteristics and mechanical properties of developed material will be discussed.

(ICACC-S9-P100-2018) Novel polymer derived silicon nitride nanofibers felt

E. Zera*[1]; P. Jana[1]; G. D. Sorarù[1]

1. University of Trento, Industrial Engineering, Italy

The present study describes a simple and cost effective way of producing polymer derived felts consisting of silicon nitride nanobelts. The novelty of the process lies in shaping a polysiloxane with the help a porous preform and converting the body into the corresponding shaped silicon nitride felt through a pyrolysis process in a reactive atmosphere. SEM investigation indicates that the Si_3N_4

nanobelts have a very high aspect ratio, being ca. 300 nm wide and several microns long. The process allows varying the density of the felt in the range 30-120 kg/m^3 with a corresponding compressive strength between 24 and 150 kPa, and 10-20% of elastic recovery. Potential application in the field of thermal insulation and electromagnetic wave absorption will also be presented.

(ICACC-S9-P101-2018) Fabrication and strengthening of porous Si_3N_4 ceramics by replacement of oxide phase with Si_3N_4 at grain boundary through carbothermal nitridation

J. Yang*[1]; Z. Xu[1]; Q. Zhi[1]

1. Xi'an Jiaotong University, China

Porous silicon nitride ceramics are attracting extensive attentions due to its high strength and low dielectric loss. However, further strength enhancement at elevated temperatures is hindered by its intergranular phase, forming from sintering additive. This paper describes the fabrication of porous silicon nitride ceramic materials, by using a replacement method of carbothermal nitridation, The initial samples were obtained from sintering of mixed powder consisted of 95wt.% Si_3N_4 and 5 wt.% Y_2O_3. After the removal of the oxide intergranular phase and the infiltration of mixtures of phenolic resin and silica sol, carbothermal nitridation process was carried out at 1550°C for 2 h under nitrogen. XRD and microstructural analysis revealed a complete replacement of oxide intergranular phase by the newly formed Si_3N_4 intergranular phase. The unmodified ceramic exhibited lower flexural strength at 1400°C, which was only 50% of room-temperature strength. Although the modified ceramic attained a slightly lower flexural strength at room temperature after the replacement of intergranular phase, its strength measured at 1400°C could attain 90% of room-temperature strength.

(ICACC-S9-P102-2018) Study on microstructures and properties of porous titanium carbide ceramics fabricated by reaction sintering process

Y. Ma*[1]

1. Xi'an Jiaotong University, Materials Science and Engineering, China

Porous ceramics contain high volume fractions of porosity and possess attractive properties, such as low weight, high specific surface area, high permeability and high adsorption. Therefore, they have been applied in industrial and engineering fields, including as fuel cell electrodes, filters, absorbers, catalyst carriers, surgical implants, reactors etc. Titanium carbide (TiC) is a typically transition metal carbide. Due to coexistence of the ionic bond, covalent bond and metal bond, TiC possesses characteristics of high Vickers hardness, high melting point, high wear resistance and electrical conductivity simultaneously. The good wet ability by metal melts, electrical conductivity and corrosion resistance in phosphoric acid made the porous TiC ceramic the most promising candidates in the aspect of specific applications. Porous TiC ceramics were fabricated by the reaction sintering process, in which TiO_2, carbon black and phenolic resin were used as the reactants. The results show that the porous TiC ceramics fabricated by reaction sintering process have a three-dimensional network structure. The addition of original TiC powder, the compacting pressure and the sintering temperature will have influence on the microstructure and mechanical properties of fabricated porous TiC ceramics.

(ICACC-S10-P103-2018) Numerical modeling of the 2D crack propagation in carbon-carbon composites

R. Piat*[1]

1. Darmstadt University of Applied Science, Germany

Finite fracture mechanics in combination with the crack propagation criteria and the principle of maximum energy dissipation are used for the development of an algorithm for modeling of the crack propagation in a porous 2D microstructure. Firstly, the proposed algorithm was verified for modeling of the crack propagation in PMMA specimens with circular holes and initial notch. The numerical results were compared to the experimental ones. The sensibility to the porosity reconstruction near the crack tip in the microstructure model on the resulting simulated crack propagation was studied by modeling of the crack path in an idealized material with different distributions of the circular pores. The interaction of the local pores and the remaining porosity and its influence on the crack propagation were also studied. The gained experience was utilized for the modeling of the crack propagation in infiltrated carbon felt using a specific microstructure reconstruction. For this modeling, very important features of this composite such as the local material anisotropy and three dimensionality of the porosity were neglected. The obtained results of simulation suggest that the porosity and the distribution of the pores have a more important influence on crack propagation than all these neglected factors.

(ICACC-S10-P104-2018) Atomic-Scale Structure of Sliding Interface of Diamond-Like Carbon Coating Including a Friction Modifier: A Molecular Dynamics Analysis

M. Saito*[1]; J. Xu[1]; Y. Ootani[1]; N. Ozawa[1]; M. Kubo[1]

1. Tohoku University, Japan

In order to reduce energy consumption of mechanical systems, it is necessary to reduce friction. Thus, many types of lubrication techniques are widely studied. Diamond-like carbon (DLC) coating is solid lubricant that is widely used in various fields including automotive industry, because DLC shows high wear resistance and low friction. It has been shown that MoDTC, a friction modifier used in engine oil, reduces friction between DLC coatings. A tribo-film including MoS_2 layers formed by chemical reactions with friction was suggested to lower the friction. Thus, atomic-scale insight into the tribo-film structure is required to improve the frictional properties. However, the atomic-scale structure is still unknown because in situ observation of the sliding interface is difficult. Thus, we performed molecular dynamics simulation to investigate the atomic-scale structure of the tribo-film. We used reactive force field (ReaxFF) to take into account the chemical reactions. First, we determined the force field parameters for Mo, S, C and H atoms so that the ReaxFF calculation can reproduce the structure of binary compounds. Next, we performed sliding simulation using a DLC/MoS_2/DLC interface model. We will report the structural change in the tribo-film associated with the chemical reaction in the sliding interface in atomic-scale.

(ICACC-S12-P105-2018) Investigation of microstructural evolution and He aggregation behavior in V_2AlC thin films under He ions irradiation

J. Wang*[1]; R. Shu[1]; Y. Dong[1]; T. Shao[1]; Q. Deng[1]; X. Zhou[1]; F. Huang[1]; S. Du[1]; Z. Wang[1]; J. Xue[1]; Y. Wang[1]; Q. Huang[1]

1. Ningbo Institute of Industrial Technology, Chinese Academy of Sciences, China

Behavior of MAX phase films under irradiation conditions are rarely reported in spite of their important potential application as coating materials in accident tolerant fuels (ATFs). Due to the different synthesis procedures, MAX phases in bulk form and in thin-film form exhibit different microstructure features, this results in different irradiation resistant behaviors. To investigate the microstructural evolution of MAX phases films under irradiation conditions is important to get a better understanding of their irradiation resistant properties and to optimize the performance of coating - cladding system in the environment of nuclear reactors. In this study, V_2AlC coating synthesized by PVD method on Zr substrate was irradiated with helium ions and subsequently annealed at 450°C to investigate the helium aggregation behavior. It is found that He bubbles are found bigger and denser along the coating- substrate interface than the He bubbles in V_2AlC coating and Zr substrate, indicating the preferential nucleation and aggregation of He bubbles along the interfaces. However, the aggregation of He bubbles along the coating – substrate interfaces decreases the cohesion of V_2AlC

coating. It is therefore crucial to optimize interfacial structure and reduce its sink strength during the practical application of the fuel coating in ATFs.

(ICACC-S12-P106-2018) Microstructure, Characterization, Thermal and Mechanical Properties of a High Entropy Diboride Ceramic

J. A. Scott*[1]; A. Stanfield[1]; G. Hilmas[1]; W. Fahrenholtz[1]

1. Missouri University of Science & Technology, Materials Science and Engineering, USA

A diboride based ceramic containing equimolar amounts of Zr, Ti, Mo, Ta, and Hf (i.e., $(Zr,Ti,Mo,Ta,Hf)B_2$) was prepared. Commercial ZrB_2, TiB_2 and HfB_2 powders were batched with TaB_2 and MoB_2 that were laboratory synthesized. The resulting mixture was densified by hot pressing at 2200°C. Theoretical density of the ceramic was calculated using lattice parameters determined from X-ray diffraction analysis and molar mass determined by the nominal composition. Archimedes' method (ASTM B962) was used to determine bulk density. Scanning electron microscopy was utilized to determine grain size. Energy dispersive X-ray spectroscopy and X-ray diffraction were utilized to confirm homogeneity of the elemental species. Heat capacity and thermal diffusivity were measured and used, along with temperature dependent density, to calculate thermal conductivity from room temperature to 2000°C. Room temperature dynamic elastic modulus, shear modulus and Poisson's ratio for the material were determined following ASTM E1876-15. Electrical resistivity measurements were taken from room temperature to 1000°C. The behavior of this ceramic will be compared to nominally pure ZrB_2 ceramics prepared under similar conditions.

(ICACC-S12-P107-2018) Densification Kinetics and Phase Evolution of Zeta Phase Tantalum Carbide

E. C. Schwind*[1]; G. Hilmas[1]; W. Fahrenholtz[1]

1. Missouri University of Science & Technology, Materials Science and Engineering, USA

Zeta phase tantalum carbide (ζ-Ta_4C_{3-x}) is an ultrahigh temperature ceramic (UHTC) with an unusual combination of high strength (>700 MPa) and high fracture toughness (>10 MPa-$m^{1/2}$). High phase purity zeta phase tantalum carbide was synthesized and densified using reaction hot pressing. Tantalum hydride powder was used as the tantalum source while a mixture of phenolic resin and carbon black were used as the carbon source. Several samples with varying stoichiometries were made, with x ranging from 0.28 - 0.52 to determine which composition would result in the highest yield of the zeta phase. Differential scanning calorimetry and x-ray diffraction coupled with Rietveld refinement were used to study phase evolution. Densification kinetics were studied using reaction hot pressing at temperatures from 1800°C to 2100°C and times from 600 sec to 6000 sec. The microstructure of the synthesized ζ-Ta_4C_{3-x} was imaged using scanning electron microscopy. Fracture toughness and flexure strength were measured at room and elevated temperatures. This study will be the first report of ζ-Ta_4C_{3-x} synthesized from Ta and C as elemental precursors, in contrast to other studies that utilized tantalum carbide as a precursor.

(ICACC-S12-P108-2018) Layer-by-layer flash pyrolysis of polysilazane based polymers into a green UHTCMC and ablation resistance of the final dense composites

P. Galizia*[1]; L. Zoli[1]; R. Raj[2]; D. Sciti[1]

1. CNR-ISTEC, Italy
2. Unversity of Colorado Boulder, USA

The oxidation and ablation resistance are key properties of the Ultra-High Temperature Ceramics (UHTC) which are usually used as thermal protection system materials, and bulk materials for heating elements. In order to improve the oxidation and ablation resistanceof Ultra-High Temperature Ceramic Matrix Composites (UHTCMCs) based on ZrB_2 matrix and 0/90 carbon fibers, we have infiltrated the green samples with a preceramic polymer. The aim is to deposit layer-by-layer preceramic polymer films around the carbon fibers having a thickness of few hundred nm in order to pyrolyse the thin polymeric films in few seconds - by means the so-called flash pyrolysis - and obtain a crack-free SiNC coating. The resulting UHTCMCs were characterized by XRD, SEM, and RAMAN spectroscopy to highlight the conversion of polymer into ceramic phase and to characterize the microstructure of the composites. Finally, the ablation resistance has been investigated and here presented.

(ICACC-S12-P109-2018) Particle refinement of ZrB_2 by the combination of borothermal reduction and solid solution

W. Guo*[1]; H. Lin[1]

1. Guangdong University of Technology, China

Flexible synthesis of ultra-fine ZrB_2 powders was achieved by borothermal reduction of a mixture of ZrO_2, boron and TiO_2. Without TiO_2 additive, coarse ZrB_2 powders with particle size of 0.81 μm were obtained, presumably due to good wettability and solubility of ZrB_2 in the by-product B_2O_3. It was found that the particle growth of ZrB_2 was effectively inhibited by the solid solution of TiB_2 (≥1 mol%). The refinement mechanism was that the solid solution of in-situ formed TiB_2 presumably lowered the wettability and solubility of ZrB_2 in the B_2O_3 liquid and significantly inhibited the coarsening of ZrB_2. The average particle size of resulting powders decreased to 0.37 μm with the addition of 10 mol% TiO_2.

(ICACC-S12-P110-2018) The influence of parameters of Self-propagating High-temperature synthesis on the structure and properties of the MAX phases in Ti-Al-C system

A. Pazniak*[2]; P. Bazhin[1]; A. Stolin[1]; D. Kuznetsov[2]

1. Merzhanov Institute of Structural Macrokinetics and Materials Science Russian Academy of Sciences, Russian Federation
2. National University of Science and Technology "MISiS", Russian Federation

The MAX phases in the Ti–Al–C ternary system such as Ti_2AlC and Ti_3AlC_2 were synthesized by Self-propagating High-temperature Synthesis (extrusion and compression) at various technological parameters (delay time, compacting pressure, deformation velocity, and temperature) and molar ratios of the initial components (titanium, alumina and carbon black). The phase composition and lattice parameters of fabricated materials have been investigated by XRD as well as their structure and morphology were examined by SEM. First, the results show that some impurities present in samples, are almost TiC and TiAl intermetallides. Second, we found that the amount of impurities depends on the molar ratio of the initial components. Finally, stress-strain behavior in the synthesized MAX phases, depending on the manufacturing method (extrusion and compression) was studied. It can be concluded that almost pure Ti_3AlC_2 and Ti_2AlC could be prepared by self-propagating high-temperature synthesis of elemental Ti, Al and C powders by varying the ratio of materials and synthesis parameters.

(ICACC-S13-P111-2018) Fabrication of Gadolinia-containing UO2 fuel pellet

J. Oh[1]; Q. Mistarihi[2]; H. Ryu*[2]; D. Kim[1]

1. Korea Atomic Energy Research Institute, Republic of Korea
2. Korea Advanced Institute of Science and Engineering (KAIST), Republic of Korea

Both longer fuel cycles and higher burnups, which improve fuel utilization and fuel cycle economics, generally require the use of a burnable absorber for the control of power distribution and moderator temperature coefficient. Burnable absorber is material, such as boron, gadolinium, erbium, or dysprosium, which in their unirradiated state have high neutron absorption. Several different types of burnable absorbers are used with PWR fuel. For example, The Westinghouse-designed Integral Fuel Boron Absorber

(IFBA) rods contain uranium pellets with a thin coating of zirconium diboride (ZrB2). Wet Annular Burnable Absorber(WABA) consists of annular pellets of alumina-borncarbide (Al2O3/B4C) burnable absorber material. And, neutron absorbers such as gadolinia (Gd2O3) or erbia (Er2O3) are mixed directly with the uranium dioxide (UO2) fuel. Gadolinia is now the most commonly used as a burnable poison in commercial reactors due to a high neutron absorption cross section. In this paper, we investigate fabrication of UO2 pellets containing Gd2O3 sphere by conventional sintering process. Also, the interface and reactivity between UO2 and Gd2O3 was examined by SEM and EDS.

(ICACC-S13-P112-2018) Study on the cold sintering process of 8 mol% Y_2O_3-doped ZrO_2 by using a novel technique

D. C. Tanase*[1]

1. Korea Advanced Institute of Science and Engineering (KAIST), NQE, Romania

Cold sintering process (CSP) is becoming highly popular in the powder sintering field, being applied to a wide range of composites with very significant results in return. In this study the process is applied to 8mol% YSZ sub-micron powder while using water as an aqueous environment to mediate densification. By milling the YSZ powder before CSP using a 3D mixer for 3h and dried for 2h, it is expected to decrease the particle size and dissolve the sharp edges which will increase the efficiency of the first CSP stage, the dissolution-rearrangement process, leading to an increase of the relative density after sintering (above 90%). During CSP the temperature was raised up to 300°C under a uniaxial pressure up to 500 MPa and holding time of 2h using a modified setup composed of a Carver press and a ceramic heating plate. By studying the sinterability of 8mol% YSZ at low temperatures can offer an insight into CSP for UO2 fuel due to their similar crystal structure. A huge advantage of CSP for UO2 fuel fabrication would be that new variations of additives could be used. Having a much lower sintering temperature, this will limit volatilization and chemical interaction of the additives with the fuel. Also this can improve energy efficiency during manufacture and minimize pellet machining after sintering.

(ICACC-S13-P113-2018) New Ceramic Coatings for Small Modular Reactors: Applications in Hard Wearing Components and Accident Tolerant Fuel

E. Williamson*[1]; M. Whiting[1]; D. Robertson[2]; J. Yeomans[1]

1. University of Surrey, MiNMaT, United Kingdom
2. Rolls-Royce, Civil Nuclear, United Kingdom

Coatings, principally in the form of hard chrome plating, have been used in nuclear reactors to increase the durability of components. Ceramic coating materials to replace this heavily regulated material have been identified as having superior performance. The desire to increase fuel lifetime in the event of a loss of coolant accident is also driving the research for ceramic coatings for fuel cladding. Both applications are motivating the test programme for this research as Rolls-Royce develops a Small Modular Reactor. Design criteria for replacement materials include stability at the 600 K operating temperatures, neutron irradiation stability, high levels of wear resistance and a high temperature steam tolerance during a 60 year lifetime. A number of candidate coating materials have been identified and these require verification that they perform under these conditions. The coating materials investigated include Cr_2O_3, WC-(W,Cr)-Ni, CrC-NiCr, CrN and CrAlN. To date, radiation damage has been emulated using ion beam technology. Electron microscopy and x-ray diffraction techniques have been used to analyse the coatings before and after irradiation and preliminary results will be presented here.

(ICACC-S13-P114-2018) Coatings on SiC-based components for Light Water Reactors

P. Gianchandani[1]; V. Casalegno[1]; T. Hinoki[2]; L. Manna[1]; M. Ferraris*[1]

1. Politecnico di Torino, Department of Applied Science and Technology, Italy
2. Kyoto University, IAE, Japan

Light Water Reactors (LWR) are the most common, successful reactors employed today and SiC-based materials have been recently proposed to be used in these reactors. This study aims to synthetize suitable coatings for SiC-based materials, able to withstand the conditions inside the reactor. Three categories of coatings (Glass Ceramics, Metal Glasses and Metals) were used to coat SiC, SiC-based composites and porous SiC. All coated samples were then placed in an autoclave in presence of water at 350 °C, 18 MPa. Prior and post autoclave examination by FESEM will be discussed.

(ICACC-S13-P115-2018) Fabrication of SiC/SiC cladding tube with CVI-SiC process

N. Tomatsu*[1]; A. Kawaguchi[1]; T. Ito[1]

1. IBIDEN Co., Ltd., Japan

Recently, SiC/SiC composites have been shown to be promising structural materials for use in nuclear reactor cores to enhance safety. We are promoting development of SiC/SiC cladding tube with filament winding technology, braiding technology and CVI/CVD technology. However the fabricating process of SiC/SiC cladding tube (CVI/CVD process) has a problem of the strength deterioration of SiC fiber. In this presentation, we report on the test results with load assumed to be given to a cladding tube under using, such as tensile load, bend load and thermal shock, and on the analysis results of the strength deterioration of SiC fiber.

(ICACC-S13-P116-2018) Thermal and Mechanic Testing of ATF Fuel U_3Si_2

L. Cai*[1]; F. Boylan[1]; E. J. Lahoda[1]; H. Shah[1]; A. Atwood[1]

1. Westinghouse, USA

Accident tolerant fuels (ATF) attract a lot of attentions due to the improved safety margins and potential economic benefits. As a promising uranium fuel candidate for ATF, U_3Si_2 offers higher thermal conductivity and a higher uranium loading than UO_2. However, U_3Si_2 has never before been manufactured and applied as fuel for LWR use, thus, significant development work is needed. An experimental thermal analysis is undertaken to quantitatively determine the behaviors of U_3Si_2 to elevated temperatures in the water vapor atmosphere. The behaviors of U_3Si_2 at the typical LWR coolant temperatures and water vapor atmosphere are revealed by thermogravimetric (TG) isothermal experiments. The results will be discussed and compared to UO_2 for better understanding. The mechanical properties and pellet handling robustness are important for a new fuel design. In the manufacturing process, pellet chipping may occur during the fuel loading process. Missing pellet surfaces (MPS) caused by chipping is detrimental to fuel performance. The pellet handling and transportation robustness needs to be evaluated and quantified. The friability test results for U_3Si_2 will be reported and compared to UO_2. The compressive strength testing is also underway to investigate the loadability of the U_3Si_2 pellets.

(ICACC-S13-P117-2018) Raman spectroscopy experiments to characterize radiation induced defects in SiC/SiC composites

S. Agarwal[2]; Y. Zhao*[2]; S. J. Zinkle[1]; W. J. Weber[2]

1. University of Tennessee, Nuclear Engineering, USA
2. University of Tennessee, Material Science and Engineering, USA

Raman spectroscopy is powerful technique to study the damage produced in ion-irradiated solids. This work mostly focuses on the determination of changes in carbon content and bonding after ion irradiation in SiC/SiC composites. SiC-SiC composites were irradiated with 10 MeV Au ions at 350°C to 1-50 dpa to characterize

radiation induced defects relevant to light water reactor (LWR) conditions. This work is currently of high importance due to the possible use of SiC/SiC composites in accident-tolerant fuel cladding that can replace the current zirconium alloy cladding for the current fleet of LWRs. Some post-irradiation characterization experiments using profilometry and atomic force microscopy (AFM) has revealed the shrinkage of fibers. The reason for this shrinkage could be due to possible changes in the carbon content and bonding which has been characterized by Raman spectroscopy on bulk samples. Additional characterization using Raman, on lamella prepared by focused ion beam (FIB) will also be summarized to provide depth-dependent information on disordering of carbon bonds.

(ICACC-S13-P118-2018) Metal silicide reaction-bonded silicon carbide for nuclear fusion applications

A. J. Leide*[1]; R. I. Todd[1]; S. G. Roberts[1]; K. Yoshida[2]; T. Yano[2]; M. Gorley[3]; D. E. Armstrong[1]

1. University of Oxford, Department of Materials, United Kingdom
2. Tokyo Institute of Technology, Laboratory for Advanced Nuclear Energy, Institute of Innovative Research, Japan
3. Culham Centre for Fusion Energy, United Kingdom

Silicon carbide has potential applications in the breeder blanket of fusion reactors, where its chemical inertness, low neutron cross-section, and radiation tolerance are especially useful. However, manufacturing large, complicated blanket components for a fusion reactor using "nuclear grade" SiC composites is currently very challenging. Large components can be made reliably to near net shape and at an economical price using reaction-bonded silicon carbide (RB-SiC). In unirradiated form, the properties are suitable for fusion applications, however, early irradiation work on commercial grades of RB-SiC suggested that radiation-induced swelling causes stresses between SiC and residual silicon leading to failure. If residual silicon can be removed or replaced, RB-SiC may become a viable nuclear material. In an attempt to solve this, SiC-$CrSi_2$ and SiC-WSi_2-Si composites with fine grain structures have been made by the reaction-bonding process, and have been investigated using micromechanical techniques, SEM, Raman microscopy, and X-ray tomography. Ion implantation is used to investigate the changes to these novel materials under irradiation, and assess their suitability for the nuclear environment.

(ICACC-S13-P119-2018) Calculation of Hot Cell Subcritical Limits at Oak Ridge National Laboratory

S. Stanfill*[1]

1. University of Florida, USA

The American National Standard, ANSI/ANS-8.15-2014, Nuclear Criticality Safety Control of Selected Actinide Nuclides, has been released, revising many of the subcritical limits from its previous issue. Oak Ridge National Laboratory (ORNL) utilizes limits from this standard in its Radiochemical Engineering Development Center (REDC) to ensure the safe handling of these nuclides. The new limits provided by ANSI/ANS-8.15-2014 are much lower than the current limits in place for key nuclides Cm-245, Am-242m, and Pu-241. To evaluate potentially increasing these limits, calculations simulating the chemical forms and reflector conditions specific to the hot cells in REDC were performed using Monte Carlo N-Particle (MCNP) software. First the calculations that the ANSI/ANS-8.15-2014 values are based on were performed to demonstrate competence in reproducing results. Next, MCNP calculations were performed modeling the REDC hot cell conditions. Finally, the safety factors used on the ANSI/ANS-8.15-2014 limits were applied to the REDC calculation results to determine new subcritical limits for the key nuclides.

(ICACC-S16-P120-2018) Textile Reinforced Cementitious Composites based on Phosphate Cements for Construction Purposes

A. Katsiki*[1]; Y. Pontikes[2]; H. Rahier[1]

1. Vrije Universiteit Brussel, Department of Materials and Chemistry, Belgium
2. KULeuven, Belgium

In this work, the prospects of using acidic activation of metakaolinite (MK) as the main binder in textile reinforced composites production are investigated. Due to the acidic environment of the fresh phosphate cementitious paste and the fact that it becomes pH neutral after hardening, it provides a mild environment for the embedded fibers. Therefore, use of E-glass fibers is feasible. The optimum matrix composition is chosen based on both calorimetric and mechanical testing experiments, and in accordance to specific requirements such as rheology and workability. Two-dimensional glass fabric sheets, randomly chopped, tri-axial, as well as a combination of 2D together with 3D symmetric glass fiber fabrics are applied to check the impregnation, workability, mechanical strength and durability of the final composite. The mechanical properties are evaluated on beams loaded in tension and under bending. The effect of the fiber volume fraction on the mechanical properties of the textile reinforced inorganic polymer (IP) composites are investigated as well. The results reveal that MKPC binder is well suited for developing textile reinforced cementitious composites, which can be promising candidates for a high-performant, fire-resistant, sustainable construction system.

(ICACC-S16-P121-2018) Geopolymers as Heterogeneous Catalysts in the Production of Biodiesel

R. Botti[1]; M. Innocentini[2]; P. Pastore[3]; L. San Gregorio[2]; P. Colombo*[1]

1. University of Padova, Industrial Engineering, Italy
2. UNAERP, Brazil
3. University of Padova, Chemical Sciences, Italy

Biodiesel is a substitute for polluting fossil fuels. The most used process to prepare biodiesel is by homogeneous transesterification of vegetable oils, despite the fact that it produces a high concentration of impurities in the product. The use of heterogeneous catalysts is quite attractive, their main advantage being that the purification step is minimized. In this work, we investigated the biodiesel production by transesterification of soybean oil with methanol, using geopolymers as heterogeneous catalysts. Two types of heterogeneous catalysts were prepared in powder form: sodium- and potassium-based geopolymers. The influence of heat treatment was also evaluated. The biodiesel was obtained in the following reaction conditions: methanol:oil ratio, 7.5:1; catalyst amount 3% of oil (w/w); reaction temperature, 60°C; reaction time, 1h. The results demonstrate that both geopolymers work as catalysts, and the conversion is higher when the potassium based geopolymer is used (~60% versus ~50%, potassium and sodium respectively), possibly due to the higher surface area of the potassium-based geopolymer. Moreover, the yield of reactions seems to be dependent on the geopolymer heat treatment. It should be noted that the biodiesel product still contains some Na or K impurities, so a further optimization of the material and the process is required.

(ICACC-FS1-P122-2018) Imprinting Biomimetic Antireflective Structures on Polymer Surface

H. Xu*[1]

1. Harbin Institute of Technology, Chemical Process, China

Excellent antireflection thermoplastic polymers hold an important position as materials for optics, electro-optical devices and sensors nowadays. As inorganic compound is gradually replaced with plastics that applies to meet the requirements of a broadening range. The advantages of plastics are significant height modulus, easily fabrication, and low cost in mass production. Among plastics,

polymethylmethacrylate (PMMA) is one of the most convenient and frequently used polymers in precision optics and electro-optics. Herein, we report a simple imprinting method to generate antireflective structures on polymer surface. The stamps used in this work were fabricated by sphere monolayer masked reactive ion etching (RIE). The imprinted polymer exhibits excellent broadband antireflection properties. The obtained PMMA structures can reduce the reflectivity to less than 1% at the wavelengths from 400 nm to 2400 nm. This technique combines the simplicity and scalability of self-assembly and nanoimprint. These antireflective structures may have potential applications in optical devices and solar cells. In summary, we present a simple imprinting technique for generating antireflection sub-wavelength arrays on polymer. The fabricated PMMA structures by using self-assembled Si imprinting stamp exhibit excellent antireflection properties on the bandwidth of 400-2400 nm.

(ICACC-FS1-P123-2018) Detection of Homologues and Isomers Based on Hollow Mesoporous Silica Sphere Photonic Crystals

C. Xiong*[1]; B. H. Geng[1]; Y. Li[1]

1. Harbin Institute of Technology, China

Recently, researchers have investigated natural opals and the wings of butterflies and peacocks because these structures may exhibit dazzling colors. The colors are attributable to periodic structures. Photonic crystals (PCs) are dielectric materials with periodic modulation of the refractive index and photonic band-gap properties. These periodic structures have attracted increasing attention in recent years because they have potential applications in many areas, such as display of structural colors, photo-catalysis, and advanced sensors. A PC sensor that can detect homologues and isomers with very similar refractive indices through stopband shifts has been prepared from highly monodispersed hollow mesoporous silica spheres (HMSSs) for the first time. The HMSS PC shows two obvious reflection peaks. When the HMSS PC sensors are infiltrated with 10 μL of homologues or isomers, the positions of the second reflection peaks remain nearly unchanged, while the first reflection peaks shift between 60 and 200 nm. Difference in the refractive indices of n-propanol and iso-propanol is only 0.008, however, the difference in the stopband shifts is approximately 25 nm, showing the high sensitivity in distinguishing homologues and isomers of the HMSS PC. The proposed strategy provides a convenient, accurate and low-cost visual method to detect homologues and isomers.

(ICACC-FS1-P124-2018) Transparent Superhydro phobic coating from silica spheres

Z. Li*[1]; N. Li[2]; L. Pan[2]; H. Xu[2]

1. High School Attached to Harbin Normal University, China
2. Harbin Institute of Technology, China

Transparent superhydrophobic thin films were prepared by utilizing SiO_2 spheres coating with perfluorosilane. That can forms a paint be sprayed, dipped, or extruded onto both hard and soft materials to create a self-cleaning surface. The transparency of the films decreased with increasing SiO_2 concentration, which was attributed to the size difference of the starting materials. The hydrophobic SiO_2 spheres can be used on cotton, wood, glass and paper as a self-cleaning applications.

(ICACC-FS1-P125-2018) Surface analysis and mimicked surface preparation of a firebrat, Thermobia domestica

Y. Hirai*[1]; S. Uemura[1]; N. Okuda[1]; M. Shimomura[1]

1. Chitose Insititute of Science and Technology, Department of Applied Chemistry and Bioscience, Japan

Friction is an important issue with regard to saving energy and preventing the wear of parts in a wide variety of fields. In recent years, biomimetics attract much attention due to their possibility for creating novel functional materials. In this report, we focused on firebrat, Thermobia domestica, because firebrat lives in narrow openings and their surfaces are frequently in contact and wear by other surfaces. So, we speculate that their body surface would be adapted to reduce friction. To investigate firebrat surface functional properties, we analyzed firebrat surfaces by field emission scanning electron microscopy (FE-SEM) and atomic force microscopy (AFM) with colloidal probes. FE-SEM observation revealed that firebrats were completely covered by scales, which surfaces have periodic microgroove structures. And groove periods are almost uniform within each scale. However, groove periods around the head vary between scales. AFM friction force measurements revealed that firebrat scale reduces friction by decreasing contact area. Further, the heterogeneous groove periods of the scales suggest that it prevents to fix the whole scales in particular outer rough surfaces. Moreover, we also prepared firebrat scale mimicked surface by using self-organized wrinkle structures. Preparation methods and their frictional properties will be discussed.

(ICACC-FS3-P126-2018) PEALD-TiN based Thin Films for High Performance Metallic Bipolar Plates of PEMFCs

C. Kim*[1]; E. Yun[1]; S. Park[1]; J. Anh[2]; S. Kwon[1]

1. Pusan National University, School of Materials Science and Engineering, Republic of Korea
2. Korea Maritime and Ocean University, Department of Electronic Materials Engineering, Republic of Korea

Metallic bipolar plate, a part in polymer electrolyte membrane fuel cells (PEMFCs), is required high electrical conductivity, corrosion resistance and low interfacial contact resistance (ICR). Au and carbon-based protective coatings have been introduced for this application, however, they are hard to be commercialized due to high cost and poor electrical conductivity, respectively. Moreover, some depositing technologies, such as CVD, PVD or electroplating, do not always achieve satisfactory performance. Thus, extensive efforts are needed to develop alternate materials and processes. Therefore, in this work, we firstly reported the TiN thin films, with good electrical and corrosion properties, deposited by plasma-enhanced atomic layer deposition (PEALD) as protective coating applied on bipolar plate. Due to the inherent merits of PEALD, low-cost but high performance TiN protective film with ultrathin thickness (25~67nm) was achieved to meet the strict requirements of the DOE (corrosion current density: $\langle 10^{-6}A/cm^2$, ICR value $\langle 20m\Omega cm^2$) by using optimized PEALD process. We investigated which type of PEALD (based on halide and metal-organic precursors) was most appropriate and discussed the reasons for the superior performance of TDMAT-based TiN for this application. Based on our study, the formation of ultrathin amorphous interfacial layers was a key factor to ensure the performance.

(ICACC-FS3-P127-2018) Block copolymer-templated hollow n-ZnO/p-Si nanodiode arrays using atomic layer deposition

D. Kim*[1]; H. Lee[1]; C. Kim[1]; W. Lee[1]; S. Kwon[1]

1. Pusan National University, School of Materials Science and Engineering, Republic of Korea

Block copolymer (BCP) self-assembly, generating highly ordered arrays with small feature sizes of 5~50 nm, such as dot, line, hole, and lamellar patterns, is one of the most promising candidates for future nanolithography. BCP nanostructures can be easily modified by atomic layer deposition (ALD), leading to conformal and well-organized hybrid-pattern at nanoscale. Hence, many researchers focused on the combintion of BCP and ALD process. Usually, etching is required as a key process for fabricating nanopatterns. However, systemically study, with BCP self-assembly, ALD, and inductively coupled plasma (ICP) dry etching process, has not been reported yet. Herein, we presented a novel methodology to obtain one-dimensional n-ZnO/p-Si nanoarrays by combining the three techniques mentioned above. In order to obtain the highly ordered nanotube pattern, we employed a self-assembled Si-containing PS-b-PSSi BCP on SU-8/p-Si wafer as a template firstly, then ICP etch-back process was performed after ALD-ZnO deposition to

produce well-defined n-ZnO/p-Si nanotube arrays with diameters of 52 nm (outer) and 25 nm (inner). In addition, n-ZnO/p-Si nanorod pattern was also achived by precisely controlling the ALD cycles and ICP etching time. This simple and useful approach provides a very convenient route to fabricate high-density nanodiode patterns without using high-cost photolithography.

(ICACC-FS3-P128-2018) Preparation of Perovskite-Structured Gadoliniumorthoferrite ($GdFeO_3$) Nanocrystals by Hydrolysis of a Novel Heterobimetallic Precursor

C. Bohr*[1]; C. Hegemann[1]; L. Wortmann[1]; J. Schläfer[1]; S. Mathur[1]

1. University of Cologne, Institute of Inorganic Chemistry, Germany

Bimetallic oxides are of substantial interest due to their unique functional properties like piezoelectricity, luminescense and cooperative effects (e.g. multiferroicity) emerging from the presence of different metallic centers in the crystal structure. Especially orthoferrites exhibit special magnetoelectric properties (e.g. G-Type magnetism) making them interesting for applications in magneto optic devices, as photo catalysts or in gas sensing. Here, the selective synthesis of phase-pure $GdFeO_3$ perovskite nanoparticles in contrast to $Gd_3Fe_5O_{12}$ garnet nanoparticles have been performed using the new bimetallic, metalorganic precursor $[GdFe(O^tBu)_6(py)_2]$ (py = pyridine) in a solvothermal procedure. The used single source precursor was synthesized by a metathesis reaction of $[Gd\{N(SiMe_3)_2\}_3]$ and $[Fe(O^tBu)_3]_2$ and characterized by single crystal X-ray diffraction, mass spectrometry, thermogravimetry and elemental analysis. The metal ratio in the single source precursor was preserved by controlling the metal ratio during the reaction process. The synthesized perovskite nanocrystals give a first glance at phase-pure $GdFeO_3$ derived from this new precursor. The application in spray pyrolysis and atomic layer deposition (ALD) is currently under investigation.

(ICACC-HON-P129-2018) Sialon-based materials prepared from the aluminium oxynitride SHS-derived powders

A. D. Wilmanski[1]; M. M. Bucko*[1]

1. AGH University of Science and Technology, Poland

Sialons which are a specific solid solutions in the Al_2O_3-Si_3N_4 system are widely applied due to their good mechanical properties, high thermal shock resistance, and corrosion resistance, especially against molten metals. Dense sialon materials are usually obtained by pressureless sintering or by HP, HIP and SPS techniques and the starting materials for sintering are usually mixtures of respective oxides and nitrides usually silicon nitride and corundum. In the present work SHS-derived aluminium oxynitride with spinel structure, γ-alon, powder was used as a precursor of sialon-based materials. The γ-alon powder was mixed with different amount of commercial silicon nitride powder and 1 mass% of yttrium oxide then compacted samples were pressureless sintered at 1750°C for 2h in argon. Materials sintered from the powder mixtures containing up to 50 mass% of γ-alon were composed of β-sialons only. Materials with the formally higher amount of γ-alon show presence of alumina and rhombohedral sialon. SEM images of the β-sialon materials are typical for such materials sintered with presence of liquide phase and sizes of the sialon grains ranged from less than 1 to more than 10 μm. Bending strength of the sintered materials is strongly depend on chemical composition of the samples, density and microstructure; their strength decrease with increase of formally amount of alumina into the β-sialon structure.

(ICACC-HON-P130-2018) Crack-Healing Ability and Strength Recovery of Ytterbium Disilicate Ceramic Reinforced with Silicon Carbide Nanofillers

S. T. Nguyen*[1]; H. Iwasawa[1]; H. Suematsu[1]; L. He[2]; T. Suzuki[1]; K. Niihara[1]; T. Nakayama[1]

1. Nagaoka University of Technology, Japan
2. Idaho National Lab, USA

The SiO_2 volatility and SiC recession in hydrocarbon-rich combustion atmosphere is the major drawback of SiC-reinforced ceramics when they are applied as coating materials for turbine blades. The composite of $Yb_2Si_2O_7$-Yb_2SiO_5-SiC is expected as a self-crack healing material that can overcome this problem, because the monosilicate can react with the SiO_2 to form disilicate and hence further reinforce the composite. In this study, the composites, fabricated with various morphology of SiC nanofillers, were pre-cracked and then annealed in an oxidizing environment to investigate their crack healing behavior. The healing effect as a function of annealing time and annealing temperature was studied. Healing mechanism and future applications of these composites were also discussed.

(ICACC-HON-P131-2018) Study on shielding method for reducing the leakage magnetic field from magnetically shielded room opening

H. Sugiyama*[1]; K. Kamata[3]; T. Nakayama[2]; T. Suzuki[2]; H. Suematsu[2]; T. Tokuda[3]

1. Nagaoka University of Technology, Department of Science of Technology Innovation, Japan
2. Nagaoka University of Technology, Extreme Energy-Density Research Institute, Japan
3. National Institute of Technology, Kagoshima College, Department of Electronic Control Engineering, Japan

There are several sources of unwanted magnetic noise, such as electrical transmission lines, induction motors, and the movement of ferromagnetic material through the Earth's magnetic field. Several types of precision instruments may be adversely affected by magnetic noise, requiring them to be placed in a magnetically shielded room (MSR) to reduce magnetic noise. An MSR typically consists of multiple shielding layers of ferromagnetic material, with openings in the walls and the floor to allow for ventilation, wiring, and plumbing connections. However, magnetic noise can leak into the MSR through those openings, and anything inside can be influenced by the leaked magnetic flux density. The leaked magnetic flux density is changed by value and direction of it outside of the MSR. Generally, a duct or a partition plate made of a ferromagnetic material is used to reduce magnetic flux density entering through room openings. In this study, we focus on the partition plate installed in the duct, and examine the effects of partition plate length and the number of plate divisions. The shape of the partition plate with high shielding effect was studied by 3-dimensional magnetic field analysis using the finite element method.

Thursday, January 25, 2018

S1: Mechanical Behavior and Performance of Ceramics & Composites

Processing-Microstructure-Properties

Room: Coquina Salon D

Session Chairs: Emmanuel Boakye, UES Inc.; Rajiv Asthana, University of Wisconsin-Stout

8:30 AM

(ICACC-S1-047-2018) Dual Function Polymer-Derived Non-Oxide/Oxide Matrix Prepared by Additive Manufacturing

R. Raj*[1]

1. Unniversity of Colorado Boulder, USA

A nanolayer-at-a-time additive manufacturing approach is employed to create a dense matrix of polymer-derived ceramics around fiber bundles of silicon carbide. Thin ultrathin films pyrolyze quickly without cracks with a cycle time of about one minute. The polymer precursor for SiCN is mixed with a precursor of hafnium oxide, which produces a matrix containing a fine dispersion of hafnium silicate. The oxide/nonoxide matrix leads to both good mechanical properties as well as remarkable oxidation resistance.

8:50 AM

(ICACC-S1-066-2018) Evaluation of boron nitride materials for electric propulsion components

J. Mackey*[1]; B. Mcenerney[2]; J. Salem[1]; P. Peterson[1]; H. Kamhawi[1]; R. Hofer[2]

1. NASA Glenn Research Center, USA
2. NASA Jet Propulsion Laboratory, USA

Historically, several grades of hot pressed hexagonal boron nitride have been used for electric propulsion components. This study investigates the material properties of a selection of commercially available boron nitride grades including HP, M26, M, BNXX, and Shapal Hi-M. The grades selected for this study are of interest because their available billet size is sufficient for electric propulsion components, or because they have heritage in electric propulsion applications. The research investigates a range of material properties, tailored and focused on enhancing performance, reliability, and economics of Hall thrusters. Moisture absorption, moisture sensitivity, hot press directionality, flexural strength, elastic modulus, thermal conductivity, thermal emissivity, thermal expansion, density, X-ray diffraction phase, microstructure, and chemical composition were all investigated to help guide the selection of advanced ceramics. A summary of the relevant material properties of the grades will be presented in context to electric propulsion systems.

9:10 AM

(ICACC-S1-049-2018) Deposition temperature effects on microstructure and lifetime of the thermal barrier coating produced with axial suspension plasma spraying

D. Zhou*[1]; R. Vassen[1]

1. Forschungszentrum Jülich GmbH, Institute of Energy and Climate Research, Materials Synthesis and Processing (IEK-1), Germany

Suspension Plasma Spraying (SPS) as a very promising process for producing high strain tolerant Thermal Barrier Coatings (TBCs) has attracted intensive interest during last decade. In this work, the effects of deposition temperature on microstructure, porosity, mechanical properties and thermal cycling lifetime of axial SPS TBCs were investigated. By increasing deposition temperature range from 280-370 °C up to 400-550 °C, column density of the coatings was barely affected; however, porosity of the coatings especially porosity within columns was greatly decreased. Due to the decrease of porosity, the coatings deposited at higher temperature exhibited higher hardness, elastic modulus and fracture toughness. The effects of mechanical properties on thermal cycling lifetime of coatings were also discussed. It seems that even though, higher fracture toughness is benefit for the lifetime; higher elastic modulus introduced higher thermal stress leading to decrease of lifetime.

9:30 AM

(ICACC-S1-050-2018) Microstructure and properties of B_4C-SiC composites by hot pressing pyrolyzed mixtures of B_4C and polycarbosilane

W. Wang*[1]; Z. Fu[1]; H. Wang[1]

1. Wuhan University of Technology, China

The B_4C-SiC composites with fine grained microstructure were fabricated by hot pressing pyrolyzed mixtures of B_4C and polycarbosilane(PCS) without and with the addition of Si. The compositions, microstructure and mechanical properties of the fabricated composites hot pressed at 1950°C for 1 hour under the pressure of 30MPa were studied. SiC derived from pyrolysis of PCS promoted densification of B_4C effectively and markedly enhanced the fracture toughness of composites. The sintering activity and mechanical properties of the composites could be further improved by the addition of Si owing to the formation of liquid Si and the elimination of free carbon during sintering. The relative density, Vickers hardness and fracture toughness of the composites with PCS plus 8wt% Si could reach 99.1%, 33.5GPa and 5.57MPam$^{1/2}$ respectively. A number of laminated flakes structures and dislocations were observed in the B_4C-SiC composite. Complicate microstructure and crack bridging by the homogenously dispersed SiC grains as well as crack deflection by SiC nanoparticles may give an explanation for the improvement in toughness.

10:10 AM

(ICACC-S1-051-2018) Insights into Transparent MgAlON Solid Solution Spinel Ceramics through Theoretical and Experimental Studies

H. Wang*[1]; B. Tu[1]; X. Liu[1]; L. Ren[1]; X. Zong[1]; W. Wang[1]; Z. Fu[1]

1. Wuhan University of Technology, China

Transparent ceramic based on spinel structure has been regarded as one of the most important material groups for both optical and mechanical applications, due to its outstanding optical transparency and mechanical properties. In this family, aluminum oxynitride (γ-AlON) and Magnesium aluminate ($MgAl_2O_4$) are the most important candidates for optical windows, domes and lenses for ultraviolet, visible, and infrared application. Nevertheless, the limited choice (i.e. $MgAl_2O_4$ and γ-AlON) still motivated the development of new spinel transparent ceramics with adjustable properties. In this work, MgAlON spinel compositions were designed based on the First-principle calculations and MgO-AlN-Al_2O_3 phase diagram, which were subsequently densified by pressureless and hot isostatic press sintering. The crystalline structure of these materials was resolved by Rietveld refinement with X-ray powder diffraction data combining with the spinel structure modeling procedure. The chemical bonding of crystals were quantitatively analyzed by the bond valence method, and properties such as the optical, mechanical, thermal properties of MgAlON transparent ceramics were investigated. Through a serial of quantitative prediction models constructed by us, the insights into the correlation among composition, crystalline structural and properties could be realized.

10:30 AM

(ICACC-S1-052-2018) Development of 'Age-hardened/ toughened' Bulk Polycrystalline 'Ceramic Alloys'

L. Gurnani*[1]; M. K. Singh[1]; R. Kathuria[1]; P. Bhargava[1]; A. Mukhopadhyay[1]

1. Indian Institute of Technology (IIT) Bombay, Powai, Metallurgical Engineering and Materials Science, India

The present research demonstrates the development of 'age-hardened ceramic alloys' in bulk polycrystalline form via facile and cost-effective processing route, as viable alternative for the more 'conventional' bulk ceramic nanocomposites, which present significant processing challenges and microstructural non-uniformity. The route, as adopted here, is based on basic physical metallurgical principles of in-situ precipitation of second phase particles during aging of supersaturated ceramic solid solutions. MgO-7wt.%Fe_2O_3 bulk supersaturated solid solutions were obtained directly during pressureless sintering in air at 1650°C (sintering-cum-solution treatment), followed by 'air quenching' using raising hearth furnace. Subsequent 'aging' treatments at lower temperatures and for different durations led to the precipitation of nanosized second phase ($MgFe_2O_4$) particles, which were uniformly dispersed inside the matrix grains, as well as along the grain boundaries. The second phase particles maintained coherency with the MgO matix, even after aging for 20 h. Such near-ideal microstructure developments, along with the presence of coherent second phase particles, are not possible in the case of ceramic (nano)composites. The bulk MgO-based ceramic alloys possess improved hardness (by up to ~52%), indentation toughness (by up to ~35%) and wear resistance (~30% reduction in wear rate) w.r.t. to monolithic MgO.

10:50 AM

(ICACC-S1-053-2018) Mechanical properties and lifetime predictions of $SrTi_{1-x}Fe_xO_{3-\delta}$ (x = 0.25, 0.35, 0.5)

R. Oliveira Silva*[1]; F. Schulze-Kuppers[2]; S. Baumann[2]; J. Malzbender[1]; O. Guillon[2]

1. Forschungszentrum Juelich, Institute for Energy and Climate Research (IEK-2), Germany
2. Forschungszentrum Juelich, Institute for Energy and Climate Research (IEK-1), Germany

Oxygen transport membranes based on mixed ionic-electronic conducting ceramics can be an alternative to existing state-of-the-art processes for oxygen production in small and medium scale. Typically, such membranes have to be operated at 800 - 900 °C and under large pressure gradients, which challenges significantly the mechanical stability of the respective ceramic components. The current work concentrates on the mechanical characterization of promising oxygen transport perovskite membranes based on $SrTi_{1-x}Fe_xO_{3-\delta}$, (STFx) with x = 0.25, 0.35 and 0.5. The materials were synthesized through solid state reaction and most mechanical testing relevant specimens were obtained by tape casting. The mechanical stability was assessed via ring-on-ring bending tests serving the estimate of materials' reliabilities and lifetime. Furthermore, aiming towards the determination of the mechanical stability at the operational relevant temperatures, flexural tests were conducted at 900 °C, where the derived fracture stress revealed to be higher. Hence, it appears that the material undergoes stress relaxation at high temperature associated to an anelastic behavior that might relate to a strong primary creep. Overall, the current study aids the adjustment of the membrane design regarding the process requirements, while data for mechanically derived lifetime predictions are acquired for STF based membranes.

11:10 AM

(ICACC-S1-054-2018) Development of SiC-coated carbon nanotubes with improved oxidation resistance as stand-alone and as reinforcement in bulk polycrystalline Al_2O_3

S. Galaveen*[1]; M. K. Satam[1]; L. Gurnani[1]; V. Thiruvenkatam's[2]; A. Mukhopadhyay[1]

1. IIT Bombay, Metallurgical Engineering & Materials Science, India
2. Vikram Sarabhai Space Centre, ISRO, Materials and Mechanical Entity, India

Multi-walled carbon nanotube (MWCNT) reinforced engineering ceramics are usually not suited for high temperature applications due to the MWCNTs getting oxidized at above 600 °C. In order to overcome this drawback, β-SiC coated MWCNTs have been developed via innovative, but facile low temperature route by dispersing MWCNTs in silica sol, followed by calcination-cum-reduction at just 600 °C in the presence of Mg and C. Thermal analysis up to 1500 °C in air confirmed significant improvement in oxidation resistance of MWCNTs upon being coated with ~5 nm thick uniform SiC layer. The SiC-coated MWCNTs were incorporate within bulk polycrystalline Al_2O_3 matrix, especially at the matrix grain interiors, by adding them in dispersed state to matrix sol under sonication, followed by quick conversion of sol to gel and subsequent spark plasma sintering at 1300 °C for 5 min in Ar. In general, the presence of MWCNTs (SiC-coated or uncoated) within the matrix grains led to significant improvements in mechanical and tribological properties (~95% reduction in wear rates). Additionally, oxidation tests in air up to 1000 °C indicated that, while the pellets containing uncoated MWCNTs lost nearly all the MWCNTs to oxidation, pellets containing SiC-coated MWCNTs could retained them; thus rendering these ceramics suitable for high temperature applications.

11:30 AM

(ICACC-S1-055-2018) Kinetic and Properties of Boron Nitride coating via Chemical Vapor Deposition

M. Wang[1]; Y. Peng*[1]; L. Jia[1]; A. Li[1]

1. Shanghai University, School of Materials Science and Engineering, China

The chemical vapor deposition (CVD) of boron nitride (BN) occurred in a vertical hot-wall apparatus using BCl_3+NH_3+H_2+N_2 precursors system. The surface deposition rate was studied under various partial pressures of BCl_3+NH_3+H_2 ($P_{BCl3+NH3+H2}$), at the temperature of 923K and total pressures of 7kPa, 12kPa respectively. Based on the experimental results, the surface deposition rate of BN increased at first then deceased with increasing $P_{BCl3+NH3+H2}$, and the maximum of deposition rate appeared at 5kPa or 6.67kPa (total pressures of 7kPa, 12kPa respectively). The relationship between the surface deposition rates of BN and $P_{BCl3+NH3+H2}$ is third-order reaction before severe homogenous nucleation in gas phase. The BN coatings could be uniformly deposited on the surface of silicon carbide (SiC) fibers shown by scanning electron microscope(SEM), except for several large grains caused by gas phase nucleation in the case of excessive $P_{BCl3+NH3+H2}$. The formation of h-BN is verified by means of X-ray diffraction(XRD), X-ray photoelectron(XPS) and FT-IR spectroscopy after heat treatment at 1473K.

S3: 15th International Symposium on Solid Oxide Fuel Cells (SOFC): Materials, Science and Technology

Interfacial Reactions

Room: Crystal
Session Chair: Tatsumi Ishihara, Kyushu University

8:30 AM

(ICACC-S3-040-2018) Probing Interfacial Properties of Heterostructures in Solid Oxide Fuel Cells (Invited)

K. Develos-Bagarinao*[1]; H. Kishimoto[1]; T. Ishiyama[1]; T. Horita[1]; K. Yamaji[1]; H. Yokokawa[2]

1. National Institute of Advanced Industrial Science and Technology (AIST), Research Institute for Energy Conservation, Japan
2. The University of Tokyo, Institute of Industrial Science, Japan

Degradation of cell performance has been attributed in part to the formation of secondary phases at heterointerfaces due to cation interdiffusion occurring between cathode and electrolyte materials used for solid oxide fuel cells (SOFCs). The formation of $SrZrO_3$ (SZ) at the interfaces of perovskite oxides such as LaSrCoFeO (LSCF) often used in conjunction with state-of-the-art electrolytes such as yttria-stabilized zirconia (YSZ), has been correlated to increased resistance with operation time. Its formation can be mitigated by utilizing suitable barrier interlayers such as gadolinia-doped ceria (GDC), however, this is unable to completely prevent interdiffusion. To elucidate the effect of SZ on oxide ion transport, we examined dense model heterostructures comprising GDC, SZ and YSZ layers using isotope exchange depth profile method. Oxide ion transport appears to be drastically inhibited at the interface of SZ with YSZ, indicating the existence of an oxide ion barrier which blocks ionic flow. This result provides a crucial insight into understanding the lowering of conductivity in complicated cathode–electrolyte layers for SOFC applications.

9:00 AM

(ICACC-S3-041-2018) Effect of Cr Concentration in Cathode Air on Performance Degradation of SOFCs with LSM/YSZ Cathodes (Invited)

J. S. Hardy*[1]; C. A. Coyle[1]; J. Neeway[1]; D. J. Edwards[1]; A. Devaraj[1]; J. W. Stevenson[1]

1. Pacific Northwest National Laboratory, Materials Science, USA

Electrochemical tests of cells with LSM/YSZ cathodes were performed in a test fixture developed for testing anode-supported SOFC button cells downstream from a chromia pellet placed in the cathode air stream at a prescribed humidity and temperature to control volatilization of Cr vapor species. A porous alumina foam coated with Cr gettering Na_2CO_3 was placed downstream from the cell to capture the Cr from the cathode air stream by reacting with Cr vapor species to form Na_2CrO_4. The coating and its reaction product are both water soluble which facilitates dissolving them from the porous substrate for subsequent ICP analysis to determine the concentration of the resulting aqueous solution. The mass of collected Cr was calculated and used to determine the average concentration in the known volume of air that flowed past the cathode over the duration of the test. Multiple tests were performed for ~700 hours with the chromia pellet at varying conditions of temperature and humidity to elicit various levels of Cr volatility. The effects of Cr concentration in the cathode air on performance degradation of cells with LSM/YSZ cathodes were found to range from no significant degradation at ≤10 ppt Cr to ~14%/kh degradation at 6.6-6.7 ppb Cr. Post-mortem TEM, APT, and STXM analyses will be discussed.

9:30 AM

(ICACC-S3-042-2018) Effect of composition of LSCF thin films on $SrSO_4$ formation

J. C. De Vero*[1]; K. Develos-Bagarinao[1]; S. Liu[1]; T. Ishiyama[1]; H. Kishimoto[1]; K. Yamaji[1]; T. Horita[1]; H. Yokokawa[2]

1. National Institute of Advanced Industrial Science and Technology (AIST), Research Institute of Energy Conservation, Japan
2. Tokyo University, Japan

Lanthanum strontium cobalt ferrite (LSCF) cathode thin films grown by physical vapor deposition such as pulsed laser deposition (PLD) are often reported to exhibit significant Sr-segregation than conventional porous cathodes (1). The segregated Sr may react with air contaminant such as sulfur (SO_2) forming $SrSO_4$ on the surface. Significant $SrSO_4$ formation in LSCF is known to affect cell performance negatively (2-3). In this work, the $SrSO_4$ formation is investigated by growing two types of LSCF films on 10 mol% Gd-doped ceria by PLD technique, one is A-site rich LSCF, and the other is stoichiometric $La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_{3-\delta}$ films. After annealing at 800°C for 100 h, the A-site rich (La-rich) LSCF films showed severe Sr-segregation whereas the stoichiometric films exhibited no apparent Sr-segregation. SEM-WDS analysis revealed that the segregated particles are $SrSO_4$. This result indicates that Sr-segregation in A-site rich LSCF is more pronounced and reacted actively with sulfur impurities in air. The implication of these results is important in understanding the factors affecting $SrSO_4$ formation in LSCF cathodes.

Contaminants

Room: Crystal
Session Chair: Henrik Frandsen, Technical University of Denmark

10:20 AM

(ICACC-S3-043-2018) Role of Select Minor Airborne Impurities on SOFC Cathode Degradation: Computational Simulation and Experimental Studies

A. Aphale*[1]; A. Uddin[3]; B. Hu[1]; J. Webster[3]; S. Belko[4]; S. Heo[2]; J. Hong[2]; P. Singh[2]

1. University of Connecticut, Center for Clean Energy Engineering, USA
2. University of Connecticut, Materials Science and Engineering, USA
3. University of Connecticut, Mechanical Engineering, USA
4. University of Connecticut, Chemical Engineering, USA

Role of a number of air contaminants (both intrinsic and extrinsic) on the chemical, structural and electrochemical behavior of SOFC cathode has been studied in 550-850°C temperature range. State-of-the-art perovskite formulations show a tendency for A-site dopant exsolution in the presence of moisture while the presence of chromium and sulfur containing gaseous species lead to the deposition and coverage of electrochemically active sites. Experimental findings pertaining to electrochemical performance degradation will be presented. Thermochemical and electrochemical processes associated with chromium and sulfur assisted electrode poisoning will be discussed. Approaches for the mitigation of electrode poisoning will be outlined.

10:40 AM

(ICACC-S3-044-2018) Correlating Oxygen Electrode Degradation to Cr Vaporization from Metallic Interconnects in Solid Oxide Cell Stacks

B. Talic*[1]; P. Hendriksen[1]

1. Technical University of Denmark, Energy Conversion and Storage, Denmark

Volatile Cr species released from stainless steel interconnects in solid oxide cell stacks lead to rapid degradation of the oxygen electrode. Many studies have been devoted to elucidate the oxygen electrode degradation mechanism and methods are available to

accurately measure the Cr vaporization rate of stainless steel interconnect materials. Coating the interconnect with $MnCo_2O_4$ has been shown to greatly reduce the Cr vaporization rate, but it is difficult to determine whether this coating material is protective enough as there are no reports of a clear correlation between the interconnect Cr vaporization rate and the oxygen electrode degradation rate. The aim of this work is to investigate if such a correlation can be found. Symmetrical cell consisting of $La_{0.58}Sr_{0.4}Co_{0.2}Fe_{0.8}O_{3-\delta}$ (LSCF) oxygen electrodes screen printed on $Ce_{0.9}Gd_{0.1}O_{2-\delta}$ (CGO) electrolyte were tested at 800 °C while exposed to Crofer 22 APU alloy that was either pre-oxidized or coated with $MnCo_2O_4$. The $MnCo_2O_4$ coating was heat treated to produce different levels of porosity, resulting in different Cr vaporization rates. Degradation of the symmetrical cells was monitored by electrochemical impedance spectroscopy measurements and post-mortem SEM and EDS analysis was used to examine Cr deposition on the oxygen electrodes.

11:00 AM

(ICACC-S3-045-2018) Study of Cr-Gettering material with LSM-based cell: Solid state reaction and validation in stack fixture test

Y. Chou*[1]; J. Choi[1]; J. W. Stevenson[1]; C. Liang[2]; B. Hu[2]; W. Rodriguez[2]; A. Aphale[2]; P. Singh[2]

1. Pacific Northwest National Lab, Materials, USA
2. University of Connecticut, Center for Clean Energy, USA

Cr poisoning is currently the leading cause for solid oxide fuel cell degradation. Mitigation by protective coatings have been successfully demonstrated in short and medium term operation. Nevertheless, there is the need for Cr mitigation should the coatings start to fail during long-term operation. PNNL has teamed up with U. Conn. to evaluate a novel Cr-getter material (Sr-Ni-oxide) developed at the university. In this work, solid state reaction was pursued as a means to make the Sr-Ni-oxide powders using mixed SrO and NiO powders calcined at 600-1000°C for 2-48h. XRD was used to identify crystalline phases. Once the optimum condition was determined, Sr-Ni-oxide was formed into small rods used for validation tests. Validation of candidate Cr-gettering materials was conducted in two areas: one upstream of the cell and the other directly on the cell in a generic stack test fixture. Commercial LSM-based YSZ/NiO anode-supported YSZ cell were used with a standard sealing system and diluted fuel (H_2:N_2=1:1) and humidified (~5% H_2O) air. The upstream Cr sources were pre-oxidized AISI 441 metal stripes. For comparison, plain cells with and without Cr sources were also tested for baseline data at 800°C for ~1000h. After the test, microstructure analysis was conducted to assess the effect of upstream and on-cell Cr gettering and chemical compatibility with contact materials.

S5: Next Generation Bioceramics and Biocomposites

Bioceramics and Biocomposites II

Room: Coquina Salon B

Session Chairs: Leif Hermansson, Doxa AB; Enrico Bernardo, University of Padova

8:30 AM

(ICACC-S5-009-2018) Highly Porous Hardystonite-based Bioceramics from Preceramic Polymers and Reactive Fillers (Invited)

E. Bernardo*[1]; H. Elsayed[1]

1. University of Padova, Dipartimento di Ingegneria Industriale, Italy

Hardystonite bioceramics have been obtained by thermal treatment, in air, of silicone resins and reactive oxide fillers. Operating with commercial silicones embedding SrO and MgO precursors ($SrCO_3$ and $Mg(OH)_2$), in addition to CaO and ZnO precursors ($CaCO_3$ and ZnO powders), a quite complex solid solution ($Ca_{1.4}Sr_{0.6}Zn_{0.85}Mg_{0.15}Si_2O_7$) could be achieved, at 1100 °C, instead of pure hardystonite ($Ca_2ZnSi_2O_7$), with improvements in biocompatibility and bioactivity, as testified by cell culture tests. Highly porous components, in the form of foams and 3D reticulated scaffolds, were obtained by gas evolution at the early stage of heat treatment (at 300-350 °C), in turn due to decomposition of hydrazine additive and dehydration of $Mg(OH)_2$, or by direct ink writing of silicone-based pastes. B-containing variants could be obtained as well, using calcium borate, in both hydrated and anhydrous form, as additional multifunctional filler. In hydrated form, calcium borate led to a substantial foaming of silicone-based mixtures, by dehydration at low temperature (420 °C); in all forms, it provided a liquid phase, leading to solid solutions ($(Ca,Sr)_2(Mg,Zn)_{1-x}B_{2x}Si_{2-x}O_7$) at only 950 °C. Compared to previously developed hardystonite cellular ceramics, the newly obtained foams and scaffolds exhibit remarkable mechanical properties (compressive strength exceeding 4 MPa, with a total porosity above 67%).

8:50 AM

(ICACC-S5-010-2018) Advanced Tissue Engineering Scaffolds Incorporated with Theranostics for Cancer Cell Targeting and Photothermal Therapy

M. Wang*[1]; L. Guo[1]

1. The University of Hong Kong, Department of Mechanical Engineering, Hong Kong

Post-surgery cancer patients face high cancer recurrence rates and hence the early detection and treatment of recurring cancer are vital for them. They may also need tissue regeneration at the original tumor site. Au-based theranostics can provide diagnostic and therapeutic functions in oncology due to their unique properties, and electrospun scaffolds have many advantages for tissue regeneration. In this study, advanced theranostics-incorporated scaffolds were fabricated using concurrent electrospinning and co-axial electrospray and their performance was studied. Electrosprayed, core-shell structured PLGA50/50 microspheres containing Au-based theranostics were found evenly distributed in electrospun PLGA75/25 nanofibrous scaffolds. Experiments were conducted for theranostics release studies by immersing advanced scaffolds in simulated body fluid for different times and released theranostics were analyzed. In vitro biological experiments were performed using HeLa cells to investigate the cancer cell targeting ability and photothermal therapy of released theranostics. Also, both HeLa cells and MCF-7 cells (the control) were incubated separately with sterilized scaffolds. Results showed that the advanced scaffolds could release theranostics in a controlled manner and released theranostics could provide cancer cell detection and photothermal therapy functions.

9:10 AM

(ICACC-S5-011-2018) Additive Manufacturing and Restoration Possibilities within the Dental area Using Chemically Bonded Ceramics (Invited)

L. Hermansson*[1]

1. Applied Research Sweden AB, Sweden

This paper relates to dental implants and restoration materials in the CASPH-system (CaO-Al_2O_3-SiO_2-P_2O_5-H_2O) especially wherein Additive Manufacturing including CAD/CAM or similar technologies, are used for machining dense blanks into a close correspondence with the cavity geometry. The same basic system, the CASPH-system can also be used as a cement for fixation of the blanks to the surrounding tooth tissue. In this way minimal tension between the biomaterial and the biological tissue is established. The use of the chemically bonded ceramic cement facilitates also the filling of a dental cavity with a waist, where the cement also works as a base. The dense blanks facilitate improved mechanical properties including wear resistance, as well as improved optical properties including transparency of the dental cavity.

9:30 AM

(ICACC-S5-012-2018) Structure / property relationships in Biomaterials at the nanoscale (Invited)

F. Rosei*[1]

1. INRS, Canada

Nanostructuring materials allows to optimize their properties. We created nanopatterns that act as surface cues, affecting cell behavior. Chemical oxidation creates nanotopographies, that allow to improve biocompatibility. Our treatment selectively inhibits fibroblast proliferation while promoting osteoblast growth in vitro. Enhancing mechano-biocompatibility may occur by coating with spider silk. Improving antibacterial properties using laser/plasma strategies will also be discussed.

10:10 AM

(ICACC-S5-013-2018) Cancer Cell Targeting and Ablation by Surface-Electrically-Charged Superparamagnetic Fe_3O_4 Composite Nanoparticles (Invited)

D. Shi*[1]

1. University of Cincinnati, Mechanical and Materials Engineering, USA

Current progress on cancer cell targeting and therapeutics will be reviewed in terms of nanomaterials development with special functionalities. In particular, a new research direction will be introduced on biomarkerless cell targeting via surface-charged nanoparticles. Cancer cells can be well targeted by using positively-charged, fluorescent, superparamagnetic Fe_3O_4-nanocomposites. The positively charged Fe_3O_4 nanocomposites bind predominantly onto cancer cells due to their negatively charged surfaces. The negatively charged nanocomposites are found, however, not to target and bind the cancer cells due to the electrostatic repulsive force between them. The negative charges on cancer surfaces are found to be originated from the cancer cell lactate secretion regulated by glycolysis. An 808 nm laser is subsequently applied to induce a photothermal hyperthermia that results in cancer cell ablation effectively. Also presented is the fundamental bio- physics and chemistry that dictate the lactate-secretion-mediated surface negative charges on the cancer cells, whose metabolic characteristics are fundamentally different from the normal cells.

10:30 AM

(ICACC-S5-014-2018) How does nanoporosity affect cell response of bioactive glass? (Invited)

U. Thamma[1]; T. Kowal[3]; M. Falk[3]; H. Jain*[2]

1. Lehigh University, International Materials Institute for New Functionality in Glass, USA
2. Lehigh University, Materials Science and Engineering, USA
3. Lehigh University, Biological Sciences, USA

In solids with a given fraction of interconnected porosity, the smaller is the pore size, larger is the exposed surface area. Accordingly, control of nanopore size and its distribution can be an effective way for designing the biodegradation rate of porous bioactive glasses and ceramics. Then the question arises: can nanopores also affect a cell's biological response irrespective of degradation rate? To answer this question we have fabricated nanoporous samples of bioactive $30CaO\text{-}70SiO_2$ glass with varying pore size but the same surface area. The results show highest attachment of MC3T3 pre-osteoblast cells on samples with ~15 nm pores, with lower values for larger or smaller pore size. This is particularly intriguing since the cells do not come in contact with glass substrate, but interact only through a complex interfacial layer comprising of modified glass, hydroxyapatite and proteins. So to understand the impact of nanopore size on the response of cells, we have characterized the bioglass-culture medium interface. Specifically, the microstructure of hydroxyapatite and conformation of a model protein in relation to nanopore size have been determined. The results show best correlation between the number density of cells and secondary conformation of protein. Implications of these results are discussed in order to answer the title question. This work is supported by National Science Foundation (IIP-1602057).

10:50 AM

(ICACC-S5-015-2018) Preparation and characterization of bio-inspired hybrid containing calcium phosphate for the environmental applications (Invited)

M. Tafu*[1]; T. Toshima[1]

1. National Institute of Technology, Toyama College, Japan

Calcium phosphate, such as hydroxyapatite (HAp) and Dicalcium phosphate dihydrate (DCPD) have good characteristics for catching heavy metal ions and fluoride ions. We have investigated utilization of DCPD-HAp nano-hybrid for immobilization of small amounts of fluoride ions in the environments. HAp and fluoraptite (FAp) in human body is high performance materials that is well-controlled crystallinity and/or crystal size, and hybridized with organic compound in nano-scale. We have interested in nano-hybridized structure of bone. We investigated effect of carbonized phase on reactivity of fluoride ions in an aqueous ion. Fluoride removal capacity was affected by not only crystallinity of HAp but also characteristics of carbonized phase. We appeared that the petal-shaped DCPD have good powder property for industrial applications and easily obtained from aqueous solution under controlled super saturation value. Stable FAp was obtained by reaction with DCPD-HAp hybrid and fluoride ions. The obtained FAp have similar particle morphology than starting DCPD particles. These results suggest that controlling morphology of DCPD particles is seems to improve efficiency of water treatment containing fluoride. In this presentation, we summarize recently achievements for preparation and characterization of bio-inspired nano-hybrid containing calcium phosphates.

11:10 AM

(ICACC-S5-016-2018) Biocompatible magnetic hydroxyapatite nanoceramics for hyperthermia treatment: Fabrication and characterization

S. Iqbal*[1]; T. Jamil[1]; A. Sabir[1]; A. Islam[1]; S. Maqsood Khan[1]

1. University of the Punjab, Lahore, Pakistan, department of Polymer Engineering & Technology, Pakistan

This study reported the synthesis of $Y_{0.2}La_xMg_{0.8-x}Fe_2O_4$ doped hydroxyapatites bioceramics having hyperthermia treatment. Sol-gel method was used to prepare the nanoferrites cosubstituted hydroxyapatites along with freeze-drying followed by heat treatment yielded design biocompatible magnetic biomedical nanoceramics. The yield was characterized by X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), and Scanning electron microscope along with composition analysis (SEM/EDS). The vibrating sample magnetometer was used to analyze magnetic behavior and cell culture was performed to check biocompatibility. To cell metabolic activity was assessed with MTT assay. This research concluded that the $Y_{0.2}La_xMg_{0.8-x}Fe_2O_4$-HA nanocomposite produced no negative effects on cell morphology, viability, proliferation and exhibited remarkable biocompatibility for hyperthermia treatment.

S6: Advanced Materials and Technologies for Direct Thermal Energy Conversion and Rechargeable Energy Storage

Thermoelectrics III

Room: Tomoka A
Session Chair: Jon Goldsby, NASA Glenn Research Center

8:30 AM

(ICACC-S6-038-2018) Defect-Induced Thermoelectricity and Phonon Scattering in Oxides (Invited)

S. Lee*[1]; J. U. Rahman[1]; W. Seo[1]; M. Kim[2]

1. Korea Institute of Ceramic Engineering and Technology (KICET), Energy & Environmental Materials Division, Republic of Korea
2. Changwon National University, School of Advanced Materials Engineering, Republic of Korea

Low temperature thermoelectric materials have been commercializing in various applications, while mid-high temperature materials have not been widely utilized yet due to high temperature instability and thereby reliability issues, and lack of modularization technology. Conductive oxide materials could be one of candidate thermoelectric materials in terms of low cost, high temperature stability, reversible reaction, and so on. There is lack of studies on the oxide materials with high concentration of defects and/or electronic charge carriers. For thermoelectric oxides, it needs to improve the figure of merit, especially, by enhancing their charge transport and lowering lattice thermal conductivity, and to study on new materials and alternative ways to control the carrier concentration and charge transport properties. In this study, we modified the stoichiometry of cation and oxygen atoms to control the carrier concentration and investigated the nonstoichiometry effects on their thermoelectric properties. Both cation and oxygen vacancies act as a donor of charge carriers and an optic and/or acoustic phonon scattering centers depending on defect type and concentration. The coupling and/or decoupling between defect and property in oxides, such as $SrTiO_3$ and $LiNbO_2$, are discussed in terms of thermoelectric properties.

9:00 AM

(ICACC-S6-039-2018) Mixed-Metal Sulphides for Thermoelectric Energy Conversion (Invited)

A. V. Powell*[1]; P. Mangelis[1]; S. Long[1]; P. Vaqueiro[1]

1. University of Reading, Chemsitry, United Kingdom

Thermoelectric devices convert thermal energy directly into electrical energy, leading to efficiency gains and reductions in fuel consumption. The interdependence of the Seebeck coefficient (S), electrical conductivity (s) and thermal conductivity (k) that define the figure-of-merit, $ZT = S^2sT/k$, presents a challenge in creating high-performance materials. A number of design strategies have emerged that seek to achieve a degree of separation between the electrical (S^2s) and thermal (k) contributions to ZT. A significant fraction of waste heat from industrial processes is released at temperatures between 373 and 535 K. Metal sulphides are attractive candidates for thermoelectric energy recovery in this region. Our recent work on chemical substitution to enhance the properties of mixed-metal sulphides will be presented. The competition between electron transfer and structural disorder on intercalation into Co_xTiS_2 will be described, together with the improvements in performance achieved by tuning the Fermi level in synthetic analogues of shandite, $Co_3In_2S_2$. Phonon-liquid electron-crystal (PLEC) behaviour in synthetic analogues of tetrahedrite ($Cu_{12}Sb_4S_{13}$) and bornite (Cu_5FeS_4) leads to figures of merit in excess of ZT = 0.5 at 573 K, suggesting that PLEC materials derived from minerals are promising candidates for energy recovery at low to intermediate temperatures.

9:30 AM

(ICACC-S6-040-2018) Rapid Synthesis and Processing of Tetrahedrite-Based Thermoelectrics for Large-Scale Power Generation Applications (Invited)

D. T. Morelli*[1]; D. Weller[1]; W. Lai[1]; J. Li[1]; M. E. Anderson[2]; G. E. Kunkel[2]; A. Ochs[2]

1. Michigan State University, Chemical Engineering & Materials Science, USA
2. Hope College, Chemistry, USA

Tetrahedrite ($Cu_{12}Sb_4S_{13}$)-based compounds demonstrate good thermoelectric properties while also being composed of earth-abundant, non-toxic elements. These compounds exhibit unusual structural, bonding, and lattice-dynamical characteristics that give rise to phonon-glass-like behavior, low intrinsic lattice thermal conductivity, and high thermoelectric figure of merit. Successful p-type doping can be achieved by substitution of nickel or zinc for copper. Successful synthesis of pure phase tetrahedrite using standard sealed-tube synthesis, however, requires multiple days or weeks. Here we present an overview of several synthesis and processing approaches that entail significantly less time than the conventional furnace-ampoule method. Mechanical alloying, spark plasma sintering (SPS) solid-state reaction, and modified polyol solution-phase methods can all result in successful and facile synthesis of both pure and p-type doped specimens. The resulting figures of merit are comparable to those obtained using conventional solid-state synthesis. When combined with the low cost of starting materials, these rapid synthesis/processing techniques can open the door to widespread use of tetrahedrite-based thermoelectrics for waste heat recovery and power generation applications.

Thermoelectrics IV / Materials for Solar-thermal Applications

Room: Tomoka A
Session Chair: Emmanuel Guilmeau, CNRS CRISMAT

10:20 AM

(ICACC-S6-041-2018) Skutterudite-Based Thermoelectric Technology for Integration into a Potential eMMRTG for Space Power Applications (Invited)

T. Caillat*[1]; I. Chi[1]; S. Firdosy[1]; C. K. Huang[1]; K. Smith[1]; J. Paik[1]; P. Gogna[1]; K. Yu[1]; J. Fleurial[1]; R. Bennett[2]; S. Keyser[2]

1. NASA Jet Propulsion Laboratory, USA
2. Teledyne Energy Systems, Inc., USA

The overall objective of the Skutterudite Technology Maturation (STM) project at NASA's Jet Propulsion Laboratory (JPL) is to advance JPL-developed skutterudite (SKD) technology to a point where it can be considered for use in a potential enhanced Multi-Mission Radioisotope Thermoelectric Generator (eMMRTG). The goal is to be prepared for flight unit development readiness by end of FY2018, should NASA Headquarters decide to proceed. Conversion efficiency values on the order of 9% have been demonstrated for SKD-based un-segmented couples when operating at a hot junction of 600C and a cold junction of 200C. This represents ~ a 25% improvement over the conversion efficiency of PbTe/TAGS MMRTG couples at beginning-of-life (BOL). The STM project entered its third year at the beginning of FY17. During the first two years of the project, JPL and Teledyne Energy Systems Inc. (TESI) have collaborated to transfer the technology to TESI, to develop the manufacturing capabilities for SKD TE materials and couples at TESI, and to demonstrate their initial performance and initiate a lifetime performance prediction. Significant progress has been made towards those goals, and this paper will summarize this progress as well as the remaining challenges to fully mature this technology.

10:50 AM

(ICACC-S6-042-2018) Material design and module construction in nanostructured PbTe- and Colusite-based thermoelectrics (Invited)

M. Ohta*[1]; P. Jood[1]; K. Suekuni[2]; T. Takabatake[3]; M. G. Kanatzidis[4]; A. Yamamoto[1]

1. National Institute of Advanced Industrial Science & Technology, Japan
2. Kyushu University, Department of Applied Science for Electronics and Materials, Interdisciplinary Graduate School of Engineering Sciences, Japan
3. Hiroshima University, Department of Quantum Matter, ADSM, Japan
4. Department of Chemistry, Northwestern University, Evanston, IL, United States and Materials Science Division, Argonne National Laboratory, USA

This talk describes our recent efforts to fabricate high-efficiency modules using newly-developed thermoelectric materials based on nanostructured PbTe and colusites $Cu_{26}A_2E_6S_{32}$ (A: V, Nb, Ta; E: Ge, Sn). We have demonstrated high thermoelectric figure of merit ZT in these materials and corresponding high conversion efficiency in the modules. For PbTe modules, we used the sintered compacts of nanostructured PbTe–0.7% Ge–4% Na with an exceptionally high ZT ~ 1.9 at 820 K for p-type legs and PbTe–0.2% PbI_2 with a high ZT ~ 1.3 at 780 K for n-type legs. We fabricated the nanostructured PbTe-based and cascaded Bi_2Te_3/nanostructured PbTe modules. The maximum conversion efficiency of ~8.5% and ~12% for a temperature difference of 590 K are achieved in the former and latter modules, respectively. The colusites were prepared by melting stoichiometric amounts of the constituent elements in evacuated and sealed quartz tubes and then sintered with a diffusion barrier. Among $Cu_{26}A_2E_6S_{32}$, the ZT of $Cu_{26}Nb_2Ge_6S_{32}$ reaches highest value of ~1.0 at 670 K. The Au-based diffusion barrier between colusites and electrodes provides the reduced electrical and thermal contact resistances, leading to enhanced conversion efficiency. This work is supported by NEDO and by METI.

11:20 AM

(ICACC-S6-044-2018) Design and Discovery of Mixed Metal Oxides for Solar Thermochemical Water Splitting

S. Millican*[1]; I. Androshchuk[1]; C. B. Musgrave[1]; A. W. Weimer[1]

1. University of Colorado, Department of Chemical and Biological Engineering, USA

While solar energy is the most abundant renewable energy resource, the capture, storage, and distribution of it remains a challenge. Solar thermochemical water splitting (STWS) provides a promising route for efficient utilization of this disperse resource since it allows for use of the entire solar spectrum to convert water to an energy dense fuel, H_2. However, despite a significant number of materials having been examined, an optimal redox material to drive this process has yet to be developed. In order to be viable for economic hydrogen production, materials must have high hydrogen productivity, fast reduction and oxidation kinetics, low thermal reduction temperatures, and long term stability and reactivity. In this work we utilize ab initio methods to assess the thermodynamic viability of a large number of spinel and perovskite metal oxides for STWS. Materials are screened for stability, oxygen vacancy formation energy, and extent of reduction. The effect of temperature on the parameters including crystal structure, cation disorder, and magnetic order and their relationship to predicted STWS ability are discussed. Compositional (doping) control is utilized to optimize materials' thermodynamic properties. Experimental results from redox cycling in a stagnation flow reactor and TGA are presented and compared to DFT models.

11:40 AM

(ICACC-S6-045-2018) On-Sun Demonstration of Solar Thermal Water Splitting for H_2 Production

A. Hoskins*[1]; S. Millican[1]; C. Czernik[1]; I. Alshankiti[1]; J. Netter[2]; C. B. Musgrave[1]; A. W. Weimer[1]

1. University of Colorado Boulder, Chemical Engineering, USA
2. National Renewable Energy Laboratory, USA

The use of solar radiation to renewably generate energy has long been recognized by the scientific community as a viable option for one pillar of a diversified energy platform. As hydrogen fuel cell and combustion technology have become increasingly viable the development of methods to efficiently and inexpensively produce hydrogen have become increasingly important. Solar thermochemical water splitting (STWS) has been proposed as means of hydrogen production which has the benefit that both the energy source and the fuel are renewable and readily available. The implementation of STWS reaction schemes in an economically viable process is extremely demanding. In order to commercially realize this technology, several challenges must be overcome including an energy efficient reactor design, stable reactor and heat exchanger materials, and robust redox active particles. Among these challenges material based limitations are the most abundant. In this work we present results showing on-sun hydrogen production utilizing dual lab-scale fluidized beds constructed of SiC modified to be steam resistant. Active hercynite particles were redox cycled at the High Flux Solar Furnace at the National Renewable Energy Laboratory and production rates of H_2 exceeding 500 umol/g were measured. The effect of environmental variability on the incident solar radiation and hydrogen production is evaluated during on-sun testing.

S7: 12th International Symposium on Functional Nanomaterials and Thin Films for Sustainable Energy Harvesting, Environmental, and Health Applications

Synthesis, Functionalization and Assembly of 1D, 2D and 3D Nanostructures II

Room: Coquina Salon C
Session Chairs: Monica Ferraris, Politecnico di Torino; Yung-Jung Hsu, National Chiao Tung University

8:30 AM

(ICACC-S7-042-2018) Energetics and Structure Relations of Low-k Amorphous SiOCH Dielectric Films (Invited)

J. Chen*[1]; S. King[2]; J. Calvin[3]; B. Woodfield[3]; A. Navrotsky[1]

1. University of California, Davis, Department of Materials Science, Peter A. Rock Thermochemistry Laboratory and NEAT ORU, USA
2. Intel Corporation, Logic Technology Development, USA
3. Brigham Young University, Department of Chemistry, USA

With the size of integrated circuit getting smaller, it is crucial to find material with low-dielectric constant to decrease the resistance capacitance delay time in the backend of the line. New generation dielectric materials, such as a-SiCH, a-SiOCH, synthesized from plasma-enhanced CVD, have now been widely used as substitutes for traditional SiO_2. While these materials exhibit low dielectric constant, their application is hindered by the propensity to decompose and lose hydrogen during nanofabrication and thermal annealing. This raises the question of their ultimate thermodynamic stability, thus it is necessary to get a full picture of their thermodynamic properties, including enthalpy and entropy of formation. We have determined the formation enthalpies at 25°C both from elements and their corresponding crystalline constituents and gaseous products by high temperature oxide melt solution calorimetry. Using these and newly measured low temperature heat

capacities, the formation entropies and Gibbs free energies of formation at 25°C are computed. The results show that these amorphous materials are thermodynamically more stable than their crystalline constituents and gaseous products at 25°C but such stability decreases with increasing temperature. Based on thermodynamic and structural analysis, their thermodynamic stability is related to structural difference, SiO density, and Si-O to Si-CH_x ratio.

9:00 AM

(ICACC-S7-043-2018) Charge storage in 2-D oxide nanosheet assemblies: 3-D assembly and conversion to tunnel structures

S. T. Misture*[1]; T. Hey[1]; P. Metz[1]

1. Alfred University, MSE, USA

Nanosheets derived from bulk layered oxides are promising for use as supercapacitor and battery electrodes, as well as for catalysis and sorption applications. In an effort to design low-density solids with hierarchical porosity, we chemomechanically exfoliate crystalline powders to form nanosheet suspensions that can be flocculated through the addition of acids and salts. Homogeneous flocculation or gelation of the nanosheets yields thin-walled macroporous solids with surface areas exceeding ~250 square meters per gram. Intersheet ion exchange with mono-, di- and trivalent cations is possible, and controlled cation introduction with subsequent optimized heat treatments yields novel 3-D tunnel-structure nanostructures. We demonstrate the use of 6 different intercalation cations and show that low levels of alkali are most useful to nucleate the topochemical conversions from layer to tunnel structures, with complete conversion possible. The structural reaction mechanism was determined using high energy X-ray scattering pair distribution function analysis.

9:20 AM

(ICACC-S7-044-2018) 3D piezoelectric nanocrystals structure for detection of ultra-small mechanical pressure

H. Bishara*[1]; S. Berger[1]

1. Technion - Israel Institute of Technology, Materials Science and Engineering, Israel

Detection of ultra-small mechanical pressures in an atmospheric environment is of a high technological interest in various applications such as a medical diagnosis and a gas leakage. In this research we report on detection of ultra-small mechanical pressure in an atmospheric environment, using low-k ceramic piezoelectric nanocrystals embedded inside alumina pores. This concept is proved for several materials: Sodium Nitrite, Lithium Sulfate and Potassium Iodate, grown from liquid solution. These materials are rarely utilized in piezoelectric devices due to their poor mechanical properties; still we could overcome this obstacle. The magnitude and sensitivity of the piezoelectric response is determined by the material and its preferred crystallographic orientation along the axis of pores. Moreover, polarization mechanisms of the materials could be studied through the piezoelectric measurements. Periodic mechanical pressures in the range of 1-10 Pa (corresponding to mechanical forces of 20-200 microNewton) were applied on the samples by a controlled air flow. The talk will include a detailed description of the preparation methods of the samples, their microstructure and piezoelectric characteristics; and a discussion on the advantages of utilizing such 3D structures of the studied nanocrystals inside a porous alumina in improving the ability to detect ultra-small applied forces.

9:40 AM

(ICACC-S7-045-2018) Serial Z-scheme Heterostructures for Photoelectrochemical Water Reduction

c. Tsao*[1]

1. Material Science and Engineering of National Chiao Tung University, Taiwan

As inspired by the natural photosynthesis, Z-scheme charge transfer mechanism has recived significant attention for various photoconversion applications. Here we devised a serial Z-scheme heterostructures based on CuO-Au-Cu_2O-Au-NiO composite films which showed incremental Z-scheme mechanism for efficient photoelectrochemcial water reduction. The samples were prepared by sequentially depositing Au, Cu2O, Au and NiO on the electrochemically grown CuO films. For CuO-Au-Cu_2O-Au-NiO, the embedded Au can mediate the interfacial charge transfer by facilitating the interband electron/hole recombination from the two different pairs of CuO→Cu2O and Cu2O→NiO, which gives rise to hole accumulation at CuO and electron concentration at NiO. With the substantially high reduction powers (ECB= -3.06 V vs. NHE for NiO) and much enhanced charge separation, the serial Z-scheme CuO-Au-Cu_2O-Au-NiO performed much better as photocathode in photoelectrochemical water reduction than the single Z-scheme samples did.

Nanomaterials for Photocatalysis, Solar Hydrogen and Thermoelectrics IV

Room: Coquina Salon C

Session Chairs: Scott Misture, Alfred University; Jiewei Chen, University of California, Davis

10:20 AM

(ICACC-S7-046-2018) Nanostructured antibacterial coatings obtained by co-sputtering of silver and silica (Invited)

M. Ferraris*[1]; F. Baino[1]; C. Balagna[1]; S. Ferraris[1]; M. Irfan[1]; M. Miola[1]; S. Perero[1]; E. Verne*[1]; S. Spriano[1]

1. Politecnico di Torino, Department of Applied Science and Technology, Italy

Nanotechnology has brought great advancements in modifying textile surfaces and deposit thin functional films for various applications including energy harvesting, environmental remediation, flexible electronics and health care. Textiles with antibacterial surfaces are of immense importance especially in health care, extreme conditions (defense applications), closed environments (aerospace applications) and textile based filters. Silver is a widely known antibacterial agent with a broad-spectrum activity against numerous bacterial and fungal strains and it overcomes the increasing problem of bacterial resistance to antibiotics, following the recent European strategy to reduce the antibiotic-based therapies. Thin composite coating developed by our group, made of silver nano clusters embedded in silica matrix via co-sputtering has excellent properties against different microbes while maintaining and preserving typical textile properties. The employed technique results in a coating with controlled silver ion release, reduced direct skin exposure to silver, increased wettability and preserved air permeability of the textiles. The coating is fully inorganic and has a high thermal resistance (up to 450 °C maintaining antibacterial activity), a high scratch resistance, an optimal adhesion to various substrates and can be sterilized by conventional techniques employed for medical devices.

10:50 AM

(ICACC-S7-047-2018) Is TiO_2 brookite a good photocatalyst for degradation of pollutants in water and in air?

S. Cassaignon*[1]; O. Durupthy[1]; C. Guillard[3]; C. Colbeau-Justin[2]

1. Sorbonne University, UPMC LCMCP, France
2. Université Paris-Sud - Université Paris-Saclay, LCP, France
3. Université Lyon 1, IRCELYON, France

Heterogeneous photocatalysis is an attractive approach for the removal of organic pollutants in air and water. However, after nearly 40 years of extensive work on photocatalysis using different chalcogenides (oxides and sulfides) and mainly TiO2, we are not able to explain why this compound is the most efficient and the differences in activity between the various polymorphs of titania, anatase, rutile and brookite. These points constitute an important technological lock for improving its efficiency. Our researches were focus on the elaboration of very well defined morphology of the three phases, their physicochemical and optical characterizations (XRD, surface area, electronic microscopy,...) but also their electronic properties using Time Resolved Microwave Conductivity (TRMC) and finally their photocatalytic properties (in water and in air) using several model pollutants. This presentation will focus on the very interesting Brookite phase.

S9: Porous Ceramics: Novel Developments and Applications

High SSA Ceramics and Membranes

Room: Coquina Salon G

Session Chair: Gian-Domenico Soraru, University of Trento

8:30 AM

(ICACC-S9-019-2018) Rational design of sol-gel derived multifunctional ceramic membranes (Invited)

A. Ayral*[1]

1. University of Montpellier, European Institute of Membranes, France

Nanoporous ceramic membranes play a key role in an increasing number of processes and operations of separation, capture, conversion applied to process intensification and environment protection. Advances in engineering of ceramic materials enable to have access to functional or multifunctional membranes and systems meeting complex specifications for sophisticated applications. For tailoring and optimizing their functional properties, in particular their permeability (flux) and their selectivity (separation efficiency), different strategies can be implemented related to the selection of the most suitable materials for managing the solid-fluid interactions in confined media, to the design of the membrane porosity and also to the design of the membrane structure and geometry. Several examples of such multiscale and rationalized approach will be presented including the case of catalytic membranes with hierarchically-porous and catalytically-active separative layers.

9:00 AM

(ICACC-S9-020-2018) Facile Fabrication of Metal–organic Frameworks (MOFs) Membranes on Ni Foam Substrates for Gas Separation (Invited)

Z. Nie*[1]

1. Beijing University of Technology, College of Materials Science and Engineering, China

Metal–organic frameworks (MOFs) are expected to have great potentials in membrane fabrication and application in gas separation due to their regular crystalline lattices with relatively well-defined pore structures. A facile strategy has been developed for the fabrication of MOF membranes(HKUST-1 and $M_3(HCOO)_6$, M=Co, Mn or Mg) anchored on Ni foams with oriented Ni_3S_2 and Co_3O_4 nano–microstructure arrays on the Ni foams as both nucleation centers and anchor bars. The oriented nano–microstructure arrays are confirmed to control the directional growth of MOF crystals in forming the membrane. The obtained MOFs membranes show a good gas separation performance, especially for the H_2/CO_2 mixture. A facile aggregation-based growth method has also been explored to prepare a Co-MOF-74 membrane on a nanostructure array modified nickel (Ni) foam substrate. Ni_3S_2 nanorod arrays directly grown from the Ni-foam substrate can aggregate and effectively regulate the growth of Co-MOF-74 crystals along a given direction. The resulting Co-MOF-74 membrane presents a high H_2 permeance and H_2/CO_2 selectivity. The use of Ni foam as the porous substrate is beneficial to reduce to the cost of resulting membranes.

9:30 AM

(ICACC-S9-021-2018) Structuring of Microporous Ceramics at Various Length Scales (Invited)

F. Akhtar*[1]

1. Division of Materials Science, Department of Engineering Sciences and Mathematics, Sweden

Microporous adsorbents, zeolites, activated carbons and aluminum phosphates, are high surface area materials with great potential for catalysis and gas separation applications in structured form such as monoliths and laminates. In this regard, recent developments on tailoring of macro-meso and micropores in structured adsorbents using binderless processing of porous adsorbent powders will be presented to produce volumetrically efficient hierarchically porous structured adsorbents with properties to overcome the limitations of conventional adsorbent materials in gas separation applications. Moreover, processing of self-standing zeolite laminates without addition of binders will be discussed with a prospective to enhance diffusion of gas molecules to develop rapid pressure/vacuum swing adsorption technologies for separation and purification of gases. A new approach, nanostructurization of microporous materials, will show that nanostructurized zeolites are effective for economical post-combustion decarbonization of gas streams, including carbon dioxide uptake capacity, carbon dioxide selectivity over nitrogen, and rapid carbon dioxide uptake and release kinetics.

Properties of Porous Ceramics

Room: Coquina Salon G

Session Chair: Manabu Fukushima, National Institute of Advanced Industrial Science and Technology (AIST)

10:20 AM

(ICACC-S9-022-2018) Optical properties of mesoporous silica layer on periodic array of plasmonic nanocylinders (Invited)

S. Murai*[1]; H. Sakamoto[1]; K. Fujita[1]; K. Tanaka[1]

1. Kyoto University, Japan

Mesoporous silica layers with open and accessible mesopores were fabricated on aluminum nanoparticle arrays. The system can support plasmonic-photonic hybrid modes that are radiatively coupled surface plasmon polaritons in the nanoparticles. The coupling is mediated either by diffraction in the plane of the array or by waveguiding in the dielectric silica layer covering the array. Upon irradiation of an array with visible light, these hybrid modes are excited and appear as sharp spectral dips in optical transmission, a manifestation of light confinement in the system. The porous nature of the layers can be taken advantage of, via pore infiltration, to facilitate control of optical properties over the layer's refractive index. We demonstrated tuning in the wavelength and spatial distribution of confined light in the system.

10:50 AM

(ICACC-S9-023-2018) An advanced method to measure the gas permeability in porous ceramics

J. Kadok*[1]; E. de Bilbao[1]; S. Brassamin[1]; J. Poirier[1]

1. CNRS, Conditions Extrêmes et Matériaux : Hautes Températures et Irradiation, France

The transport properties are important parameters that take place in porous ceramics. This is the case of the permeability which plays a key role in refractory materials used in the field of metallurgy. Permeability to gas is involved for example in the dewatering of refractory castable or in the oxygen pick up by submerged nozzle during the steel continuous casting. Permeability to liquid is also implicated in the resistance to the penetration of corrosive fluids like slags. The ISO 8841:1991, DIN EN 993-4, and ASTM C577-7 standards are reliable methods for the measurement of the gas permeability in a Darcian regime. However, refractory materials exhibit very heterogeneous connected porosity engendering non-linear effects when it comes to measure their permeability and this even at low pressures. A dedicated permeameter has been designed to perform accurate permeability measurements, taking into account non-linear effects. The pressure drop and the flow rate can be controlled to perform test in viscous conditions and active Klinkenberg's effects. Liquid permeability can therefore be also derived from gas permeability measurements. Experiments were carried out on different industrial refractory materials and results are compared with standard measurements. The impact of the corrosion by slags on the permeability of low cement castables will be also presented.

11:10 AM

(ICACC-S9-024-2018) Property – porosity relations of cellular biogenic and bioinspired ceramics

K. G. Nickel[1]; K. Klang*[1]; G. Buck[1]; M. Loeber[1]; C. Lauer[1]; C. Berthold[1]

1. University Tuebingen, Applied Mineralogy, Germany

Highly porous (Φ up to 0.9) natural cellular calcium carbonate materials from the spines of lance and slate sea urchins are characterized by low elastic moduli but relative high values for compressive strength. Mechanical properties including the energy dissipation were determined by uniaxial compression of spine segments and pin indentation. Porosities were characterized by gravimetry (mean porosities), image analysis of infiltrated segments (local porosities) and computer tomography (check for accuracy). The mechanical behavior of the natural materials is compared to synthetic bioinspired materials of comparable porosity made from Alumina and/or Zirconia manufactured via ionotropic gelation and/or freeze casting. Both biogenic and bioinspired materials are mainly open-cell networks with channel-like porosities and hence highly anisotropic in terms of pore shape. Sea urchin spine structures are graded and/or layered in their porosities, introducing another complexity. We will discuss the systematics of the relations between elastic moduli and porosity, strength and porosity and elastic moduli and strength in the light of relations taking into account pore shape, size (general power law mixing rule with Eshelby-Wu coefficients of Pabst (J.Europ.Ceram.Soc 34 (2014)) and gradation.

11:30 AM

(ICACC-S9-025-2018) Preliminary studies on adsorption of hydrogen in polymer derived mesoporous Si-O-C ceramics

S. Ravindran*[1]; P. K. Chauhan[1]; P. Rajagopalan[1]

1. Birla Institute of Technology & Science Pilani, Hyderabad Campus, Mechanical Engineering, India

Present work deals with adsorption of hydrogen on hierarchically aligned mesoporous Si-O-C ceramics. Synthesis of the ceramic is to be carried out through catalyst assisted cross linking (curing, platinum) followed by pyrolysis of the cured sample at 1400°C for ensuring crystallinity and free carbon content in the porous ceramic. Crystallinity and free carbon content were observed and established through XRD & Raman spectroscopy respectively. Pore morphology in the ceramic was established through scanning electron microscopy (SEM). The structure of the pore & quantification of the specific surface area (SSA) was carried out through BET analyser. Use of Sievert apparatus for studying hydrogen adsorption on the surface, variables being pressure, temperature and flow rate of hydrogen etc. Based on the observations from the adsorption studies and experiments conducted, we establish the energetically favoured binding sites on the surface for adsorption. We also analyse the significance of aligned pore volume in the ceramic. Further, quantification of hydrogen adsorbed through physisorption & chemisorption in agreement with various adsorption isotherms to be carried out along with the variation of surface energy of the pore with regards to free carbon content. The work also aims to establish the desorption condition for all the cases considered.

S10: Virtual Materials (Computational) Design and Ceramic Genome

Modeling of Structure and Property III

Room: Coquina Salon F

Session Chair: Jingyang Wang, Shenyang National Laboratory for Materials Science, Institute of Metal Research, Chinese Academy of Sciences

8:30 AM

(ICACC-S10-008-2018) A multiscale numerical software for designing cellular ceramics with prescribed thermal radiative properties up to very high temperatures (Invited)

B. Rousseau*[2]; A. Biallais[1]; J. Vicente[3]

1. SATT Ouest Valorisation, France
2. LTeN UMR CNRS 6607, France
3. IUSTI UMR CNRS 7343, France

Knowledge of thermal radiative properties (TRPs) of cellular ceramics are mandatory to determine the energy balance of high-temperature engineering systems in which they are involved. Experimental setup being able to provide such properties in operating environment (high temperature up to 2000K, corrosive or spatial atmosphere) are rather uncommon so that numerical strategies based on multiscale modelling are more and more favored. In this invited talk, a homemade software named genMat, (C++, Qt) aiming at computing the TRPs of any cellular ceramics will be described. genMat allows the calculation, from 3D reconstructed images with controlled textural features, of the spectro-directional dependence of macroscopic TRPs (reflectance, transmittance, emittance) as well as the quantities (absorption and scattering coefficient, scattering phase function) useful for the 3D resolution of the Radiative Transfer Equation. A Monte Carlo algorithm ensures the propagation of photons at the mesoscopic scale by taking account the spectral dependence of the complex refractive index of each solid phases composing the porous material. The versatility of genMat is for example useful for designing virtual cellular ceramics with gradual topological disorder or to anticipate the evolution of TRPs when corrosion phenomena occur at very high temperatures.

9:00 AM

(ICACC-S10-009-2018) Effect of Transition Metal Impurities on Electronic Structure of Zirconium Carbide

Y. Zhou*[1]

1. Missouri University of Science & Technology, Materials Science and Engineering, USA

Zirconium carbide has high hardness (up to about 30 GPa) and high melting temperature (>3800°C). Based on these properties,

ZrC_x ceramics are a promising materials for applications in extreme environments such as those associated with nuclear fission and hypersonic flight. Previous studies have characterized thermal and mechanical properties, but the effects of transitional metal impurities have not been studied extensively. For the present work, atomistic simulations based on density functional theory will be used to systematically investigate the bonding and electronic structure of ZrC_x. The effects of transition metal impurities on the bonding and electronic structure will be studied. In particular the effect of Hf content will be examined based on the presence of Hf in Zr-based materials. Changes in bonding and electronic structure with Hf content and other transition metal impurities such as Y, Ti, Nb, and W will be discussed.

9:20 AM

(ICACC-S10-010-2018) Influence of Random Packing Density on Sintering Kinetics

E. Hernandez*[1]; M. C. Golt[2]; B. McWilliams[2]

1. ORISE @ ARL, USA
2. US Army Research Laboratory, USA

Sintering is an essential processing step in manufacturing of powder-based materials. Understanding the kinetic evolution of sintered materials is essential in the manufacturing community. A recently developed kinetic Monte Carlo developed by Tikare et al. enables the simulation of sintering by modeling Ashby's six diffusion paths. The model developed by Tikare et al. requires user input of several kinetic and thermodynamic parameters. To understand their influence on sintering kinetics, a parametric study is performed. Then, after determining optimal sintering conditions, sintering of several randomly packed configurations are performed. Packing density, among other configurational variables, will be studied.

9:40 AM

(ICACC-S10-011-2018) Theoretical explanation of the ultralow and anisotropic thermal expansion of $Mg_2Al_4Si_5O_{18}$

Y. Li*[1]; J. Wang[1]

1. Institute of Metal Research, High-performance Ceramics Division, China

Magnesium cordierite $Mg_2Al_4Si_5O_{18}$ is a representative silicate widely used in ceramic industry and of promising application due to its ultralow coefficient of thermal expansion and good thermal shock resistance. In this work, first-principle calculations are performed to investigate the thermal expansion behavior in low cordierite β-$Mg_2Al_4Si_5O_{18}$ which crystallizes in an orthorhombic, pseudo-hexagonal system. According to the quasi-harmonic approximation and Grüneisen theory, temperature dependences of thermal and mechanical properties of $Mg_2Al_4Si_5O_{18}$ are predicted. The origin of ultralow thermal expansion in $Mg_2Al_4Si_5O_{18}$ can be attributed to the negative thermal expansion along c axis and the negative Grüneisen parameters, both of which are related with the unique framework structure of corner-linked tetrahedrons. The calculated results also suggest that the anisotropy of thermal expansion may arise not only from anisotropic elasticities but also anisotropic Grüneisen functions. This work reports an anisotropic behavior of thermal expansion in $Mg_2Al_4Si_5O_{18}$ and further provides an insight into the mechanism of low thermal expansion in $Mg_2Al_4Si_5O_{18}$.

Modeling of Performances I

Room: Coquina Salon F
Session Chair: Bin Liu, Shanghai University

10:20 AM

(ICACC-S10-012-2018) Grain Boundary Resistance for Phonon Thermal Conduction: A Perturbed Molecular Dynamics Study (Invited)

M. Yoshiya*[1]; K. Funai[1]; S. Fujii[1]; T. Yokoi[1]

1. Osaka University, Department of Adaptive Machine Systems, Japan

Thermal conduction can be controlled by grain boundary (GB) in the materials. The GB scattering of phonon has been discussed with an analogy to electronic conduction. While only electrons near Fermi level contribute to electronic conduction, all the modes of phonon can contribute to thermal conduction, in principle. In addition, mean free path of phonons are different from that of electrons, depending on materials. These two differences complicate the understanding of thermal conduction compared with electronic conduction. The experimental facts that nano-structuring of materials occasionally succeeding in modifying those two transport properties seems to pave new ways for optimizing those conductivity: High electronic conductivity and low thermal conductivity for thermoelectricity, as an example of many. However, our knowledge of phonon transport is still limited, impeding the optimization of the transport properties. In this study, we performed perturbed molecular dynamics simulations to evaluate local thermal conductivity in the vicinity of GB, followed by detailed analyses. It is found that GB resistance for thermal conduction is not as simple as we expected based on the analogy to electronic counterpart, and therefore more clear understanding must be obtained to fully exploit GBs for optimizing those transport properties.

10:50 AM

(ICACC-S10-013-2018) Tailoring thermal expansion of RE-silicate EBC candidates: challenges and opportunities (Invited)

J. Wang*[1]

1. Shenyang National Laboratory for Materials Science, Institute of Metal Research, Chinese Academy of Sciences, High-performance Ceramics Division, China

High-temperature capable hybrid T/EBC for SiC-based CMCs critically requested the better property matching between TBC and EBC layers. Rare earth silicates, typically including RE_2SiO_5 orthosilicates and $RE_2Si_2O_7$ pyrosilicates, are important EBC materials due to their excellent reliability in the extreme operating combustion environments. Unfortunately, typical thermal barrier coating candidates demonstrated obviously larger thermal expansion coefficients than RE-silicate EBC materials. This mismatch greatly challenged long-term thermal stability and durability of structural component. We recently discovered striking characters of phonon anharmonicity in RE silicates. With this deeper understanding of phonon secrets, it may provide the opportunity to tailor thermal expansion behavior through strategic phonon engineering. We also highlight the very low lattice thermal conductivity of RE silicates. Our results may support the innovative design of T/EBC system for SiC-based CMCs.

11:20 AM

(ICACC-S10-014-2018) Predicting the Fracture of ZrB_2-Carbon Based Composites using the Extended Finite Element Method

L. Jarvis*[1]; M. A. Zaeem[1]; G. Hilmas[1]; W. Fahrenholtz[1]; J. Watts[1]

1. Missouri University of Science & Technology, Materials Science and Engineering, USA

The ultra-high temperature ceramic (UHTC) zirconium diboride (ZrB_2) is often researched for extreme environment applications. Its high strength (> 400 MPa) and thermal conductivity (> 100 W/m•K) is promising for aerospace applications, but low fracture toughness (< 3 MPa•$m^{1/2}$) limits its use. Experimental research has shown that

the fracture toughness can be increased through engineering architectures that promote crack deflection. The goal of this research is to predict how these architectures will fail using the extended finite element method (XFEM). Accurate prediction of the crack propagation in these structures requires the implementation of interface mechanics into the method. User subroutines were written for ABAQUS 6.14 and benchmarked by comparing the stress intensity factors to closed-form solutions. Three different experimental architectures were then simulated with applied tension and bending: monolithic, laminated, and fibrous monolithic. The results were compared to experimental four-point bending tests in order to validate the accuracy of the model. With a valid model, new architectures can be designed that provide graceful failure and further increase the fracture toughness.

11:40 AM

(ICACC-S10-015-2018) Effects of Nanobubble Collapse on Precision Polishing : Molecular Dynamics Study

Y. Aoyama*[1]

1. IMR Tohoku University, Japan

Manufacturing high-performance semiconductor devices, fabrication of a highly-planar surface is important. For fabricating the highly-planar surface, chemical mechanical polishing (CMP) is used as precise polishing technique. Especially, the improvement of CMP performance for hard materials such as GaN and SiC is strongly desired. In order to increase the CMP performance to hard materials, we focused on a nanobubble, which is recently expected as a new method for the improvement of the CMP performance. When the nanobubble collapses, the liquid around the bubble forms a jet which creates a protrusion and secondary water hammer shock wave. Therefore, we considered that applying the jet phenomena increases the efficiency of CMP process. To establish the efficient CMP, it's necessary to reveal the effects of the nanobubble collapse on precise polishing of solid surfaces. In order to reveal the effects of the nanobubble collapse, we performed collapse simulation of the nanobubble on a solid surface by molecular dynamics (MD) method. The MD simulations with ReaxFF are performed with "LASKYO" software developed in our group. First, we made the nanobubble in water solvent by removing the solvent molecules from the nanobubble spheres. Next, we created shockwave and conducted nanobubble-collapse on the solid surface. In this conference, we report the effects of nanobubble collapse on the solid surface.

S12: Advanced MAX/mxene Phases and UHTC Materials for Extreme and High Temperature Environment

Properties, Oxidation, and Tribology II

Room: Tomoka B

Session Chair: Surojit Gupta, University of North Dakota

8:30 AM

(ICACC-S12-047-2018) Structure-property relations in zirconium diboride based ultra-high temperature ceramic composites

S. K. Kashyap*[1]; R. Mitra[1]

1. Indian Institute of Technology, Kharagpur, Metallurgical and Materials Engineering, India

In the present study, ZrB_2-SiC composite with varying amounts of LaB_6 has been prepared either by pressure-less sintering or spark-plasma sintering (SPS), and thereafter a comparative study of their microstructures, hardness, and creep behavior have been carried out. Composites synthesized by SPS at 1800°C under the load of 70 MP are found to exhibit higher relative densities and hardness than composite sintered by pressure-less sintering at 2000°C. Compressive creep test has been performed at 1300°C under three different stresses, i.e. (approx. 47, 62 and 78 MPa). Microstructural examinations and phase identification of sintered and post creep samples have been carried out to understand the densification mechanism and deformation behavior, respectively. The extent of dislocations, which were indicated by TEM analysis, showed that particle rearrangement and deformation are the main densification mechanism. Based on the results obtained in creep investigation, it was observed that 10 vol % LaB_6 composite showed higher creep resistance than others.

8:50 AM

(ICACC-S12-048-2018) Tribological and Wear Properties of Nanolaminated MoAlB

A. Benamor*[1]; S. Kota[2]; M. Barsoum[2]; M. Hadji[1]

1. University Of Blida 1, Mechanical Department, Algeria
2. Drexel University, USA

MoAlB is thus far the only transition metal boride that forms protective alumina scales when heated at high temperatures in air. It is also characterized by relatively low hardness compared to the di- and monoborides, high compressive strengths, and good electrical and thermal conductivity, making it potentially interesting as a wear resistant material. In this work, the tribological behavior of fully dense polycrystalline MoAlB ceramics were investigated at room temperature, sliding against 100C6 steel ball and Al_2O_3 counter-surfaces via pin-on-disc tribometer at loads up to 10 N. Interestingly, against the 100C6 steel ball, the wear track is characterized by the formation of ripples whose morphology varies with load and sliding time. Given that this phenomenon has previously only been observed on steel surfaces and some polymers (e.g. UHMWPE and rubber), SEM and XRD analysis of the wear surface is used to propose a mechanism for the ripples formation in the relatively brittle MoAlB ceramics against steel counter-surfaces.

9:10 AM

(ICACC-S12-049-2018) Influence of SiC_w content on densification, microstructure, mechanical properties and abrasive wear behaviour of spark plasma sintered ZrB_2-$MoSi_2$-SiC_w composites

T. R. Paul*[1]; M. K. Mondal[1]; M. Mallik[1]

1. National Institute of Technology, Durgapur, Metallurgical and Materials Engineering, India

The present study focusses on densification, microstructure, mechanical properties and abrasive wear behavior of three ZrB_2-20 vol.% $MoSi_2$-(5-20) vol.% SiC_w ceramic composites densified by spark plasma sintering at different combinations of temperature and pressure to investigate the densification mechanism involved during sintering. Vicker's hardness and indentation fracture toughness have been measured. The sliding wear of the investigated composite has been studied in pin-on-disk equipment at room temperature at different loads and three different sliding speeds. Results show that a fully dense composite is obtained at 1800 °C at a pressure of 60 Mpa. It has been found that both the Vickers hardness and density of the composites increased with increasing sintering temperature, pressure and amount of SiC_w. The maximum relative density, hardness and fracture toughness are 98%, 21 GPa and 6 MPa $m^{1/2}$. The tribological properties of the composites are significantly improved with increasing the amount of SiC_w. Results also show that the specific wear rate increases with increasing the applied loads. Microstructural characterization of worn surfaces suggest that wear mechanism is mainly controlled by grain fracture, grain pullout and microcracking.

S13: Advanced Ceramics and Composites for Nuclear Fission and Fusion Energy

Design and Test Technologies

Room: Coquina Salon H
Session Chairs: George Jacobsen, General Atomics; Michael Jenkins, Bothell Engineering and Science Technologies

8:30 AM

(ICACC-S13-043-2018) SIC-SIC CMCS for Nuclear Applications: Update on Progress of Working Group for Graphite and Composites in the ASME BPV Code Section III, Division 5 for High Temperature Reactors (Invited)

M. G. Jenkins*[1]; S. T. Gonczy[2]; Y. Katoh[3]

1. Bothell Engineering and Science Technologies, USA
2. Gateway Materials Technology, USA
3. Oak Ridge National Lab, USA

Future nuclear high-temperature reactors (HTRs) planned by US DOE will use SiC-SiC CMCs to enhance fuel performance and improve accident tolerance because SiC-SiC CMCs are tolerant to the irradiation and chemical environments. Because SiC-SiC CMCs are nonconventional materials they are of special concern because the mission of the US Nuclear Regulatory Commission (NRC) is to license and regulate the nation's civilian use of byproduct, sources and special nuclear materials. NRC regulates nuclear reactors and new reactor design as well as the reactor materials. NRC not only imploys, but is legally required to use, consensus codes and standards as an integral part of the regulatory process. ASME Boiler and Pressure Vessel (BPV) Code Section III "Rules for Construction of Nuclear Components" is included in the NRC regulations. Division 5 High Temperature Reactors (HTR) of Section III has supported a working group on graphite and composites (e.g., SiC-SiC CMCs) for many years. For SiC-SiC CMCs to be incorporated into current and future nuclear reactors, they must be included in ASME BPV Code as acceptable materials. This paper provides an update of progress on ew portions of ASME BPV Code including design and qualification of SiC-SiC CMCs for nuclear applications.

9:00 AM

(ICACC-S13-044-2018) Transient Mechanical Testing of SiC/SiC Composite Tubes to Simulate Pellet-Cladding Mechanical Interaction During Reactivity Insertion Accidents

M. N. Cinbiz[2]; N. R. Brown*[1]; K. Terrani[2]; K. Linton[2]

1. Pennsylvania State University, Mechanical and Nuclear Engineering, USA
2. Oak Ridge National Laboratory, USA

Advanced technology nuclear fuel cladding candidates are being developed to enhance the mechanical response of nuclear fuel rods during beyond design-basis accidents in light-water nuclear reactors. Silicon carbide fiber/silicon carbide matrix (SiC/SiC) composites are considered as a candidate for advanced technology nuclear fuel cladding because of their corrosion resistance, in addition to their superior mechanical strength. The mechanical performance of SiC/SiC composites must be evaluated during design-basis accidents. The reactivity insertion accident (RIA) is a design-basis accident where the cladding may be subjected to mechanical straining caused by the pellet-cladding mechanical interaction due to the rapid thermal expansion of the fuel. We investigated the mechanical behavior of SiC/SiC composite samples which were subject to modified-burst testing at various strain rates in the range of 10 ms to 6 sec. The evolution of the average hoop strain was monitored by an in-situ digital image correlation system which includes a novel mirror setup to determine the failure location on the samples' outer surface. The results focus on the mechanical response under expected reactivity insertion conditions and limiting conditions. The failure of the SiC fibers was characterized by the average hoop strain evolution during RIA transients.

9:20 AM

(ICACC-S13-045-2018) Micro-Mechanical Characterization of the PyC interphase in SiC/SiC composites

J. Kabel*[1]; P. Hosemann[1]; C. Deck[2]; T. Koyanagi[3]; Y. Katoh[3]; I. Love[4]

1. University of California Berkeley, Nuclear Engineering, USA
2. General Atomics, USA
3. Oak Ridge National Lab, USA
4. Oregon State University, USA

Ceramic fiber-matrix composites (CFMCs) have gained significant attention as an engineering material for their high temperature strength and unique resistance to catastrophic brittle failure. In the nuclear industry, SiC_{fiber} /PyC/ SiC_{matrix} composites have received specific interest for improved safety regarding corrosion, temperature, and irradiation limitations compared to zirconium alloyed clad in traditional LWR's. This research investigates experimental micro-mechanical methods to aid in the validation CFMC physics models for micro-crack propagation, and discusses implementation of these mechanics and property values into bulk component modelling. Micro-pillar compression was employed to evaluate the interfacial debond shear strength (τ), internal friction coefficient (μ), and fracture energy release rate (Γ) of the PyC layer at the SiC_{fiber}/SiC_{matrix} interface. Micro-pillars (3μm) of varying PyC thickness (50-1300nm) and irradiation conditions (~1 and ~11 dpa) were fabricated from CVI composites with SA3 and HNLS fibers via focused ion beam milling. For HNLS fiber composites with 50nm thick PyC, it was found that; τ = 265 MPa, μ = 0.25, and Γ = 2.5 J/m^2. Composite irradiation was observed to increase μ to 0.36 while decreasing τ to 103 MPa. Additionally, increased PyC thickness was observed to decrease μ to 0.17 and τ to 133MPa, without significant influence on Γ.

9:40 AM

(ICACC-S13-046-2018) Flexural Strength of Ceramic Matrix Composite Tubes: Draft ASTM Standard Test Method

M. G. Jenkins*[1]; J. E. Gallego[1]

1. Bothell Engineering and Science Technologies, USA

US DOE is planning to use advanced materials for the core and the reactor unit components in various advanced reactor concepts. Ceramic matrix composites (CMC), in particular silicon carbide (SiC) fiber SiC-matrix (SiC-SiC) composites, could greatly expand the design window for various components in terms of operating temperature, applicable stress, and service life, as compared to heat-resistant metallic alloys, while significantly improving accident tolerance and safety margins. Potential CMC tubular components include fuel rods, control rod sleeves, and control rod joints. Possible failure modes for these components include axial and hoop tension, axial flexure, axial and diametral compression, and axial shear. A draft ASTM standard test method has been developed and submitted for full-consensus ballot to determine the flexural strength of ceramic matrix composite tubes subjected to bending. Modeling and empirical tests of composite tubes provided validation of the parameters specified in the test method. The draft standard test method addresses the following experimental issues -- test specimen geometries/preparation, test fixtures, test equipment, interferences, testing modes/procedures, data collection, calculations, reporting requirements, precision/bias.

10:20 AM

(ICACC-S13-047-2018) Development of the underwater acoustic emission technique for composite damage characterization (Invited)

T. Nozawa*[1]; H. Tanigawa[1]

1. National Institutes for Quantum and Radiological Science and Technology, Japan

The acoustic emission (AE) method is recently recognized as a powerful tool to characterize composite's damage accumulation process and has widely been applied in composite tests. For instance, a fracture origin can be determined by analyzing time lag of AE travels at individual sensors attached directly on the composite surface. This fracture location algorithm works most effectively in 1D line analysis with sound accuracy. However, for the 2D/3D cases, there is a drawback that the location uncertainty increases since AE wave velocities are varied due to the inherent anisotropy of the composites. In contrast, we have been developing the underwater AE technique as the alternative option. The underwater AE generally travels straightly in water regardless of material anisotropy. Specifically we believe that this will be more advantageous in monitoring and inspection of silicon carbide composite fuel cladding as an accident tolerant fuel for the light water fission reactor. To raise reliability of damage monitoring by this developmental underwater AE technique, it is essential to establish a data calibration method oriented to the experimental setup. This study therefore aims to evaluate the effects of direct/indirect wave interaction, water temperatures, material and geometry of the tank, etc. to discuss the capability of the underwater AE method as a realistic option for composites.

10:50 AM

(ICACC-S13-048-2018) Mechanical and Thermo-Mechanical Property Measurement for Silicon Carbide Based Accident Tolerant Fuel Cladding (Invited)

G. Jacobsen*[1]; K. Shapovalov[1]; X. Huang[2]; C. Deck[1]

1. General Atomics, Nuclear Technologies and Materials, USA
2. University of South Carolina, Mechanical Engineering, USA

Due to their inherent accident tolerance, General Atomics (GA) is developing silicon carbide (SiC) matrix, SiC fiber reinforced composite (SiC-SiC) based cladding as part of an accident tolerant fuel concept to be used as a drop in replacement for the Zircaloy/UO_2 fuel system currently used in light water reactors. An update on efforts on mechanical testing of the tubular prototypical SiC-SiC cladding structures will be presented. Room temperature testing, including uniaxial tensile, elastomer insert, pressure burst, c-ring, and endplug pushout testing, was used for statistical evaluation and property analysis. Digital image correlation, acoustic emission, and helium leak detection were complimentarily performed during testing and key results will be presented. Details of test methodology, such as the use of passive grip methods to facilitate >85% test efficiency, will be discussed. High temperature isothermal test results will be presented up to 1800 °C showing full retention of strength. Thermo-mechanical testing replicating mechanical contact between pellet and cladding with a realistic radial thermal gradient was performed. The implication of these test results towards SiC as an accident tolerant fuel cladding and the development of specifications will be discussed.

11:20 AM

(ICACC-S13-049-2018) SiC-SiC ATF Cladding Development: Hermeticity Test Method with High Pressure Helium Internal Gas Pressurization

C. P. Shih*[1]; G. Vasudevamurthy[1]; A. S. Blacklock[1]; G. Jacobsen[1]

1. General Atomics, USA

Excellent thermo-mechanical and irradiation properties make continuous SiC fiber reinforced CVI SiC matrix (SiC_f-SiC_m) composite tubes a highly desirable candidate to replace Zircaloy cladding in light water reactors (LWRs) to improve accident tolerance. These ceramic composites has been engineered to achieve the acceptable levels of hermeticity to gaseous fission products. Currently no standard test method exists to measure the hermetic behavior of composite tubes, especially while under load, an issue considered critical before their ultimate deployment in LWRs. In this context, a test setup is developed to evaluate the hermeticity of nuclear grade SiC_f-SiC_m composites ATF cladding tubes with direct tube pressurization using a high pressure helium source and a high sensitivity helium leak detector for leak detection. This setup is capable of pressurizing the tubes to an internal pressure of 32 MPa, which is higher than the fuel rods upper-bound end of life internal pressure of ~20 MPa for current pressurized water reactors. The direct pressurization method closely simulates the in-the-field scenario and provides reliable data on the hermeticity of the SiC_f-SiC_m composites tubes. The results of the experiments and hermeticity data will be presented and the resulting implications to normal operating and accident conditions discussed.

11:40 AM

(ICACC-S13-050-2018) Evaluation of Elastic Properties of SiC/SiC Tubular Specimens using Resonant Ultrasound Spectroscopy

G. Singh*[1]; T. Koyanagi[1]; C. Petrie[1]; K. Terrani[1]; Y. Katoh[1]

1. Oak Ridge National Lab, USA

SiC/SiC cladding is one of the leading candidates for accident tolerant fuel-cladding systems for light water reactors. Research and development of SiC/SiC cladding will involve determining the elastic properties of pre/post irradiation SiC/SiC specimens. In the present work, the elastic properties of CVI SiC/SiC tubular specimens, irradiated under fast neutron flux and high heat flux, were evaluated using Resonant ultrasound spectroscopy (RUS) and finite element analysis. The results show significant decrease in the elastic modulus

S14: Crystalline Materials for Electrical, Optical and Medical Applications

Optical Material III

Room: Tomoka C

Session Chairs: Luisa Bausa, Universidad Autonoma de Madrid; Jean-Rene Duclere, Laboratoire SPCTS

9:00 AM

(ICACC-S14-036-2018) Transparent polycrystalline ceramics with crystalline orientation controlled by a magnetic field (Invited)

T. S. Suzuki*[1]; T. Ashikaga[2]; B. Kim[1]; K. Morita[1]; H. Kiyono[2]

1. National Institute for Materials Science (NIMS), Japan
2. Shibaura Institute of Technology, Japan

Transparent polycrystalline ceramics provides flexibility in size and shape design and can be expected to apply for a wide field. Microstructure control is very important and extremely low porosities are indispensable for high transparency. When materials possess anisotropic crystal structure, optical birefringence at grain boundaries also affects transmission. If the crystal direction is aligned, effect

of birefringence at grain boundary can be reduced. The colloidal processing in a strong magnetic field was able to control the crystallographic orientation even in diamagnetic ceramics. In this process, a strong magnetic field is applied to the particles in a stable suspension. The driving force for the magnetic alignment is a magnetic torque generated from the interaction between the magnetic anisotropy of particles and the applied magnetic field, thus the orientation of the crystal depends on the axis having easy magnetization. The particles become rotated to an angle that minimizes the system energy and one-dimensional orientation can be achieved. The c-axes of alumina and AlN are aligned by a static magnetic field and a rotating magnetic field, respectively. After slip casting, SPS was used for reduction of porosities. Transparency can be improved by the alignment of the c-axis in alumina and AlN.

9:30 AM

(ICACC-S14-037-2018) Increasing the absorption efficiency of Yb^{3+} doped anisotropic crystals by means of disordered plasmonic networks (Invited)

L. Sanchez-García[1]; M. Ramirez[1]; J. Carvajal[2]; R. Sole[2]; M. Aguilo[2]; F. Diaz[2]; L. E. Bausa*[1]

1. Universidad Autonoma de Madrid, Fisica de Materiales, Spain
2. Universitat Rovira i Virgili, Fisica i Cristal lografia de Materials, Spain

Increasing Yb^{3+} absorption efficiency is relevant in a variety of applications such as bio-imaging, photovoltaics devices or NIR-driven photocatalysis. In this work metallic nanoparticles connected in disordered plasmonic networks (DPNs) are deposited on Yb^{3+} doped $RbTiOPO_4$ crystals. The coexistence of localized and delocalized plasmonic modes in DPNs results into a broad spatial and spectral distribution of their plasmonic resonance, useful to overlap the optical response of Yb^{3+} in the NIR spectral region. Up to a 5 fold enhancement of Yb^{3+} photoluminescence is observed. The results are analyzed by means of the calculated near field components of the DPNs, and the experimental absorption cross-section of the electric-dipole transitions of Yb^{3+} ions along the different crystallographic directions of RTP. We show that in the near field regime, the presence of the DPNs generate additional polarization field components to that of the incident field, which allow the access to the largest transition dipolar moment of Yb^{3+} ions. As a result, a much more efficient excitation route takes place for those Yb^{3+} ions at the immediacy of the metallic networks. The work provides fundamental insights for designing metallic nanoparticle arrangements to enhance the optical properties of Yb^{3+} ions, and can be extended to other dielectric hosts as well as to other Lanthanide ions.

10:20 AM

(ICACC-S14-038-2018) Effect of transition metal dopant valencies on stability within the alumina lattice

N. Ku*[1]; V. L. Blair[1]; A. L. Fry[1]; M. Kornecki[1]; S. Raju[1]; R. E. Brennan[1]

1. U.S. Army Research Laboratory, USA

Research has been conducted to create laser host materials by doping alumina (Al_2O_3) with various metal cations. The addition of rare earth dopants within the alumina lattice has been shown to largely influence the magnetic response of the material, enabling the use of advanced processing techniques under applied magnetic fields. These techniques aid in the ability to engineer the microstructure, specifically in the alignment of grains. The addition of transition metal dopants in place of the rare earth is expected to further increase the magnetic response of alumina and thereby better assist microstructure control. This work will investigate the stability and solubility limits of transition metals, including iron, manganese, nickel, and cobalt, in the alumina lattice. A precipitation process will be used to synthesize doped alumina powder. A challenge will be the charge mismatch between divalent dopants, such as nickel (Ni^{2+}) and cobalt (Co^{2+}), and the trivalent aluminum cation (Al^{3+}). The use of a tetravalent co-dopant will be examined to offset the charge mismatch and stabilize the dopant within the lattice.

10:40 AM

(ICACC-S14-039-2018) Association of metallic nanostructures with periodically poled $LiNbO_3$ crystals for enhanced nonlinear processes at nanometric dimensions (Invited)

A. Gómez-Tornero[1]; D. Hernandez-Pinilla[1]; P. Molina[1]; C. Tserkezis[2]; L. E. Bausa[1]; M. Ramirez*[1]

1. Universidad Autonoma de Madrid, Spain
2. Technical University of Denmark, Department of Photonics Engineering, Denmark

Here, we combine ferroelectric domain engineering and polarization-mediated chemistry to obtain finely designed plasmonic arrays of closely spaced interacting silver nanoparticles. The metallic arrangements are formed on the domain wall surfaces of linear (1D) and hexagonal (2D) ferroelectric domains spatially distributed on the polar surface of a $LiNbO_3$ crystal. Then, the coupling between the nonlinear optical response provided by the crystal and the localized surface plasmon resonances supported by the metallic nanostructures is analyzed to develop hybrid plasmonic-nonlinear optical sources with improved performances at the nanoscale. The results provide a cost effective and versatile approach for fabricating scalable plasmonic super-structures and offers alternative avenues to develop hybrid plasmonic metamaterials with potential applications in ultra high-density data storage, optical circuits, or ultra-sensitive detection.

11:10 AM

(ICACC-S14-041-2018) Elaboration of new tellurium oxide based glasses, glass-ceramics, ceramics and their associated nonlinear optical and lasing properties (Invited)

J. Duclere*[1]; M. Dolhen[1]; S. Chenu[1]; G. Delaizir[1]; A. Bertrand[1]; M. Dutreilh-Colas[1]; J. Cornette[1]; J. De-Clermont-Gallerande[1]; T. Hayakawa[2]; N. Ghribi[1]; V. Couderc[4]; M. Allix[3]; O. Masson[1]; P. Thomas[1]

1. Laboratoire SPCTS, Chimie, France
2. Nagoya Institute of Technology, Japan
3. CEMHTI, France
4. XLIM, France

Tellurium dioxide-based (tellurite) materials and especially glasses are currently considered as very promising materials for being integrated in nonlinear optical devices (ultra-fast optical swiches) because of their high nonlinearities (hyper-susceptibility $\chi(3)$ exceeding by far that of glassy SiO_2 - factor larger than 50 times), their high Raman gain coefficients and their good visible and near-infrared light transmittance (up to 6 μm). Therefore, some substantial literature has been devoted to the studies of such tellurite glasses during the last decade. This communication will present a review of some research activities developed at the SPCTS laboratory in Limoges (France), over the past 5 years, on these tellurite materials. In particular, a focus will be made on: (i) the synthesis of new glasses, glass-ceramics and recently transparent ceramics, (ii) the study of the associated nonlinear (3^{rd}-and 2^{nd} orders) optical properties, with some emphasis on the dependence with the chemical composition, (iii) the promising bulk lasing properties of the transparent tellurite ceramics.

S16: Geopolymers, Inorganic Polymers and Sustainable Materials

Mechanical Properties, Infrastructure, and Sustainable Materials

Room: Ponce de Leon
Session Chair: Ange Therese Akono, University of Illinois at Urbana-Champaign

8:30 AM

(ICACC-S16-015-2018) Adhesion of Unreinforced Metakaolin Geopolymer to Common Metal Substrates (Invited)

T. A. Carlson*[1]; M. Ziemann[1]; D. Hernandez[1]; P. Stynoski[1]; C. P. Marsh[1]; G. Kutyla[2]; W. M. Kriven[2]

1. USACE, USA
2. University of Illinois at Urbana-Champaign, USA

Geopolymers have a wide variety of possible compositions and reinforcements and have been investigated as possible replacements for cements, ceramics, and polymers in several applications. Use as a protective coating or adhesive for metal could utilize geopolymers' oxide chemistry which has shown potential for stronger bonding than traditional polymer coatings. A metakaolin-based geopolymer of composition $M_2O:Al_2O_3:4SiO_2:11H_2O$ (where M was Na or Cs) was used to bond together polished metal substrates. The metals tested were mild steel, stainless steel, copper, brass, and bronze. The sample configuration was bonded cylinder faces, tested in tension to determine the strength of the bond. In many cases, the failure was cohesive within the geopolymer, indicating that the adhesive bond to the metal was as strong or stronger than the geopolyer itself. Electron microscopy and spectroscopy methods were used to characterize the metal to GP interface. This work showed that the pH, base metal, and alloying elements play an important role in geopolymer adhesion. In this work, we show that geopolymers have the potential to be coatings or adhesives for many metal substrates.

9:00 AM

(ICACC-S16-016-2018) Characterization of Metakaolinite Phosphate Cementitious matrix suitable for Textile Reinforced Composites (Invited)

A. Katsiki*[1]; Y. Pontikes[2]; H. Rahier[1]

1. Vrije Universiteit Brussel, Department of Materials and Chemistry, Belgium
2. KULeuven, Belgium

The aim of this work is to study a newly developed cementitious matrix systematically, to be used in combination with E-glass fibers. An experimental study on the chemistry, microstructure and mechanical properties of the matrix material is presented. The setting reaction of metakaolinite phosphate cement (MKPC) is investigated using isothermal calorimetry (TAM Air), differential scanning calorimetry, scanning electron microscopy, X-ray diffraction, and infrared spectroscopy. The presence of metakaolinite powder and phosphoric acid solution leads to the formation of phospho-aluminosilicate cement. The reaction stoichiometry is studied by varying the Al/P molar ratio from 0.44 to 2.50. The results reveal that the optimum stoichiometric value for the reaction of metakaolinite with the phosphoric acid is close to 1 Al/P molar ratio. The compressive strength is found to be strongly dependent on the P_2O_5 concentration. In fact, the decrease in the Al/P molar ratio leads to an increase in strength up to a maximum around an Al/P ratio of 1. After that, by further decreasing the Al/P molar ratio, a decrease in strength occurs again due to excess of phosphates and even self-degradation of the specimens after initial hardening. The investigated MKPC mortar achieves strengths up to 70MPa in compression after 7 days, which render it a promising candidate for construction purposes.

9:30 AM

(ICACC-S16-017-2018) Geopolymer Roof Tile (Invited)

A. Reggiani*[1]

1. GeoMITS srl, Italy

Motivation : heavy traditional roof tiles are burned in furnace at more than 1000°C, their properties depends on calcining, but their weight create problems about transportation, production costs and weight are an issue. Solution : Geopolymer roof tiles are made at room temperature, are durable, are cheap, they use 0 km raw materials and they are lightweight, more easy to deliver. Method: powder precursors : Metakaolin, slag, fly ash, white and black dense microsilica. Aggregates : inert sands and partially reactive fillers. User friendly Silicates used : sodium based (MR=2)and potassium based (MR=1,6). Metakaolin-slag fluid geopolymer mortar easy to fill in plastic, silicon or polyhurethane molds, condenses in few hours at room temperature (depending on type of metakaolin used). Fly ash-slag-microsilica fluid geopolymer mortar condenses in few hours (depending on silicate used). Organic content remains extremely low, no superplasticizers are added and after hardening geopolymer tile has the same properties from the bottom to surface. Results : Geopolymer roof tiles are acid-sulphate resistant, water-oil repellent, frost/defrost resistant, with high flexural and compressive strength. Depending on different sources of alumina silicates color can vary from whitish, to cream, from red brick to grey, up to absolute black. Conclusion : Geopolymer roof tile represents the present and the future of roof tiling worldwide

Sustainable Materials

Room: Ponce de Leon
Session Chair: Gregor Gluth, Bundesanstalt für Materialforschung und -prüfung (BAM)

10:20 AM

(ICACC-S16-018-2018) Porous glass-ceramics from alkali activation and sinter-crystallization of waste glass mixtures

P. Rabelo Monich[1]; A. Rincon[1]; D. Hoellen[2]; E. Bernardo*[1]

1. University of Padova, Dipartimento di Ingegneria Industriale, Italy
2. Montanuniversitaet Leoben, Department of Environmental and Energy Process Engineering, Austria

Alkali-activated aqueous slurries of fine glass powders, mostly deriving from the plasma processing of municipal solid waste ('Plasmastone'), were found to undergo progressive hardening, at low temperature (80 °C), owing to the formation of C-S-H (calcium silicate hydrate) gels. Before complete setting, slurries could be easily foamed by vigorous mechanical stirring, with the help of a surfactant; finally, the resulting open-celled structure could be 'freezed' by a subsequent sintering treatment, with crystallization of Ca-Fe silicates. The densification of the struts upon firing was enhanced by mixing Plasmastone with recycled glasses, up to 40 wt%, and operating on the firing temperature (from 800 to 1000 °C). A total porosity exceeding 75 vol%, comprising both well-interconnected macro-pores and micro-pores on cell walls, was accompanied by good compressive strength, well above 1 MPa. The stabilization of pollutants generally increased with increasing firing temperature and glass content, with some exceptions; no practical leaching was observed from samples deriving from Plasmastone combined with 30 wt% borosilicate glass from the recycling of pharmaceutical vials.

10:40 AM

(ICACC-S16-019-2018) Bone Ash Reinforced Geopolymer using Metamax, Mymenshingh Clay and Synthetic Mymenshigh Clay-derived Metakaolin (Invited)

A. W. Bhuiya[1]; D. Ribero[1]; M. Hu[1]; W. M. Kriven*[1]

1. University of Illinois at Urbana-Champaign, USA

Natural calcined bone ash of hydroxyapatite and dicalcium phosphate were investigated as reinforcements of potassium geopolymer (KGP). Particulate reinforcements of 5, 10 and 15 wt % each of hydroxyapatite and dicalcium phosphate were added to potassium geopolymer to compare with KGP using MetaMax, KGP(MT), potassium geopolymer with Mymensingh clay metakaolin, KGP(MW) and potassium geopolymer using synthetic Mymensingh clay metakaolin, KGP(MW-SYN). Microstructural properties and mechanical properties were investigated (SEM, XRD, Instron) for geopolymer samples at both RT and HT. The crystalline peaks observed in KGP (MW) as well as in potassium geopolymer reinforced with hydroxyapatite and dicalcium phosphate. Thermally treated geopolymer at 1150^{0}C also exhibited crystalline peaks of leucite, kalsilite, monetite and quartz confirming the signature of geopolymer ceramics at elevated temperature. Geopolymer composites after thermal exposure at 1150^{0}C revealed microstructural integrity with the formation of phosphate glass, while a self-glazed surface was developed in KGP (MW) after being heated at 1125 ^{0}C. RT flexure strength increased to 18.3 MPa in KGP (MW), 11.0 MPa in KGP (MT)-15HA and 11.1 MPa in KGP (MT)-15DCP, 14.74 MPa in KGP(MW-SYN) and 14.5 MPa in KGP(MW-SYN)-5DCP compared to that in pure KGP (MT), 8.4 MPa.

11:00 AM

(ICACC-S16-020-2018) The material properties of cellulose nanofiber (CNF) reinforced metakaolin based geopolymer composites (Invited)

S. Cho*[1]

1. Hyundai Motor Company, Republic of Korea

The cellulose nanofiber is the next generation fibers which have higher mechanical properties than carbon nanofibers, especially elongation. Incorporating small amount of CNF into metakaolin based geopolymers would significantly increase their strength and elongation, which is not possbile in traditional fiber reinforced geopolymer composites. The microstructure, mechanical and thermal properties of CNF reinforced geopolymer composites were investigated in this work.

11:30 AM

(ICACC-S16-021-2018) Preliminary Results on the Performance-Based Specification for Amazonian Geopolymer Composites (Invited)

R. A. Sa Ribeiro*[1]; M. G. Sa Ribeiro[1]; M. G. Sa Ribeiro[2]; M. R. Sardela[3]; W. M. Kriven[4]

1. INPA-National Institute for Amazonian Research, LTEE-Structural Engineering Laboratory, Brazil
2. Architect and Urban Planner, Brazil
3. University of Illinois at Urbana-Champaign, Frederick Seitz Materials Research Laboratory, USA
4. University of Illinois at Urbana-Champaign, Materials Science and Engineering, USA

This is an ongoing joint Brazil-USA testing program on the performance-based specification for Amazonian geopolymer non-reinforced and bamboo-reinforced for use in sustainable construction. The bamboo-reinforced geopolymer composites are intended to be an alternative to conventional cements and concretes. This project uses bamboo cultivated in the Amazon region and metakaolin attained from calcined Amazonian kaolin. While fly ash-based geopolymers are now an established technology, metakaolin-based cements and concretes still need to be developed and optimized based on the knowledge of the specific characteristics and properties of local resources. As part of this collaborative research, bamboo particles and fibers were produced at INPA and taken for tests at UIUC. In addition, kaolin was brought from the Amazon, and calcined into metakaolin at UIUC laboratories. The performance specifications include drying shrinkage, compressive strength-elastic modulus-Poisson's ratio, exposure to sulfuric and hydrochloric acid and sulfate resistance. In addition, scanning electron microscopy, energy dispersive spectroscopy and x-ray fluorescence were used to investigate the microstructure of the composite materials. X-ray diffraction confirmed the formation of geopolymer.

S17: Advanced Ceramic Materials and Processing for Photonics and Energy

Photonics II

Room: Halifax A/B

Session Chairs: Farid Akhtar, Stockholm University; Marta Cerruti, McGill University

8:30 AM

(ICACC-S17-035-2018) Mixed Quantum Dot Organic Antennas for Light Harvesting (Invited)

J. R. Caram*[1]

1. University of California, Los Angeles, Chemistry, USA

Hybrid materials consisting of organic light absorbers and quantum dot emitters have many potential applications in photochemistry and solar light harvesting. Here we present hybrid systems consisting of supramolecular dye aggregates and nanocrystal lead sulfide quantum dots. We demonstrate greatly enhanced nanocrystal emission at low QD concentrations in the presence of an aggregate acceptor. Simulations demonstrate that extended exciton diffusion and enhanced molecular transition dipoles enable greatly enhance QD emission. Such systems provide strong absorption in the visible with large effective stoke shifts, and may be suitable for luminescent solar concentrators.

9:00 AM

(ICACC-S17-036-2018) Optical characterization of sub-wavelength photonic structures (Invited)

J. A. Zapien*[1]; Y. Foo[1]

1. City University of Hong Kong, Department of Materials Science and Engineering, Hong Kong

Optical techniques provide highly desirable non-invasive, non-destructive, fast, and contactless characterization capabilities of use for materials characterization and sensing. Spectroscopic ellipsometry (SE) achieves extreme sensitivity because it is a phase sensitive and self-referenced technique akin to a "single beam interferometry". The power of SE to provide non-imaging subwavelength scale information in semiconductor and dielectric 1D gratings is well know in the semiconductor industry where it has been used to determine optical critical dimension (OCD) characterization of purposely made 1D gratings using the Rigorous Coupled-Wave Analysis (RCWA) technique. However, RCWA presents difficulties for the modeling of 2D and plasmonic structures. Our group has provided the first systematic demonstration of a suitable alternative to RCWA for the optical characterization of such complex samples by SE using the Finite-Difference Time-Domain (FDTD) method. This approach provide numerical results with precision equivalent to ~ 1/2-monolayer thickness sensitivity and provide an alterative computational technique that can extend the application of SE to provide detailed information on complex samples including photonic and plasmonic subwavelength structures of interest for sensing and energy applications. Financial support from the RGC of the HKSAR, China (Project CityU- 122812) is acknowledged.

9:30 AM

(ICACC-S17-037-2018) A Close Look at Various Types of Light-Matter Interaction in Organic Semiconductors: From Molecular to Device Physics. (Invited)

E. Orgiu*[1]

1. Institut National de la Recherche Scientifique, Energy Materials Telecommunications, Canada

Within my talk, I intend to give an overview of the efforts in understanding light and matter interactions in organic semiconductors by presenting three interesting cases: Light-matter interaction in presence of strong coupling with the vacuum field; the use of light at selected wavelengths to induce charge density variations in bi-component blends including photochromic molecules (diarylethenes) and a semiconducting conjugated polymer (poly(3-hexylthiophene); measurements of a photogenerated current through novel device geometries in highly crystalline molecular nanowires.

10:20 AM

(ICACC-S17-038-2018) Imaging the thermal properties of two-dimensional thermoelectric materials for direct energy conversion (Invited)

G. Fanchini*[1]

1. University of Western Ontario, Physics and Astronomy, Canada

Determining and imaging the thermal properties at the nanoscale is a demanding experimental challenge. So far, virtually any techniques used to image nanoscale thermal properties require to position the sample in contact with voluminous probes that act as undesirable thermal sinks and dramatically affect the measurements. Here, we present near-field scanning thermoreflectance imaging (NeSTRI), a new scanning probe technique in which an aperture-type near-field optical microscope at sub-wavelength resolution is used to determine the thermoreflectance of thin films in non-contact mode. Thermal conductivity of micrometre-size multilayer graphene platelets is determined and is consistent with previous macroscopic predictions. NeSTRI has also been used to measure graphene thin films decorated with a copper nanoparticle (Cu-NP) layer, before and after the deposition of of Cu-NPs and after Cu-NP removal. In this system, we have been able to show that the decrease of thermal conductivity of graphene in contact to metal nanoparticles is due to phonon scattering by Dirac electrons in graphene, and not to metal-graphene interfacial thermal resistance. eSTRI is uniquely suited to understanding the thermal properties of a large class of thermoelectric materials for direct energy conversion.

10:50 AM

(ICACC-S17-039-2018) An Overview of the Potential Energy-Related Applications of the Emerging Wide Bandgap Semiconductor Material: b-Ga_2O_3 (Invited)

D. J. Rogers*[1]

1. Nanovation, France

Recently, there has been a surge in interest for the wide bandgap (Eg ~ 4.9 eV) semiconductor gallium oxide (Ga_2O_3). A key driver for this boom is that single crystal wide area bulk β-Ga_2O_3 substrates have become commercially available and a variety of methods have been shown to give high quality epitaxial growth. Although Ga_2O_3 has a number of polymorph forms (α-, β-, γ-, δ- and ε) the more stable monoclinic phase (β-Ga_2O_3) has attracted the most attention. Amongst a whole range of potential commercial applications power electronics and bulk photovoltaics could have enormous impact on global energy consumption. In this paper we give an overview of the perspectives for these devices with examples from the development work carried out at Nanovation.

11:20 AM

(ICACC-S17-040-2018) Environmental friendly Carbon Dots: characterization and application in energy devices

D. Benetti*[1]; Y. Zhou[1]; H. Zhao[1]; A. Vomiero[2]; F. Rosei[1]

1. Institut National de la Recherche Scientifique, Énergie Matériaux Télécommunications, Canada
2. Lulea University of Technology, Engineering Sciences & Mathematics, Sweden

Generating power directly from solar radiation represents a promising opportunity towards addressing the increasing demand for clean energy, also reducing environmental impact caused by excessive carbon emissions. This challenge may be addressed by using carbon Dots (Cdots) which represent an emerging class of semiconducting nanomaterials. C-dots are exclusively composed of non-toxic elements (C, N and O) and can be synthesized in large quantities via a simple solvothermal approach. Compared to conventional semiconducting quantum dots (QDs), carbon dots (Cdots) have superior advantages of non-toxicity, environmental friendliness, low-cost and simple preparation using abundant carbon based feedstock. It has been shown that doping the core of Cdots with certain amount of nitrogen can tune their optical and electrical properties. Exploiting this ability, we synthesized different type of Cdots with absorption and emission spectra in the range 380 nm to 650 nm. We then verified their ability to inject electrons/holes in different materials, such as TiO_2 and Graphene Oxide. Thanks to their properties, the Cdots were then employed for realizing different devices such as Luminescent Solar Concentrator and perovskite solar cells.

Honorary Sympoisum: Advancing Frontiers of Ceramics for Sustainable Society Development - International Symposium in Honor of Dr. Mrityunjay Singh

Advancing Frontiers of Ceramics VI -Composite Materials 2

Room: Coquina Salon E
Session Chairs: Michael Halbig, NASA Glenn Research Center; Hagen Klemm, FhG IKTS Dresden

8:30 AM

(ICACC-HON-044-2018) A Brief History of Thermal Protection Systems (Invited)

S. M. Johnson*[1]

1. Johnson Consulting, USA

Thermal protection systems (TPS) are used to protect space vehicles from the heat of entry or rentry into an atmopshere after space flight. A vehicle requires propulsion to leave and a themal protection system to enter or come back. These materials must protect the vehicle from both the convective and radiative heating that occurs during flight through an atmosphere. They must be efficient and reliable, which means they must behave predictably and protect the vehicle and contents as efficiently as possible with the minimum mass and or volume. This talk will trace some of the developemts in these materials over the years, with particular empahsis on US space missions. Both reusable and ablative systems will be discussed, and how the use of these materials has changed over time. Some future needs for space exploration will also be discussed.

9:00 AM

(ICACC-HON-045-2018) Progress and Plans for CMC Research at NASA Glenn in 2018 (Invited)

J. E. Grady*[1]

1. NASA Glenn Research Center, Ceramic & Polymer Composites Branch, USA

As part of NASA's Aeronautics research, Glenn Research Center has developed SiC/SiC Ceramic Matrix Composites for 2700°F turbine engine applications in the next generation of ultra-efficient aircraft. In this presentation, the development of fiber and matrix constituents and fabrication processes that enabled this advancement will be reviewed, and characterization of the resulting improvements in CMC mechanical properties and durability will be summarized. Progress toward the development and experimental validation of models predicting the effects of the engine environment on durability of Ceramic Matrix Composites and Environmental Barrier Coatings will be summarized. Results from current collaborative research with industry and other government agencies will be reviewed. Research plans for 2018 and opportunities for future collaborations will also be summarized.

9:20 AM

(ICACC-HON-046-2018) Layerwise Fabrication and Manufacturing of Complex Materials and Structures for Propulsion Applications

M. C. Halbig*[1]; M. Singh[2]

1. NASA Glenn Research Center, USA
2. Ohio Aerospace Institute, USA

The next generation of materials and structures for turbine engine and electric motor applications requires specially developed layerwise manufacturing. The layers are tailored by developing optimized processing approaches in correlation with an iterative analysis of microstructures and properties. The layers are particularly enabling for 3D shapes. Robust joining and integration technologies were developed to allow complex shapes to be fabricated and integrated with a larger system. The critical need for tailoring interlayer compositions for optimum joint properties is highlighted and an overview of several joining approaches is provided. Another layerwise technology is in additive manufacturing which offers advantages over conventional manufacturing to provide components that are more geometrically complex, compact, multi-material, innovatively cooled, integrated, and multifunctional. The management of layered manufacturing is being conducted to better adhere layers, minimize porosity, improve properties, build complex shapes with internal cooling, and to build 3-phase coils for stator designs for electric motors.

9:40 AM

(ICACC-HON-047-2018) Effects and uses of electrical resistivity in fiber reinforced composites containing SiC, Si and C (Invited)

G. N. Morscher*[1]

1. University of Akron, Mechanical Engineering Dept., USA

Jay Singh has infiltrated the world of non-oxide composites in a big way. Much of that has centered around the development of SiC, Si and/or C containing fiber composites. Here we would like to examine the use of electrical resistance to understand the make-up of various SiC, Si and C containing composites and to assess damage accumulation. An overview of some of the findings will be presented for SiC/SiC and C/SiC made from a variety of processes and how the resistance properties of the respective composites can be used to understand something of the composite microstructure and integrity as well as matrix cracking either in the form of distributed damage or more localized crack growth at room and elevated temperatures.

10:20 AM

(ICACC-HON-048-2018) Residual and Creep Stresses in Ceramic Composites (Invited)

R. J. Kerans*[1]

1. University of Dayton, USA

The use of ceramics in myriad structural applications of broad importance to sustainable development is dependent upon the long-term viability of fibrous ceramic composites. Life management of these composites is largely uncharted territory. Among the specific challenges is the prediction and accommodation of internal stresses. Residual stresses in fiber and matrix in ceramic composites result primarily from two aspects of a composite's history; processing and creep. These stresses can have significant effects on the mechanical properties of the composite, and they can lead to significant changes in the properties over the lifetime of a component. These changes can be extremely important in understanding and predicting lifetime behavior. In this work, various possibilities for residual stress states and their evolution in service were modeled. A simple approach to plotting the constituent and composite stresses-strains was helpful in illustrating the effects. Actual composite behavior confirms the modeled behavior, and directly provides a measure of the residual stress states.

10:50 AM

(ICACC-HON-049-2018) Fundamental research on advanced ceramics boosted by next-generation aerospace and energy developments (Invited)

G. L. Vignoles*[1]; E. Bouillon[2]; P. David[3]

1. Univeresity of Bordeaux, LCTS - Lab for ThermoStructural Composites, France
2. Safran Ceramics, R & T Department, France
3. CEA, Materials R & D Dept., France

Aeronautic and space technology are currently vividly growing sectors of global economy, since they offer solutions to an exponentially increasing societal demand on transportation and communication. Historically energy- and cost-intensive, these sectors are currently committing themselves to large efforts in reducing expenses and increasing transportation efficiency. Advanced ceramic materials, and in particular Ceramic-Matrix Composites, offer many opportunities to meet these challenges. However, these materials, chosen to be used in extremely demanding conditions, require very elaborate developments at the industrial scale, which in turn trigger the need to better understand and control the basic physico-chemical and mechanical phenomena taking place during the synthesis and the use of these materials. In this talk, we will review some current efforts carried out at LCTS in understanding, modelling and controlling the fabrication and behaviour in use of some advanced ceramics such as continuous SiC-fiber reinforced ceramics, carbon/carbon composites and reinforced ceramic foams.

11:10 AM

(ICACC-HON-050-2018) Progress in polymer-derived SiC-based fibers (Invited)

T. Ishikawa*[1]; R. Usukawa[1]

1. Tokyo University of Science, Yamaguchi, Applied Chemistry, Japan

Since the first precursor process using polycarbosilane was developed, lots of polymer-derived SiC-base fibers have been developed. Through these developments, the heat-resistances of the SiC-based fibers were remarkably increased from 1200°C to 2000°C. Of these fibers, SiC-polycrystalline fibers (Tyranno SA, Hi-Nicalon Type S, and Sylramic) show the highest heat-resistance up to 2000°C, and then have been actively evaluated for the aerospace applications as SiC/SiC composites. However, to extend the application field, increase in the fiber's strength is eagerly required. Up to now, through our research, the relationship between the strength

and residual defects of the fiber, which were formed during the production processes, has been clarified. Tyranno SA is produced by heat-treatment processes of amorphous Si-Al-C-O fiber which is synthesized from polyaluminocarbosilane. During the heat-treatment processes, a decomposition of the Si-Al-C-O fiber and the subsequent sintering of the decomposed fiber proceed, accompanied by a release of CO gas and compositional changes, to obtain the dense structure. As these structural changes proceed in each filament, a strict control should be needed to minimize residual defects. The present SiC-polycrystalline fibers contain several types of defects. By a decrease in the defects, much higher strengths will be expected. In this paper, the important controlling factors will be discussed.

11:30 AM

(ICACC-HON-051-2018) Short fiber ceramic matrix composites fabricated by Fused Filament Fabrication (FFF) (Invited)

H. Klemm*[1]; J. Abel[1]; A. Michaelis[1]; M. Singh[2]

1. FhG IKTS Dresden, Germany
2. Ohio Aerospace Institute, USA

Ceramic matrix composites with SiC short fibers have been fabricated by Additive Manufacturing (AM) using a thermoplastic approach. By means of Fused Filament Fabrication (FFF) or Fused Deposition Modelling (FDM) a ceramic part is shaped layer by layer using a thermoplastic filament which is extruded through a heated nozzle. According to the degrees of freedom for movement of the printing head (and printing bed) green bodies with complex geometries can be fabricated. The possibility of reinforcing the ceramic matrix with fibers makes this AM-technology interesting for special materials like CMCs. In this study a ceramic feedstock with SiC fibers (Si-Tuff® 7-Series), SiC powder and a thermoplastic binder system was used. A tailored filament with approximately 1.8 mm in diameter was developed, fabricated by extrusion and printed by FFF to achieve first demonstration parts. After binder burnout ceramic matrix composites were fabricated by polymer infiltration and pyrolysis (PIP) and liquid silicon infiltration (LSI). Various thin-walled composites with unidirectional fiber orientation and different geometries were obtained. Finally, the materials fabricated were characterized regarding their microstructure and mechanical properties.

11:50 AM

(ICACC-HON-072-2018) Sustainability Science in a Global Landscape: implications for the field of Ceramics research (Invited)

L. Birla*[1]

1. Elsevier B.V., Netherlands

Elsevier's report on Sustainability Science in a Global Landscape, in collaboration with SciDev. Net, contributes to the understanding of sustainability science as a research field and the dialogue between science and society in sustainable development. In this relatively young field, this study establishes a baseline, both in the definition and the understanding of sustainability science, from which we may follow its progression and trajectory. Six key themes that encompass the 17 United Nations Sustainability Development Goals are examined in the report: Dignity, People, Prosperity, Planet, Justice and Partnership. Derived from this study and using in-house analytic expertise (SciVal, Scopus and Research Intelligence), an overview on sustainability topics related to ceramics and the trends over the past decade will be presented and evaluated, with a view of identifying future developments.

FS1: Bio-inspired Processing of Advanced Materials

Bio-inspired Processing II

Room: St. John
Session Chair: Di Zhang, Shanghai Jiao Tong University

8:30 AM

(ICACC-FS1-010-2018) Bio-process Inspired Synthesis of Nitrogen-Doped Anatase TiO_2 (Invited)

Z. Fu*[1]

1. Wuhan University of Technology, State Key Lab of Advanced Technology for Materials Synthesis and Processing, China

In studying and mimicking the well-defined structures or unique functions of biomaterials, scientists have succeeded in designing and synthesizing bio-inspired materials or bio-inspired functions. Furthermore, the fantastic structure-forming process in biological systems is also evolution results of many billions of years, which efficiently and accurately fabricate biominerals under environmentally benign conditions, in contrast to our present technological world where harsh conditions are commonly prerequisites. Hence, the natural structure-forming process itself is also worth learning by scientists to develop new synthesis and processing techniques for materials, which can be refereed as 'bio-process inspired synthesis and processing'. In this paper we will report a natural organism directed synthesis of N-doped TiO_2 at ambient temperature by directly implanting precursor into living mussels. The amorphous precursor transforms to N-doped anatase TiO_2 with hierarchical nanostructure in living mussels. Synthetic TiO_2 exhibits high phase stability and enhanced visible-light photocatalytic activity, owing to the modification of the inherent band gap during the in vivo mineralization. The intracellular proteins, instead of extracellular matrices, promote the crystallization process of TiO_2 by adopting a proton-transport catalysis strategy with the lowest energy barrier.

8:50 AM

(ICACC-FS1-011-2018) Metal butterfly wing scales and their plasmonic applications (Invited)

J. Gu*[1]; W. Zhang[1]; Q. Liu[1]; D. Zhang[1]

1. Shanghai Jiao Tong University, State Key Lab of Metal Matrix Composites, China

The structural characteristics of natural species have been optimized by natural selection for millions of years. They offer specific functions much more effectively than artificial approaches to some extent. The combination of natural morphologies and manually selected functional materials generates a series of structures suitable for many applications. This presentation mainly focuses on the strategies by which natural butterfly wing scales can be replicated in metals. Plasmonic properties inherited from both the sub-micrometer structures of butterflies and coupled plasmonic metals are highlighted with regard to various applications, including surface-enhanced Raman scattering (SERS) and metal-sensitized water splitting.

9:10 AM

(ICACC-FS1-012-2018) Ceria-based catalytic regeneration of wall-flow Diesel Particulate Filters made of biomorphic Silicon Carbide (Invited)

J. Ramirez-Rico*[1]; J. M. Fernandez[1]; A. Gómez Martín[1]; P. Orihuela Espina[1]; R. C. Martín[1]; J. Becerra-Villanueva[1]

1. Universidad de Sevilla, Spain

When used as substrate in Diesel Particulate Filters (DPF), biomorphic Silicon Carbide (bioSiC) made from Medium Density Fiberboard (MDF) has good filtration efficiency and a large particle storage capacity. Like any other particle filter, bioSiC DPF must

be regenerated periodically to eliminate the accumulated soot and restore the initial pressure drop. In order to explore the regeneration possibilities of this bioceramic substrate, two small-scale prototypes of wall-flow filter have been manufactured and coated with cerium oxide by Solution Combustion Synthesis (SCS). The samples were loaded with soot in a diesel engine test bench and regenerated by electric heating in an independent reactor. During the regenerating process, temperature and pressure drop were monitored, and the resulting combustion gases were analysed in order to determine the conversion rate. Results show that the catalytic combustion of soot takes place at laboratory above 200°C. When the temperature reaches 350°C, despite the soot conversion rate is still low (<20%), the pressure drop is reduced more than 40%. These preliminary results are positive and comparable with those of other catalysed commercial substrates. Further research is still needed to optimise the process and to determine if this technology is competitive in the current market.

9:30 AM

(ICACC-FS1-013-2018) Bioinspired functionally graded alumina

I. Hussainova*[1]; M. Drozdova[1]; R. Ivanov[1]

1. Tallinn University of Technology, Estonia

Multifunctionality has emerged as a strategic priority for development of novel materials. As nature provides a rich source of inspiration, a large number of approaches is related to mimicking the features of biological species, in which jointed frameworks and complex materials impart multiple functionalities integrated over a wide range of length scales. Here we report development of multifunctionally graded ceramics demonstrating gradient of both mechanical and electrical properties, which is achieved by controlled incorporation of graphene encapsulated ceramic nanofibers into a host structure. Alumina was chosen as an important structural ceramic. The composites with alternation of grain sizes in layers were produced by adding thin inter-layers of graphenated alumina nanofibers. The samples were consolidated by Spark Plasma Sintering technique at 1350 °C in nitrogen atmosphere under 50 MPa pressure. The effect of inter-layers on microstructure, mechanical and electrical properties of alumina as well as anisotropy of these properties were studied. Vickers hardness, fracture toughness and nanoindentation tests have been performed to show graduation of the mechanical properties. Materials with gradient grain size and, therefore, hardness throughout the bulk combined with highly anisotropic or directional electroconductivity are reported in this study.

9:50 AM

(ICACC-FS1-014-2018) Graphene Nanowires Anchored to 3D Graphene Foam via Self-assembly for High Performance Li and Na Ion Storage

X. Liu*[1]

1. Heilongjiang University of Science and Technology, China

Herein, we report a new member of 'graphene family', a reduced graphene nanowire on three-dimensional graphene foam (3DGNW). The novel graphene nanowires were synthesized via a template strategy involving reduction and assembly process of nanosized graphene oxides, pyrolysis of polystyrene spheres template and catalytic reaction between GO and PS decomposition products. When evaluated as anodes material for Li and Na ion batteries, the 3DGNW exhibits relatively low discharge-voltage plateau, excellent reversible capacity, rate capability, and durable tolerance. For anode of Na ion batteries, a reversible capacity of more than 301 mAh g^{-1} without capacity fading after 1000 cycles at rate of 1C were achieved. Even at rate of 20C, a high reversible capacity of 200 mAh g^{-1} can be retained. The superior electrochemical performance is ascribed to hierarchical multidimensional graphene architecture, high graphene crystallinity, expansile graphene interlayer distance, and extensively lateral exposed edges/pores, which can promote the electron and ion transport.

10:30 AM

(ICACC-FS1-015-2018) Bioinspired Design and Fabrication of Nano-Carbon Reinforced Bulk Aluminum Composites (Invited)

Z. Li*[1]; Z. Tan[1]; G. Fan[1]; D. Xiong[1]; Q. Guo[1]; Y. Su[1]; D. Zhang[1]

1. Shanghai Jiao Tong University, China

A new strategy of bio-inspired design and fabrication was explored to uniformly distribute nanocarbon in metal matrix composite and coordinate the strength-ductility dilemma. In this report, CNT/Al and GNS/Al composites with reinforcements aligned along the extrusion direction between Al lamellae was fabricated by an approach of flake powder metallurgy, which resorts to make the nanocarbons and metal matrix more compatible both in the surface properties and geometries. Then the nano reinforcement-coated Al nanoflakes were used as building blocks for stack assembly, and then a nacre-like nanolaminate structure could be eventually formed by controlled deformation processing. Compared to the composites of the same reinforcement content but random distribution, the bio-inspired nanolaminate composites exhibited a simultaneous enhancement in tensile strength, Young's modulus and uniform elongation.

10:50 AM

(ICACC-FS1-016-2018) Bioinspired approach of a Fe_2O_3/carbon composite for use in a high-performance lithium ion battery

Y. Li*[1]; D. Zhang[1]; Q. Liu[1]; J. Gu[1]; W. Zhang[1]; S. Zhu[1]

1. Shanghai Jiaotong University, School of Material Science and Technology, China

A newly bioinspired hybrid anode material based on rice husk (RHC) was developed via facile fabrication. In which, activated carbon matrix with hierarchical porous structure inherits from rice husk, nanoparticles of SnO_2 were uniformly deposited on the surface of the activated carbon with graphite layers formed in situ, and then SnO_2 was coated with carbon during the hydrothermal process. The resultant nanocomposite has shown an outstanding high rate cycling performance. The outstanding electrochemical performance is likely to be related to the synergistic effects of the unique combination of properties. These include excellent electric conductivity, hierarchical structures to enhance lithium-ion transport, nanosized particles, and large surface area, together with the surface coating of carbon on the nanoparticles, which help to alleviate the effects of volume changes, shorten the distance for Li^+ diffusion, facilitate the transmission of electrons, and keep the structure stable.

11:10 AM

(ICACC-FS1-017-2018) Bioinspired Layered Composites Based on Inorganic Nanosheets and Their Application in Flexible Energy Devices

Y. Zheng*[1]; Y. Wang[1]; J. Zhao[1]; Y. Li[1]

1. Harbin Institute of Technology, China

Inorganic nanosheets with atomic or molecular thickness and infinite planar lengths have been emerging as an ideal candidate for advanced flexible electrode materials due to their unique properties. However, it remains a great challenge to assemble inorganic nanosheets into reliable flexible electrode with enhanced electrochemical and mechanical performances by using traditional approaches. Inspired by the hierarchical layered structure and interfacial interactions of nacre, we demonstrated a nacre-like structure macroscopic fiber electrode from highly ordered assembly of transition metal oxides and carbides nanosheets by a wet-spinning process. The strong synergistic interfacial interactions of the high degree order sheets would be beneficial to efficiently utilizing the nanoscale components, which endows the electrode with excellent integrated mechanical properties and electrochemical performances in terms of volume energy densities and cyclic behaviors. This present study clearly demonstrates a novel architecture flexible electrode-design

paradigm based on inorganic nanosheets, which could open up new opportunities to develop advanced flexible energy systems.

11:30 AM

(ICACC-FS1-018-2018) Graphitized carbon materials from biomass resources as electrodes for energy storage systems

J. Ramirez-Rico*[1]; A. Gómez Martín[1]; J. M. Fernandez[1]; M. Ruttert[2]; T. Placke[2]

1. Universidad de Sevilla, Spain
2. University of Münster, Germany

Carbon obtained by pyrolysis of biomass precursors is considered hard or non-graphitizing since no subsequent heat treatment leads to ordered graphitic domains. However, by the prior addition of a transition metal such as iron or nickel into the raw precursor, the graphitization can be concurrently induced during pyrolysis from low temperatures in a single processing step. Following this approach, in this work we report a scalable and simple method to synthetize holey graphene-like sheets from medium density fiberboard wood using nickel as catalyst. The remaining metallic particles after pyrolysis were removed from the structure by acid etching, leaving in-plane nanopores into the wrinkled sheets. The performance of resulting graphitic carbons at a wide range of carbonization temperatures were further investigated as electrodes for lithium ion batteries and supercapacitor (ECs) devices providing further insight into the energy storage mechanism involved. Results showed that the capacity and energy density is directly related to the degree of graphitization, microstructure, pore structure as well as surface area. They also suggested that these materials are promising candidates for green energy storage applications in order to develop more convenient and sustainable devices to meet the future energy demand while ensuring environment protection.

FS3: Chemical Processing of Functional Materials: Understanding the Conversion of Molecular Structures to Solid-State Compounds

Single Source Precursors I

Room: Coquina Salon A
Session Chairs: Sanjay Mathur, University of Cologne; Thomas Fischer, University of Cologne

8:30 AM

(ICACC-FS3-001-2018) Solution based synthesis of advanced materials; from molecules to advanced materials (Invited)

G. Westin*[1]

1. Uppsala University, Sweden

There is an increasing demand for complex multi-functional materials of high elemental and structural complexity, often hierarchically structured with sizes down to a few nano-meters. Further, these advanced structures have to be of high quality and produced at low cost, which means few and fast processing steps why it is anticipated that molecular based solution processes will be the main way of their fabrication. Although the last decade has seen a rapid development of solution based processing routes there is still a strong need for new molecular based processes, where a firm connection between the target materials composition and micro-structure and the precursor structure and process steps in-between is present. By using low temperature synthesis and proper molecules there are also great possibilities to achieve far from thermodynamically stable doping levels, composites and inorganic materials built with memory of the molecular precursors. Here we will describe molecular based solution processes to; (i) Ln doped glasses, (ii) thin- and ultra-thin complex oxide films, and (iii) thin- and ultra-thin metal and oxide-metal composite films, and connect the final structures with the precursors and steps in-between. Some examples showing scalability of the processes through applications and up-scalings will also be given.

9:00 AM

(ICACC-FS3-002-2018) Synthesis of nanocrystalline Gd_2O_2NCN from a versatile single-source precursor (Invited)

E. Ionescu*[1]

1. Technical University Darmstadt, Materials Science, Germany

Nanocrystalline Gd_2O_2NCN (Pm1) was synthesized upon ammonolysis of bis[[(N-carboxymethyl, N-carboxy-κO-methyl) amino-κN]-ethyl]-glycinato(3-)-κN,κO]gadolinium(III) (di-ethylenetriamine pentaacetic acid gadolinium (III) dihydrogen salt or gadopentetic acid, Gd-H_2DTPA hereafter) at 900 °C. The conversion of Gd-H_2DTPA into Gd_2O_2NCN takes place in several steps, most probably via transient formation of iminodiacetate-, glycinate- and carbamate-containing complexes of Gd. Thermal treatment in air of Gd-H_2DTPA at 750 °C delivers nanocrystalline bixbyite-type Gd_2O_3 (Ia); in Ar or N_2 atmosphere the formation of monoclinic Gd_2O_3 (C2/m) was observed at 1300 °C. The synthesized Gd_2O_2NCN converts upon thermal treatment at T > 1000 °C in air, nitrogen or argon atmosphere into monoclinic Gd_2O_3 (C2/m). In ammonia atmosphere, Gd_2O_2NCN seems to be stable against decomposition, even upon prolonged exposure to 1000 °C. This study indicates that Gd-H_2DTPA may be a robust, low-cost and flexible precursor for nanoscaled Gd-based nanopowders. Moreover, precursor approaches based on metal complexes using H_5DTPA as well as related compounds such as H_4EDTA or H_4DOTA as strongly chelating ligands are suggested as promising access pathways towards nanocrystalline mixed-anion compounds in the M/O/C/N system.

9:30 AM

(ICACC-FS3-003-2018) Chemical formation and PL properties of β-SiAlON:Eu^{2+} phosphors derived from single source precursors (Invited)

Y. Iwamoto*[1]

1. Nagoya Institute of Technology, Japan

Single source precursors for β-SiAlON:Eu^{2+} phosphors were designed and synthesized by chemical modification of perhydropolysilazane (PHPS) with $Al(OCH(CH_3)_2)_3$ and $EuCl_2$ (Al/Si = 0.09, Eu/Si = 0.05). The in-situ GC-MS analysis for the reaction of PHPS with $Al(OCH(CH_3)_2)_3$ in xylene at 413 K showed evolution of $HOCH(CH_3)_2$. The subsequent TG-MS analysis resulted in the detection of the residual xylene and the low molecular weight gas species such as propene (C_3H_6) and propane (C_3H_8) at 473 to 673 K, while the elimination of HCl was observed above 1073 K. These results suggested that $EuCl_2$ facilitated the N-Al bond formation accompanied by the elimination of $HOCH(CH_3)_2$, then N-Eu bond formation could be completed above 1073 K. The single source precursor were successfully converted to β-SiAlON:Eu^{2+} by pyrolysis at 1273 K under flowing N_2 for 1 h, and subsequent heat treatment at 2073 K in N_2 (920 kPa) for 1 h. The polymer-derived β-SiAlON:Eu^{2+} phosphors exhibited a typical green emission peak centered at around 535 nm when excited at 420 nm. Further study on the chemical composition controlling at molecular scale level and the related PL properties will be shown and discussed from a viewpoint to develop novel silicon oxynitride-based phosphors through polymer-derived ceramics (PDCs) route.

10:00 AM

(ICACC-FS3-004-2018) In-situ toolbox for studying nucleation and growth in oxide thin films and hierarchical structures prepared from aqueous solutions (Invited)

A. B. Blichfeld*[1]; T. D. Vu[1]; T. Grande[1]; M. Einarsrud[1]

1. Norges Teknisk-naturvitenskapelige Universitet, Department of Materials Science and Engineering, Norway

In a recently started project, From Aqueous Solutions to oxide Thin films and hieratical Structures, we are aiming at developing an aqueous synthesis platform for thin films (TFs) and hierarchical structures based on an in-situ characterization toolbox. One of the tools is a cell for hydrothermal studies using synchrotron XRD. $MNbO_3$(M=K,Na) was investigated, and the formation of phase pure $KNbO_3$ was realized. In the initial stage of formation for Na, a large number of phases were only resonantly resolved; fast detector and synchrotron XRD made it possible to investigate these on a sub-second scale. Other studies with the setup involves formation of TiO_2 where surface functionalization was shown to facilitate growth by the orientated attachment mechanism. In one of our recent studies on TFs of $Bi_{0.5}Na_{0.5}TiO_3$, highlights the need for high control of processing parameters for obtaining phase pure materials. Ex-situ studies might not give the full picture, and an in-situ cell for the pyrolysis step in chemical solution deposition processing of TFs has been developed. Understanding the evolution of the nucleation and growth of the thin film will aid designing the procedure for fabrication and tuning the desired piezoelectric properties. $BaTiO_3$ has been used studied, where the degree of preferred orientation can be tuned via the pyrolysis procedure.

Single Source Precursors II

Room: Coquina Salon A

Session Chairs: Yuji Iwamoto, Nagoya Institute of Technology; Ralf Riedel, TU Darmstadt

10:50 AM

(ICACC-FS3-005-2018) Atomic/molecular layer engineering of novel inorganic-organic thin-film materials

M. Karppinen*[1]

1. Aalto University, Department of Chemistry and Materials Science, Finland

Hybrid inorganic-organic materials have the capacity to exhibit tailored combinations of properties even beyond those traditionally seen for inorganics or organics separately. An elegant, yet industrially feasible way to build up such materials in atomic/molecular level accuracy is to combine the state-of-the-art gas-phase thin-film deposition technique of inorganics, i.e. ALD (Atomic Layer Deposition), with the emerging MLD (Molecular Layer Deposition) technique for organics. The combined ALD/MLD technique enables the atomic/molecular layer-by-layer fabrication of inorganic-organic thin films through sequential self-limiting gas-surface reactions of gaseous precursors. We have developed ALD/MLD processes for a rich variety of hybrid materials with different inorganic and organic constituents. In particular, we have employed the ALD/MLD technique to fabricate flexible hybrid thin-film materials for e.g. textile-integrated oxide-organic thin-film thermoelectrics in which the periodically introduced ultrathin organic layers between oxide layers hinder phonon transport, an all-solid-state Li-organic microbattery, and exotic luminescence applications. Also discussed are the new directions foreseen for the ALD/MLD technique related to metal organic framework (MOF) and guest-accessible coordination-framework materials.

11:10 AM

(ICACC-FS3-006-2018) Subvalent Iridium Precursors for Atom-Efficient Chemical Vapor Deposition of Ir and IrO_2 Thin Films

L. Jürgensen*[1]; M. Frank[1]; M. Pyeon[1]; L. Czympiel[1]; S. Mathur[1]

1. University of Cologne, Institute of Inorganic Chemistry, Germany

Due to the applications in catalytic oxygen evolution reactions (OER) and optoelectronics iridium and iridium dioxide thin films are of growing interest. New heteroleptic Ir(I) compound exhibiting high volatility and defined thermal decomposition under CVD conditions is reported. The new iridium precursor [(COD)Ir(ThTFP)] (COD = cyclooctadiene, ThTFP = (Z)-3,3,3-trifluoro-1-(thiazol-2-yl)prop-1-en-2-olate) unifies both reactivity and sufficient stability through its heteroleptic constitution to provide a precise control over compositional purity in CVD deposits. The solution integrity of monomeric Ir(I) complex was investigated by 1D and 2D NMR spectroscopy, EI mass spectrometry, whereas the molecular structure was confirmed by single crystal diffraction. CVD experiments demonstrated the suitability of the iridium compound for an atom-efficient (high molecule-to-precursor yield) gas phase deposition nanocrystalline iridium films that could be converted into crystalline iridium dioxide upon heat treatment to demonstrate their electrocatalytic potential in oxygen evolution reaction.

11:30 AM

(ICACC-FS3-007-2018) In-situ Analysis of Precursor Decomposition Patterns in Chemical Vapor Deposition Reactions

T. Fischer*[1]; M. Grosch[1]; S. Mathur[1]

1. University of Cologne, Institute of Inorganic Chemistry, Germany

Chemical Vapor Deposition (CVD) is a well-known and widely studies technique for thin-film processing as well as fabrication of one-dimensional nanostructures. The selection of target materials is closely coupled to accessible precursor chemistries and understanding of precursor decomposition pathways. Most metal organic CVD processes are based on complex precursors, where the ligand sphere is drastically influencing the thermal decomposition and thin-film formation behavior. Directly coupled in-situ quadrupole mass spectrometry enables the analysis of airborne molecular species and fragments formed during CVD processes as well as assessing their individual temporal evolution during the reaction. By direct correlation of mass spectrometrical data with surface temperatures and reactor pressures under varying process conditions more profound understanding of precursor chemistry and thin-film formation can be established. This presentation will focus on the formation of metal oxide thin-films by thermal decomposition of homo- and heteroleptic metal alkoxides and β-arylalkenolates, respectively. Possible precursor reaction pathways and the influence of decomposition products on the film formation are discussed.

S1: Mechanical Behavior and Performance of Ceramics & Composites

Development, Testing and Modeling of Ceramic and Metal-Ceramic Systems

Room: Coquina Salon D

Session Chairs: Charles Lewinsohn, CoorsTek, Inc.; Michael Jenkins, Bothell Engineering and Science Technologies

1:30 PM

(ICACC-S1-057-2018) Microstructure-based modeling of the ultimate tensile strength of ceramic matrix composites

E. Maillet*[1]; D. Dunn[1]

1. GE Global Research, USA

Ultimate tensile strength (UTS) is one of the key mechanical properties controlling performance. In ceramic matrix composites

(CMCs), UTS depends not only on the strength of the reinforcing fibers but also on attributes of the microstructure that determine local fiber stress. These attributes include fiber radius, coating thickness, distance between fibers, fiber and matrix moduli. Existing models for UTS predict the behavior of fibers with average attributes. However, the microstructure of CMCs varies locally and these changes are expected to affect UTS. The present paper proposes a new approach to model UTS. The stress on each fiber is estimated analytically based on local microstructural characteristics. UTS is then predicted using a progressive failure model. Effects of local microstructure variations on UTS are discussed.

1:50 PM

(ICACC-S1-058-2018) High temperature bending behavior of SiC/Si-binary eutectic alloys

T. Tsunoura*[1]; K. Yoshida[2]; T. Yano[2]; T. Aoki[3]; T. Ogasawara[4]

1. Tokyo Institute of Technology, Department of Materials Science and Engineering, Japan
2. Tokyo Institute of Technology, Laboratory for Advanced Nuclear Energy, Japan
3. Japan Aerospace Exploration Agency, Aeronautical Technology Directorate, Japan
4. Tokyo University of Agriculture and Technology, Department of Mechanical Systems Engineering, Japan

SiC fiber-reinforced composites fabricated by Si melt infiltration method have received attention as high temperature structural materials for jet engine components. Amorphous SiC fibers have not been applied in the composites using Si due to high process temperature and fiber degradation. In our previous studies, amorphous SiC fiber-reinforced Si binary eutectic alloys composites (SiC_f/alloy) have been successfully fabricated by melt infiltration method below 1400°C using Si binary eutectic alloy. In this study, high temperature bending behavior of the SiC_f/alloy and SiC particle-reinforced Si binary eutectic alloys composites (SiC_p/alloy) was evaluated. Three-dimensional preforms consisting of amorphous SiC fibers were used as the reinforcement, and the fibers were coated with CVI-C as an interphase and CVI-SiC for protecting the CVI-C and the matrix. Then, various Si binary eutectic alloys were infiltrated into the preform. SiC_p/alloy were also fabricated using the eutectic alloys. Bending test were carried out up to 1200°C. The SiC_p/alloy showed large fracture strain at high temperature due to ductile transition of silicides. SiC_f/alloy showed higher maximum bending stress than that of SiC_f/Si because amorphous SiC fiber was not degraded and high toughness of the matrices was expected at high temperature.

2:10 PM

(ICACC-S1-059-2018) Effects of Cooling Hole Arrays in SiC/SiC Composites

G. C. Ostdiek*[1]

1. US Air Force, AFRL/RQTI, USA

This paper describes research on SiC/SiC CMC composites with cooling holes similar to those used in hot section gas turbine engine turbomachinery. Some research has been reported on single holes in CMC material, but none on multiple holes. For this research, ANSYS and Air Force Research Laboratory B-Spline Analysis Method composite damage modeling software were used to model and predict failure in various hole patterns including 0, 1, and multiple holes. Several multiple-hole patterns were modeled, with a standard design for metallic turbomachinery cooling hole geometry used as a baseline design. Modeling included room and high temperature environments, with through-thickness and in-plane thermal gradients from the effects of cooling air blown through the cooling holes, as it would be in actual turbomachinery. It is believed that cooling holes in actual turbomachinery would experience some temperatures in the pesting regime with resulting increased oxidation issues. This may be exacerbated by a lack of environmental barrier coating in drilled cooing holes. The results from this research can be expected to contribute to a design guide for turbine cooling hole diameter and spacing for this material, and provide insight into vulnerability to environmental degradation.

2:30 PM

(ICACC-S1-060-2018) Novel manufacturing process for short fiber reinforced oxide/oxide CMCs

G. Puchas*[1]; S. Knohl[1]; W. Krenkel[1]

1. University of Bayreuth, Ceramic Materials Engineering, Germany

Fabrics are the most commonly used type of fiber preform in oxide/oxide CMCs, because these ordered fiber structures enable high fiber volume contents (> 35 vol.%) and therefore high material strengths (> 250 MPa). However, due to the restricted drapability of the fabrics, the complexity in shape of components based on fabrics is limited and they are generally anisotropic due to the 0 ° / 90 ° - orientation of fibers in the fabric. Short fibers as reinforcement for CMCs offer a high degree of freedom regarding the shape complexity of components but are connected with a decline in strength of the material. Contrary to non-oxide CMCs such as C/C or SiC/SiC, little research has been done on short fiber reinforced oxide/oxide CMCs. A novel, semi-automatic manufacturing process for short fiber reinforced oxide/oxide CMCs using Nextel™ 610 fibers and an alumina-zirconia slurry was developed. This in-situ process combines the infiltration and placement of the short fibers on a profile to form complex near net shape components in one step. Selected tiles and complex components produced via the aforementioned manufacturing process were evaluated by µ-CT and SEM regarding their homogeneity and the distribution of fiber orientation in said components. The novel manufacturing process will be compared to an already established prepreg process regarding material properties, costs and possibilities of component design.

2:50 PM

(ICACC-S1-061-2018) $Ba_{1-x}Sr_xTiO_3$ Reinforced Cu Matrix Composite for Electronic Packaging Applications

S. Kumar[1]; A. Dwivedi[1]; M. Ahmad*[1]

1. Indian Institute of Technology, Ceramic Engineering, India

For thermal management in electronic devices, materials with high thermal conductivity and co-efficient of thermal expansion (CTE) matching with that of the device is needed. SiC reinforced metal matrix (Cu, Al) composites are one of the most desirable material for level 1 packaging. In order to match the thermal expansion of devices the reinforcements of SiC particulate can be is as high 75%. Cu is having lower CTE and greater thermal conductivity than Al, however Cu tend to react with SiC during processing. $Ba_{1-x}Sr_xTiO_3$ (BST) has a low coefficient of thermal expansion and it exhibits negative thermal expansion during tetragonal to cubic transformation. The tetragonal to cubic transformation temperature can be regulated between room temperature to 120 ºC by changing Sr/Ba ratio. We have synthesised BST reinforced Cu matrix composite with varying BST fraction and achieved densities in excess of 90% by sintering in 5%H_2 atmosphere. Composite having 40% BST fraction exhibits a thermal conductivity as high as 292.213 $Wm^{-1}K^{-1}$ and a CTE value of 6.41x10^$^{-6}$ K^{-1}. These results fare better with the reported values of thermal conductively (~170 W/m*K) and CTE (~7-8x10^$^{-6}$ K^{-1}) of Al-50%SiC. These results show that BST-Cu can dissipate heat more effectively at the same time will develop less thermal stress than the Al-SiC composites.

3:30 PM

(ICACC-S1-062-2018) Out-of-plane electrical transport properties in conducting ceramic matrix composites

Y. P. Singh*[1]; R. Mansour[2]; G. N. Morscher[1]

1. University of Akron, Mechanical Engineering, USA
2. Teledyne Scientific Company, Composite Materials, USA

To fully exploit the method of direct current potential drop technique, a reliable measurement of out-of-plane electrical properties of ceramic matrix composites is essential. However, this task becomes challenging for multiple reasons including the architectural complexity of these materials, and different electrical conductivity of constituents. In addition, the small thickness of most CMC panels imposes physical difficulties on measuring the out-of-plane electrical resistivity using the conventional four linear probe methods, while the two-probe method requires the accurate knowledge of different contact resistances involved. This work provides an indirect method for determining the out-of-plane electrical resistivity for CMC materials utilizing the data from in-plane electrical resistivity. The results obtained from the indirect method were compared to the values acquired from direct measurements of the out-of-plane electrical resistivity.

3:50 PM

(ICACC-S1-063-2018) Development of an Advanced Composite Consisting of Iron Matrix Reinforced with Ultra High Temperature Ceramic Particulate (TiB_2) with Optimum Properties

B. Jahani*[1]; M. Salimijazi[1]; F. Azarmi[1]; A. Croll[2]

1. North Dakota State University, Mechanical Engineering, USA
2. North Dakota State University, Physics, USA

This study was intended to investigate the mechanical properties and microstructure of iron based composite reinforced by ultra-high temperature ceramics fabricated by powder metallurgy technique. The fabrication parameters were optimized and composite samples with different volume fraction of TiB_2 were fabricated and were subjected to different mechanical tests. The results indicated improving in mechanical properties of Fe-TiB_2 composites by increasing the volume fraction of TiB_2 up to 20 vol%. More TiB_2 particles didn't improve the mechanical properties of composite, instead adversely affected it due to increasing the chance of agglomeration and porosity in entire microstructure. Another finding showed the twofold characteristic of TiB_2 on mechanical properties of composite via increasing the hardness and decreasing the bulk density of composite. Finite Element Analysis (FEA) have also been performed on microstructural based developed models to simulate failure of composites. Numerical simulation results could verify the experimental results.

4:10 PM

(ICACC-S1-064-2018) Commercial Development of C/SiC and SiC/SiC Composites Using Melt-Infiltration

K. Machida[2]; E. Ness[1]; S. Aonuma[2]; C. Lewinsohn*[1]; H. Nakanishi[2]

1. CoorsTek, Inc., USA
2. CoorsTek K.K., Japan

CoorsTek Inc.is the world's largest manufacturer of technical ceramics and supplies over one hundred types of engineered ceramic materials, including several compositions of monolithic silicon carbide (SiC). CoorsTek, has extensive experience processing silicon carbide via several routes, including CVD, reaction bonding, and melt infiltration for commercial production of silicon carbide wafer boats and wafer manipulators. At CoorsTek KK, in Japan, this core competency has been leveraged to accelerate production of silicon carbide matrix composites such as carbon fiber reinforced brakes for performance cars. The most recent evolution of this technology is the development of continuous SiC fiber ceramic composites, using the silicon melt infiltration process developed for automotive brakes and semiconductor products. Improved Tyranno SA woven fabric from Ube has been used as reinforcement. After coating fibers by a proprietary process, fabric was dipped in a silicon carbide slurry and dried to produce a prepreg. The fabric was cut into sections, stacked, and pressed at elevated temperature to form a laminated body. Following a burnout process, silicon infiltration was performed to produce a body with low porosity. Results from SEM and optical microscopy, as well as room-temperature flexural strength tests, will be presented.

4:30 PM

(ICACC-S1-065-2018) The effects of Y_2O_3 addition on mechanical and electrical properties of Al_2O_3/Ti composites

S. Shi*[1]; T. Sekino[1]; T. Goto[1]; S. Chou[1]

1. Osaka University, Japan

Among various ceramic/metal composites, Al_2O_3/Ti system is one of the most promising materials not only for advanced structural parts but also for biomaterial applications. However, the interfacial reaction products, such as TiAl and Ti_3Al intermetallic compounds, Ti-O and $TiAl_2O_3$ compounds weaken mechanical properties. In order to inhibit the interfacial reaction, we chose Y_2O_3 was used as an effective sintering aid. In Al_2O_3-based ceramics, Y_2O_3 was known to segregate to grain boundary and react with Al_2O_3 to form stable $Al_5Y_3O_{12}$ (YAG) which shows superior high fracture toughness, high Vickers hardness and high temperature creep resistance. YAG phase can homogenize stress field distribution and plays an important role in crack deflection. In addition of these effects, the decrease of Ti particles reacting with Al_2O_3 is considered to contribute to the further increase in electrical conductivity. Therefore, in current study, Al_2O_3/Ti composites with 1wt%-5wt% Y_2O_3 were fabricated by hot-pressing sintering (HPS) method at 1500 °C in Ar atmosphere. The formation of $Al_5Y_3O_{12}$ (YAG) was identified by XRD analysis. SEM observation revealed refined microstructure and decreased Al_2O_3 grain size. The relations between mechanical and electrical properties and Y_2O_3 addition will be discussed.

S3: 15th International Symposium on Solid Oxide Fuel Cells (SOFC): Materials, Science and Technology

Anode I

Room: Crystal
Session Chair: Tai-Nan Lin, Institute of Nuclear Energy Research

1:30 PM

(ICACC-S3-046-2018) Perovskite Oxide Materials for Solid Oxide Fuel Cell Anodes (Invited)

S. Barnett*[1]

1. Northwestern University, USA

Solid oxide cell anodes that contain only oxide phases are desirable to avoid problems with Ni-based anodes, including coking in hydrocarbon fuels and degradation due to fuel impurities or redox cycling. This talk will describe various perovskite oxides that provide good anode performance. An electrochemical model will be discussed and it is shown that anode performance is often limited by the dissociative adsorption of hydrogen. One way to improve such anodes is by the addition of a reducible cation in the oxide formulation, resulting in the formation of performance-enhancing metallic nanoparticles on oxide surfaces in situ during cell startup and operation. For example, polarization resistance equal to that of state of the art Ni-YSZ has been observed for $Sr(Ti,Fe,Ni)O_3$ anodes where Ni-Fe nanoparticles form. This talk will examine the microstructural evolution of these "exsolution anodes," and discuss how exsolved metal nanoparticles enhance electrochemical performance by promoting hydrogen dissociation.

2:00 PM

(ICACC-S3-047-2018) Composite Oxide containing $La_{0.5}Sr_{0.5}Mn_{0.9}Al_{0.1}O_3$ for Active Anode for Dry Hydrocarbon type Solid Oxide Fuel Cells (Invited)

T. Ishihara*[1]; A. M. Bahrain[2]

1. Kyushu University, International Institute for Carbon-Neutral Energy Research, Japan
2. Kyushu University, Department of Applied Chemistry, Japan

Direct utilization of hydrocarbon for Solid Oxide Fuel Cells (SOFCs) using oxide ion conductor have the advantage of cost, high energy conversion efficiency and expanding the application area like vehicle APU. By eliminating the initial external reforming process of hydrocarbon fuels to syngas, significant advantages in cost and simplicity of gas handling process can be expected. In this study, Co and Al co-doping to $La_{0.5}Sr_{0.5}MnO_3$ (LSM55) for anode were studied and it was found that co-doping Co and Al is effective for decreasing anodic IR loss and the power density could be much increased by doping Co for Mn site of LSM doped with Al. However, power density is still not high because of low surface activity. Mixing oxide ion conductor to Co and Al doped $LaMnO_3$ anode was further studied for increasing power density. Among the examined mixed conductor, it was found that mixing Sm doped CeO_2 is effective for increasing power density and the MPD of 850 mW/cm^2 was achieved at 1173 K at 20 wt% SDC mixed with Co and Al doped LSM anode. Impedance and current interruption analysis suggests that while anodic overpotential is dominant on Al and Co doped LSM anode, it is decreased significantly by mixing SDC and so the increased anodic performance could be assigned to increased electrical conductivity by partial electronic conductivity of CeO_2.

2:30 PM

(ICACC-S3-048-2018) Enhanced Stability of Infiltrated Nickel Catalyst Particles in Ni-YSZ Anodes

Y. Lu*[1]; P. J. Gasper[1]; B. Mo[1]; S. Gopalan[1]; U. Pal[1]; S. Basu[1]

1. Boston University, Material Science Engineering, USA

Liquid infiltration of nanoparticle catalysts in the anode electrode has been shown to improve SOFC performance in various ways, such as increasing catalytic performance, improving sulfur tolerance, and enabling use of hydrocarbon fuels. However, these nanoparticles are unstable during SOFC operation. This study examines the stability of infiltrated nickel nanoparticle catalysts in Ni-YSZ cermet anodes during electrochemical testing. Specifically, the particle stability and effect on electrochemical performance of infiltrated nickel and Gadolinium Doped Ceria (GDC) or nickel and Niobium Doped Strontium Titanate (STN) were exmained. Electrochemical testing involving I-V and EIS measurements were made under various operating conditions. For each operating condition, SEM images of fractured cross sections of electrochemically tested cells and untested cells were compared to quantify particle stability during electrochemical testing. High resolution TEM results show the impact of sequential infiltration versus coinfiltration of metallic and oxide phases. Results show that the sequential infiltration of oxide phases improves particle stability and electrochemical performance.

Anode II

Room: Crystal
Session Chair: Mihails Kusnezoff, Fraunhofer IKTS

3:10 PM

(ICACC-S3-050-2018) Degradation of solid oxide cells: CO_2 electrolysis and carbon formation (Invited)

T. L. Skafte*[1]; C. Graves[1]

1. Technical University of Denmark, Energy, Denmark

The solid oxide electrochemical cell is a promising technology for efficient energy storage, but large-scale employment is currently hindered by limited durability. Targeting niche applications initially will allow for gradual employment of economies-of-scale and facilitate entry into larger markets. One such case is electrolysis of CO_2 into CO and O_2, which is relevant for decentralized CO production for e.g. specialty chemicals, or even rocket fuel production on Mars. However, lifetime and efficiency of the cells is limited due to degradation and failure of the commonly employed nickel electrocatalyst. With cell testing and multi-physics modeling we show that sulfur poisoning and carbon formation are especially problematic for this specific mode of operation and severely limits the realizable efficiency. We further elucidate the mechanism responsible for an observed inhibition of carbon formation on doped ceria using operando x-ray photoelectron spectroscopy and density functional theory calculations. The accumulated knowledge gathered from these studies paved the way for development of a novel Ni-free cell design, which is carbon tolerant and redox activated.

3:40 PM

(ICACC-S3-051-2018) Modification of Electrodes in Solid Oxide Electrochemical Cells for Electricity Storage with Syngas Production

Y. Zhang*[1]; M. Han[1]

1. Tsinghua University, State Key Laboratory of Power Systems, Department of Thermal Engineering, China

Solid oxide electrochemical cells (SOCs) are regarded as efficient devices for energy storage and conversion. It can operate in both fuel cell and electrolysis mode. Recently, methane assisted solid oxide electrolysis cell (SOEC) becomes highly promising because it reduces the electricity consume and utilizes the produced O_2. However, studies on charge/discharge characteristic of a single cell under continuous methane assisted SOEC and fuel cell modes are few. In this work, charge/discharge characteristic and electrode performance of a tri-layer structure SOC is investigated, where $La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_{3-\delta}$ (LSCF) nanoparticles are infiltrated to cathode backbones of NiO/YSZ|YSZ|porous-YSZ substrate. In discharge, methane is sent to anode and air is sent to cathode to generates electricity; whilst in charge, air is substituted by H_2O and/or CO_2 in cathode to storage electricity and generates syngas. Ni/YSZ anode has been modified by BaO and GDC using infiltration method to prevent carbon deposit. Excellent electrochemical performance and long-term stability are obtained. Then, LSCF cathode performance in both air and H_2O/CO_2 atmosphere is evaluated. Last, charge/discharge cycle stability of the cell are conducted. It is found that after initial degradation, the cell presents well stability over multiple cycles.

4:00 PM

(ICACC-S3-052-2018) Nanostructured ceria as an exceptionally active and stable mixed-conducting electrocatalyst

C. Graves*[1]

1. Technical University of Denmark, Department of Energy Conversion and Storage, Denmark

The solid oxide electrochemical cell can efficiently store renewable electricity in the form of hydrogen, methane and other fuels by electrolysis of steam/CO_2, and also operate reversibly to produce electricity from fuels. A major obstacle to commercialization is insufficient device lifetime due to loss of electrode performance. State-of-the-art nickel-based fuel-electrodes suffer from gradual deactivation that is largely due to mobility of nickel and can even be completely destroyed by oxidation and carbon deposition. We have been developing nanostructured ceria based electrodes that overcome these issues and outperform conventional electrodes. In the fuel environment, acceptor-doped ceria is a mixed ionic-electronic conductor with a high concentration of oxygen vacancies, which provide a high density of surface reaction sites. Our high surface area ceria electrodes can provide a polarization resistance of 0.01 Ω cm^2 at 600 °C, an order of magnitude lower than conventional

nickel-based electrodes. The electronic conductivity and chemo-mechanical stability of ceria is too low to function by itself as a practical electrode, so it must be used in a composite with a more stable, electron-conducting material. This presentation will report electrochemical and structural details of our nanostructured ceria electrocatalysts and summarize our progress in integrating them into practical devices.

4:20 PM

(ICACC-S3-053-2018) In-depth study of the poisoning effects for H_2S and CO_2 on the Hydrogen Electrode for Proton Conducting SOFC

S. Sun*[1]; Z. Cheng[1]

1. Florida International University, Mechanical and Materials Engineering, USA

Previous studies revealed that the hydrogen electrodes of proton conducting SOFC appear to show much better resistance against H_2S (at ppm level) compared to conventional oxide-ion conducting SOFC while get poisoned by CO_2 (at low percentage level) which is often not regarded as a poison. In addition, when using both anode-supported full cell (ASC) of Ni - $BaZe_{0.1}Ce_{0.7}Y_{0.1}Yb_{0.1}O_3$ (BZCYYb)/BZCYYb/ $La_{0.6}Sr_{0.4}Co_{0.8}Fe_{0.2}O_3$ (LSCF) and anode symmetrical cell (SymC) of Ni-BZCYYb/BZCYYb/Ni-BZCYYb, different electrochemical responses had been observed for H_2S: it does not seem to negatively impact the anode-supported full cell, which has a thicker anode (~600 um) while yields considerable poisoning for the anode symmetrical cell with a thinner anode (~30 um). In comparison, consistent poisoning behavior for CO_2 was observed on both types of cells. In this study, an in-depth investigation on the effects of anode as well as electrolyte thickness on the H_2S and CO_2 poisoning effects was conducted using electrolyte-supported full cell (ESD), and the electrochemical responses were recorded in the temperature range of 750°C to 450°C. The experimental results on all three types of cells (ASC, ESC, and SymC) will be presented and their implications on the electrocatalytic roles of proton conducting oxides in hydrogen electrode reaction for proton conducting SOFCs will be discussed.

S5: Next Generation Bioceramics and Biocomposites

Bioceramics and Biocomposites III

Room: Coquina Salon B

Session Chairs: Akiyoshi Osaka, Okayama University; Chloe Goldbach, NovaBone Products; Priscila Melo, Newcastle Univeristy

1:30 PM

(ICACC-S5-017-2018) Cube honeycomb models for porous bone architecture: FEM simulation (Invited)

A. Osaka*[1]; C. Xuewen[2]; G. Wang[2]; M. Todo[3]; S. Guo[2]; J. Wang[2]

1. Okayama University and Henang University of Science & Technology, Faculty of Engineering, Japan
2. Henang University of Science and Technology, Sch Mat Sci & Eng, China
3. Kyushu University, Dept Molecular & Material Sciences, Japan

Mechanical property of porous scaffolds is no less important than the chemical and biological ones, such as tissue or cell compatibility, tissue growth induction in association of cell migration, controlled biochemical stability, and so forth. We propose here a cubic honeycomb body (1x1x1 cm^3) composed of 512 small cubes (8x8x8) of ~100 μm thick edges as a zero-th order model of porous materials. We can control the mechanical properties by modifying the raw material itself or the edge thickness. We derived the equivalent stress ($s_{v\,max}$) of von Mises theory from Finite Element Method calculation (ANSYS; Pa, USA) for polyethylene (PE) with varied number of lost transvers-edges and edge thickness, assuming Y = 1.1 GPa and Poisson's ratio = 0.4 for PE. Certain uniaxial load 5~10 N was added in the z-axis. $s_{v\,max}$ depended little on the loose of the transvers edges (0 ~ 16 edges lost) but it decreased rapidly with the edge thickness giving a mild break around ~60 μm. This suggested the critical edge thickness leading to cracking.

1:50 PM

(ICACC-S5-018-2018) An Injectable Glass Polyalkenoate Cement Engineered for Fracture Fixation and Stabilization

B. A. Khader*[1]; S. A. Peel[2]; M. Towler[1]

1. Ryerson University, Mechanical and Industrial Engineering, Canada
2. University of Toronto, Division of Oral & Maxillofacial Surgery & Anaesthesia, Faculty of Dentistry, Canada

Glass polyalkenoate cements (GPCs) have potential as bio-adhesives due to their ease of application, appropriate mechanical properties, radiopacity and chemical adhesion to bone. Aluminum (Al)-free GPCs have been discussed in the literature, but have proven difficult to balance injectability with mechanical integrity. For example, zinc-based, Al-free GPCs reported compressive strengths of 63 MPa, but set in under 2 min. Here, the authors design injectable GPCs (IGPCs) based on zinc-containing, Al-free silicate compositions containing GeO2, substituted for ZnO at 3% increments through the series. The setting reactions, injectability and mechanical properties of these GPCs were evaluated using both a hand-mix (h) technique, using a spatula for sample preparation and application and an injection (i) technique, using a 16-gauge needle, post mixing, for application. GPCs ability to act as a carrier for bovine serum albumin (BSA) was also evaluated. Germanium (Ge) and BSA containing IGPCs were produced and reported to have working times between 26 and 44 min and setting times between 37 and 55 min; the extended handling properties being as a result of less Ge. The incorporation of BSA into the cement had no effect on the handling and mechanical properties, but the latter were found to have increased compression strength with the addition of Ge from between 27 and 37 MPa.

2:10 PM

(ICACC-S5-019-2018) Processing of a fibre reinforced composite using PLLA and Phosphate glass fibres for medical applications

P. Melo*[1]; M. Dalmina[2]; A. Ferreira-Duarte[1]; P. Gentile[1]; M. German[3]; M. Marshal[4]; K. Dalgarno[1]

1. Newcastle Univeristy, School of Engineering, United Kingdom
2. Newcastle University, School of Chemistry, United Kingdom
3. Newcastle University, Dental School, United Kingdom
4. Glass Technology Services, United Kingdom

Composites have been introduced for clinical application for their ability to mimic the hierarchical structure of human tissues. The combination of two different materials with desired properties is a main advantage, namely for load bearing applications, eliminating problems such as stress shielding and post-implantation fractures, common in metals. The use of degradable matrices reinforced by stronger materials is seen as a viable solution for the problem. Balancing the materials ratios allows the control of the degradation rate and consequently of the mechanical strength. In this study a medical graded PLLA was used as matrix while phosphate glass fibers were used as reinforcement, on a 5wt%. Rods of composite were made by melting PLLA granules mixed with fibers at 200°C in a mould. The rods were then pelletized and processed to create a filament with random diameter using a twin screw extruder. The resultant filament was pelletized and used as feedstock for compression moulding. The material was successfully produced but results are under analysis. The mechanical properties and material degradation will be assessed and the structural integrity of the polymer analysed by GPC.

2:30 PM

(ICACC-S5-020-2018) A Closer Look at Binary Boron-Rubidium Glasses

K. O'Connell*[1]; U. Werner-Zwanziger[2]; D. Boyd[1]

1. Dalhousie University, Allied Oral Sciences, Canada
2. Dalhousie University, Department of Chemistry and Institute for Research in Materials, Canada

Of recent, there is an increased interest into boron-based glasses for medical applications where the degradability of these networks can be utilized. This is most prominent in areas of wound healing and bone regeneration, provoked primarily by the (i) controlled release of therapeutically useful inorganic ions, and the (ii) transient nature of these glasses. The network degradation mechanisms are thought to primarily be a congruent process of network hydrolysis. To gain a greater insight into and control degradation, we utilized a known binary x B_2O_3 - (1-x) Rb_2O glass series, ranging in 5 – 40 mol.% Rb_2O. ^{11}B MAS NMR was used to determine the relative fraction of B[4] and B[3] in each glass. Subsequently, 3 glasses were chosen for degradation studies. XRD, SEM, and ^{11}B and ^{87}Rb NMR were used to monitor the structural changes that occurred due to exposure to water. It was found that a portion of each glass crystalized, primarily during the first minute and that the level of crystallization varied (ranging from 75-25%) with the Rb content: increased Rb_2O content (up to 30 mol.%) caused a decrease in the level of network crystallization, maintaining amorphous nature. This spontaneous crystallization adds to our understanding into the complex nature of boron glasses, their dissolution characteristics, and potential future applications.

2:50 PM

(ICACC-S5-021-2018) Characterization of Spray Dried, Sol-Gel Derived Bioactive Glasses for Hemostatic Applications

C. M. Goldbach*[1]; M. Demir[1]; D. Rodriguez[1]; L. Howell[2]

1. NovaBone Products, Research & Development, USA
2. Particle Solutions, USA

Purpose: Sol-gel derived bioactive glasses (45S5) intended for hemostatic applications were produced via spray drying and other processes. These glasses were characterized for surface area, pore size, particle morphology, particle size, absorption capacity, and dissolution rate. Methods: After spray drying, the glasses were processed with UV and/or a hydrogen peroxide soak followed by heating at ≥300C. Surface area and pore structure were analyzed via nitrogen gas adsorption. Absorption capacity testing was conducted at NAMSA and NovaBone. In dissolution testing, the glasses were immersed in SBF at 37C to assess dissolution percentage based on weight change. SEM micrographs provided particle size and particle morphology. Results: Surface area and pore structure data was collected; the surface area results ranged from 30-80m2/g. The average particle size is 18μm, with all particles <25μm. The SEM micrographs showed the spherical morphology of the glass powders. Additional testing is needed for absorption capacities and dissolution rates. Conclusions: The glass treatments resulted in increased surface area and porosity. The spray drying process produced particles <25μm in size with spherical morphology. The glasses have high absorption and delayed dissolution but further testing is needed. The next step iwill be to further investigate the hemostatic properties in animal models.

3:30 PM

(ICACC-S5-022-2018) Bone Reconstruction in the Extraction Sockets of Diabetic Patients Grafted with SCPC Bioactive Ceramic

A. El-Ghannam*[1]; H. ElShamy[2]; K. Allam[2]

1. University of North Carolina at Charlotte, USA
2. Cairo Univerity, Oral Surgery, Egypt

Oral rehabilitation of diabetic patients is challenged by impaired bone formation in extraction sockets and reduction in alveolar ridge height. We used silica-calcium phosphate (SCPC) granules to enhance bone regeneration in extraction sockets in type 2 diabetic patients. Thirty male patients were divided into 3 groups: (A, n=10) diabetic patients grafted with SCPC granules, (B, n=10) diabetic patients without graft and (C, n = 10) non diabetic patients grafted with SCPC. SCPC was well tolerated in all patients as indicated by normal color and texture of the covering mucosa and absences of infection, allergic reaction, or ulceration. Direct digital radiography system showed a significantly higher increase in bone density from 2-6 months postoperatively in SCPC grafted groups compared to control ungrafted one. Statistical analysis showed comparable bone density in sockets grafted with SCPC in groups A and C indicating the strong stimulatory effect of SCPC on new bone formation in diabetic patients. The capacity of SCPC to correct the abnormal bone and mineral metabolism in diabetic patient is attributed to the stimulatory effect of SCPC on stem cell differentiation.

3:50 PM

(ICACC-S5-023-2018) Systematic Study on Solid-State Synthesis of Monticellite ($CaMgSiO_4$) Based Bioactive Ceramic Powders Obtained from Boron Derivative Waste

L. Koroglu*[1]; E. Ayas[1]

1. Anadolu University, Materials Science and Engineering, Turkey

In last decades, monticellite ($CaMgSiO_4$) bioactive ceramics have received much attention as bone graft substitutes due to their excellent biocompatibility, high bioactivity and superior mechanical and thermal properties compared to hydroxyapatite. Turkey has almost 72% of global boron reserves. Throughout production of 1 million tons of borax pentahydrate ($Na_2O2B_2O_35H_2O$), 900 thousand tons of solid boron derivative wastes are generated in Eti Mine Works Kirka Plant. The fairly high amount of waste causes costly storage problems and serious environmental issues. In present study, monticellite based bioactive ceramic powders were systematically synthesized at different temperatures below 1000°C, which are lower than that reported in the literature, for various dwell times gradually increased from 1 minute to 4 hours using boron derivative waste, obtained from Kirka Plant. Trace elemental analysis, qualitative phase analysis, microstructure analysis and particle size measurement of boron derivative waste and synthesized powders were investigated extensively. The detailed knowledge about phase transformations in boron derivative waste was gained during systematic study. The obtained results confirmed that monticellite based bioactive ceramic powders were successfully synthesized from boron derivative waste in cost-effective and eco-friendly way.

4:10 PM

(ICACC-S5-024-2018) In vitro Behavior of $CaSiO_3$-$CaCO_3$-SiO_2 Composites

B. Beyoglu*[1]; R. Riman[1]

1. Rutgers University, Material Science and Engineering, USA

Biocompatibility and osteoinductive potential of High Temperature Sintering (HTS) and/or Low Temperature Solidification (LTS) applied $CaSiO_3$ scaffolds were evaluated by in-vitro cell proliferation and differentiation tests, using two types of cells, mouse osteoblast progenitor cells and human Mesenchymal Stem Cells respectively. To evaluate dissolution behavior of these ceramics, LTS and/or HTS applied $CaSiO_3$ samples immersed into Simulated Body Fluid (SBF)

for time periods of 1, 7, 14, and 21 days. After SBF soaking, ionic concentration of Ca, Si and P ions and pH change of SBF and weight loss of the samples were monitored. TF-XRD and FESEM methods were used to investigate apatite formation ability of LTS and/or HTS applied $CaSiO_3$ samples. Ionic concentration of soluble factors in the SBF after infiltration of the $CaSiO_3$ samples indicated applying LTS process to sintered $CaSiO_3$ addressed the drawback of rapid dissolution of sintered $CaSiO_3$ by lowering release of ions from toxic levels. In-vitro cell experiments showed that $CaSiO_3$-$CaCO_3$-SiO_2 composites possess a promoted cell proliferation and significant greater osteogenic differentiation compared to only sintered $CaSiO_3$ and osteoinductive 45S5 bioglass. By applying LTS process on sintered $CaSiO_3$, the optimum concentration of soluble factors to activate cell proliferation and osteogenic differentiation reached with Si concentration of ~60ppm and Ca concentration of ~270ppm.

4:30 PM

(ICACC-S5-025-2018) The complex geometry of ceramics based on $Ca_{(3-x)}(K_yNa_{(1-y)})_{2x}(PO_4)_2$ for better permeability of bone grafts

P. Evdokimov*[1]; V. Putlayev[1]; N. Orlov[2]; A. Tikhonov[2]; E. Klimashina[1]; T. Safronova[1]; A. Garshev[1]; Y. Filippov[3]

1. Lomonosov Moscow State University, Chemistry Department, Russian Federation
2. Lomonosov Moscow State University, Materials Science Department, Russian Federation
3. Lomonosov Moscow State University, Institute for Mechanics, Russian Federation

The permeability of the bone graft determines the velocity of ingrowth of newly formed bone. The implant should have not only the high porosity but also have the predefined geometry of interconnected system of macropores (around 100m). Unmistakably, structures with high porosity lose their mechanical properties, so there should be a compromise for the best permeability and strength properties. In this study, we have tried some variants of structures with different porosities to find out the better combination of permeability and strength properties (stiffness, strength) for bone graft. SolidWorks 2013 software (Dassault Systèmes SolidWorks Corp.) with additional modules Simulation and FlowSimulation was used to carry out modelling. In this work, models with gyroid, diamond and Kelvin structure were tested. Calculations showed up that models with Kelvin structure have a better combination of properties than other structures with the same porosity. Such implants based on Ca3-x(KyNa1-y)2x(PO4)2 with Kelvin structure were fabricated via 3D-printing for in vivo tests to recover damaged bone tissue. The authors would like to thank the RSF (Grant no: 14-19-00752) for providing financial support to this project.

4:50 PM

(ICACC-S5-026-2018) Antibacterial and cytotoxic activities of novel tantalum-containing glass polyalkenoate cements

A. Alhalawani*[1]; M. Towler[1]

1. Ryerson University, Mechanical & Industrial Engineering, Canada

Objective: The effect of incremental tantalum (Ta) addition on the in-vitro antibacterial and cytotoxic properties of aluminium-free glass polyalkenoate cements (GPCs). Methods: A series of glasses based on the system $48SiO_2$-(36-X)ZnO-6CaO-8SrO-$2P_2O_5$-XTa_2O_5 with X varying from 0 mol% (TA0) to 0.5 mol% (TA2) were synthesized and characterized. Cements were prepared by individually mixing the glasses with poly(acrylic acid) (PAA, Mw, ~213,000) and deionized water. The cements were formulated in a powder:liquid ratio of 1:1. The antibacterial properties of the cement discs (10mm diameter, 2mm thick, n=3) were evaluated on agar plates after 1, 7 and 30 days, against one Gram-negative bacterium (E. coli) and two Gram-positive bacteria (Staph. aureus and Strep. epidermidis). Cytotoxicity of the cement discs (10mm in diameter, 2mm thick, n=3) was evaluated using primary bovine articular chondrocytes. Cell viability was assessed using a Methyl Tetrazolium (MTT) assay up to 7 days. Results: The formulated cements exhibited antibacterial activity against E. coli (9mm±0.4), Staph. aureus (8mm±0.4) and Strep. epidermidis (15mm±0.6). Cytotoxicity testing showed that Ta incorporation results in no toxicity effect. Conclusion: The incorporation of Ta did not show any adverse effect on the antibacterial and cytotoxic properties of GPCs, therefore have the potential in a range of hard tissue applications.

S7: 12th International Symposium on Functional Nanomaterials and Thin Films for Sustainable Energy Harvesting, Environmental, and Health Applications

Synthesis, Functionalization and Assembly of 1D, 2D and 3D Nanostructures III

Room: Coquina Salon C

Session Chair: Yogendra Mishra, Kiel University

1:30 PM

(ICACC-S7-048-2018) Photonic Crystal Enhanced Fluorescence and Its Applications (Invited)

M. Li*[1]

1. Institute of Chemistry, Chinese Academy of Sciences, Key Lab of Green Printing, China

Fluorescence techniques are widely used in many areas and disciplines due to their high sensitivity, ease of operation, and various readout modes, especially for fluorescence assays. Fluorescence emission is crucially depended on its surroundings besides the intrinsic property of fluorophore, known as Purcell effect. There has been immense interest in both instruments and methods to enhance fluorescence signal and achieve highly sensitive fluorescent assay. Photonic crystals (PCs), periodic dielectric or metallo-dielectric nanostructures, have a photonic band gap and can modify the spontaneous emission and light propagation. Thus, the PC provides a superior candidate for fluorescence enhancement. In our work, we pioneer in using the colloidal PCs to achieve an ultra-sensitive DNA detection, using colloidal PC patterns to achieve fluorescent ultratrace detection, high-throughput screen, versatile anti-counterfeiting patterns and so on. It is evident that the whole field is still a juvenile one and offers ample opportunity for optical device design and discovery. The results demonstrate the new possibilities of innovative devices based on PC enhanced fluorescence for a wide range of applications.

2:00 PM

(ICACC-S7-049-2018) Au@Cu_2O Core@Shell Nanocrystals as Peroxidase Mimics for Efficient E. coli Disinfection

M. Kuo*[1]

1. National Chiao Tung University, Taiwan

Redox couples of transition metals such as Cu^+ and Cu^{2+} are capable of interacting with hydrogen peroxide to produce hydroxyl radicals, which is known as Fenton-like reaction and can be used for environmental applications as well. For catalytic applications, Au nanocrystals as peroxidase enzyme mimics have demonstrated promising potential in environmental remediation. Concerning the photocatalytic applications, the combination of Cu_2O and Au presents effective charge separation under visible light illumination, a key factor of improving the photocatalytic activity. In this work, Au@Cu_2O core@shell nanocrystals were synthesized and used as peroxidase mimics for carrying out E. coli disinfection. By combining the peroxidase mimic features and pronounced charge separation, Au@Cu_2O nanocrystals can perform practical photocatalytic decomposition of E. coli under light illumination, but still show remarkable activity of E. coli disinfection after irradiation was switched off.

S9: Porous Ceramics: Novel Developments and Applications

Applications of Porous Ceramics

Room: Coquina Salon G
Session Chair: Alberto Ortona, SUPSI

1:30 PM

(ICACC-S9-026-2018) Production and Emerging Applications of Porous Aluminosilicates from Low-Temperature Geopolymer Processes (Invited)

D. Seo*[1]; S. Chen[1]

1. Arizona State University, School of Molecular Sciences, USA

Geopolymers provides a unique playground in exploring and producing new porous ceramic materials because of their innate dense gel network structure that consists of fused aluminosilicate nanoparticles (10 - 50 nm). We overview our recent efforts in harnessing the innate nanostructural nature of the material to produce new types of nanostructured alkali aluminosilicates. We show that a simple reactive emulsion templating with biorenewable oil can produce hierarchically porous geopolymer materials with coexisting controllable mesopores and spherical macropores, without a need of significantly modifying the conventional geopolymer synthetic process. Impregnation of the porous material with iron oxide has allowed us to produce to an effective arsenic sorbent for ground water purification. The same synthetic method has allowed us to synthesize highly-crystalline hierarchical zeolites with an exceptional CO_2 capacity, sorption kinetics, selectivity and regeneration capability and hence may realize cost-effective CO_2 separation to mitigate the global warming problem. Control of alkali activation and curing processes could lead to geopolymer nanoaggregates which may have a great potential in commercial applications such as polymer nanocomposites.

2:00 PM

(ICACC-S9-027-2018) Ag nanoparticle-deposited anodized titania nanotube arrays for electrodes of dye-sensitized solar cells

G. Kawamura*[1]; T. Wai Kian[1]; Z. Lockman[2]; H. Muto[1]; A. Matsuda[1]

1. Toyohashi University of Technology, Japan
2. Universiti Sains Malaysia, Malaysia

Surface plasmon resonance (SPR) of noble metal nanoparticles (NPs) has been attracting much attention because of its unique physical properties. Among various applications of SPR suggested so far, dye-sensitized solar cell (DSSC) is one of the most promising applications. Several efforts have been carried out to fabricate "plasmonic DSSCs", however the photo-current conversion efficiencies (PCEs) are not that high as expected. This is mainly due to uncontrolled structures of photoanodes doped with noble metal NPs. In this work, vertically aligned anodic titania nanotube arrays were prepared and used as photoanode to make electron flow easier. Then, several methods of Ag NP deposition on the photoanode were employed for controlling the dispersion state of Ag NPs. As a result, Ag NPs were found to be deposited only on the upper side of titania nanotube arrays when they were deposited through photo-reduction of Ag ions or electrophoretic deposition of Ag NPs with normal DC current. On the other hand, Ag NPs were homogeneously deposited throughout nanotubes when they were deposited by electrophoretically with DC accompanied with AC current. A DSSC with the homogeneously Ag-deposited titania nanotube arrays showed enhanced PCE of 5.01%, which is 1.37 times higher than PCE obtained from DSSC without Ag NPs.

2:20 PM

(ICACC-S9-028-2018) Novel Developments of Cellular Ceramics for Energy- and Environmental Applications (Invited)

U. F. Vogt*[1]; B. Fumey[2]; A. Bonk[3]; M. Gorbar[4]; G. Plesch[5]

1. Empa, Swiss Federal Laboratories for Materials Science and Technology, Materials for Energy Conversion, Switzerland
2. Empa, Swiss Federal Laboratories for Materials Science and Technology, Urban Energy Systems, Switzerland
3. Deutsches Zentrum für Luft- und Raumfahrt e.V, Institut für Technische Thermodynamik, Germany
4. ZHAW School of Engineering, IMPE, Switzerland
5. Comenius University, Department of Inorganic Chemistry, Slovakia

Reticulated ceramic foams composed of materials like Al_2O_3, ZrO_2, CeO_2, YbO_2, SiC with an open porosity of 75–90% and an open three-dimensional network structure are suitable for specific energy- and environmental based applications. The pore size of ceramic foams can vary from 10 to 100 ppi, corresponding to pore-diameters from 4500 to 500 μm. The presentation will give an overview on numerous applications for energy- and environmental related applications. For thermo-photovoltaics (TPV), Yb_2O_3 based foams are utilized to convert radiation into electricity by using silicon photocells. For cooking or heating with hydrogen, a novel catalytic diffusion burner based on highly porous Pt coated SiC ceramics has been developed. H_2 and CO (Syngas) has been synthesized from H_2O and CO_2 by a solar-thermochemical driven RedOx reaction, utilizing a dual porosity CeO_2 ceramics. The purification of water from organic pollutants by a photocatalytic process could be verified by Al_2O_3 foam substrates, coated with catalytic active anatase. Fluid dynamics of selected foams are investigated for waste-gas treatment of automotive catalysts. To increase the mechanical stability of foam ceramics, we have developed a method to impregnate hollow foams struts with ceramic slurry by a vacuum infiltration process, leading to a considerably increased strength. Specific results of the different applications will be presented.

2:50 PM

(ICACC-S9-029-2018) Environmental Remediation using Silicon Oxycarbide (SiCO) Porous Materials

S. I. Aguirre-Medel*[1]; P. Kroll[1]

1. University of Texas, Arlington, USA

We report synthesis of template-free porous silicon oxycarbide (SiCO) materials, their microstructural characterization and performance in environmental applications. In a first approach we combine a siloxane precursor containing Si-H bonds with cross-linkers bearing vinyl groups in highly diluted solutions (80-95 vol%). After gelation and solvent exchange using supercritical CO2 we obtain SiCO aerogels. A second approach uses standard sol-gel processing combined with long-term ambient condition drying to obtain a porous material. In both synthetic pathways, we anneal the porous materials in controlled atmospheres at high temperatures into SiCO ceramics. We characterize chemical composition together with specific surface area, pore size distribution, and total porosity of the materials. We further investigate the capacity of the materials to clean aqueous solutions containing organic dyes. The mesoporous SiCO ceramics absorb significant amounts of methylene blue. We use UV-Vis spectroscopy to monitor the disappearance of the characteristic absorbance peak of this dye.

Mechanical Properties of Porous Ceramics

Room: Coquina Salon G
Session Chair: Ulrich Vogt, Empa, Swiss Federal Laboratories for Materials Science and Technology

3:30 PM

(ICACC-S9-030-2018) Weibull Statistics for Strength Data of Porous Ceramics - ISO Proposal (Invited)

K. Yasuda*[1]; N. Okabe[2]; M. Takahashi[2]; S. Honda[3]; H. Kita[4]; S. Tanaka[5]; T. Akatsu[6]; S. Taruta[7]; H. Muto[8]; H. Miyazaki[9]; N. Shinohara[10]; S. Yamamoto[11]; T. Ono[12]; H. Ohnishi[13]; Y. Takahashi[14]; T. Mitsuoka[15]; M. Takanashi[16]; I. Kawashima[17]; A. Sugai[19]; M. Asayama[18]

1. Tokyo Institute of Technology, Japan
2. Ehime University, Japan
3. Nagoya Institute of Technology, Japan
4. Nagoya University, Japan
5. Nagaoka University of Technology, Japan
6. Saga University, Japan
7. Shinshu University, Japan
8. Toyohashi University of Technology, Japan
9. The National Institute of Advanced Industrial Science and Technology, Japan
10. AGC Asahi Glass, India
11. ASUZUC INC., Japan
12. Kyocera Corp., Japan
13. NIKKATO Corporation, Japan
14. Noritake Co., Limited, Japan
15. NGK Spark Plug Co., Ltd, Japan
16. IHI Corp., Japan
17. Kobe Steel, Ltd., Japan
18. Toshiba Corp., Japan
19. KUBOTA Corporation, Japan

This invited talk explains a basic idea in the ISO standard proposal of Weibull statistics for strength data of porous ceramics, and gives an opportunity to discuss it from every possible angle with the audience. Now, ceramics industry mainstream has been changed from materials themselves to device and component; dense ceramics to porous ceramics; structural applications to functional applications. However, strength reliability is still important for porous (functional) ceramics as well. So, Japanese industry and academia have collaborated for several years to investigate applicability of Weibull statistic to strength data of porous ceramics. After having round robin tests of wide variety of porous ceramics, it is revealed that most cases obey 2-parameter Weibull statistics, but not for some porous ceramics. To exclude such the case, screening by Weibull plot should be introduced in the ISO standard before calculating the parameters by maximum likelihood method. Valid condition to apply 2-parameter Weibull statistics is that the square of correlation factor of Weibull plot should be larger than 0.9. In the presentation, typical examples are shown for the discussion. This work was supported in part by METI, Japan.

4:00 PM

(ICACC-S9-031-2018) Bending Strength Test for Porous Ceramics with Various Pore Structure

S. Honda*[1]; H. Ohnishi[4]; T. Ono[3]; H. Kita[2]; M. Takahashi[5]; Y. Takahashi[6]; S. Tanaka[7]; S. Taruta[8]; T. Mitsuoka[9]; H. Muto[10]; K. Yasuda[11]; S. Yamamoto[12]; Y. Yoshizawa[13]

1. Nagoya Institute of Technology, Japan
2. Nagoya University, Japan
3. Kyocera Corporation, Japan
4. Nikkato Co. Ltd., Japan
5. Ehime University, Japan
6. Noritake Co., Limited, Japan
7. Nagaoka University of Technology, Japan
8. Shinshu University, Japan
9. NGK Spark Plug Co., Ltd, Japan
10. Toyohashi University of Technology, Japan
11. Tokyo Institute of Technology, Japan
12. ASUZUC INC., Japan
13. The National Institute of Advanced Industrial Science and Technology, Japan

Recently, porous ceramic materials are used as SOFC, support material of ceramic separated membrane system, etc. Development of porous material tended to be focused on the functional properties, such as the permeability and the reactivity at surface of pores. So, to the measurement and analysis method of fracture strength of porous ceramics are not paid the sufficient attention. In this talk, we report the bending test data obtained from the round robin tests in Japan, and discuss how to measure the strength distribution of porous ceramics. Samples are porous alumina, electrode material of SOFC and bio-material. Porosity and pore size were widely changed in 18-90 %, 1.3-350 micrometer. Bending test was carried by the three or four bending method using the rectangular bar shaped specimen. Specimen size effect of fracture strength was investigated using different size specimen cutting from same bulk. Every samples showed elastic deformation and followed by brittle fracture. Apparent specimen size effect for the fracture strength and that of distribution were observed in SOFC electrode. Most of the bending strength data could be expressed by 2-parameter Weibull distribution. This work was supported in part by METI, Japan.

4:20 PM

(ICACC-S9-032-2018) Compressive Strength and Permeability of Porous SiOC via Two-Stage Freeze Casting

N. Arai*[1]; K. Faber[1]

1. California Institute of Technology, Materials Science, USA

Porous ceramics have been of interest due to a broad range of application such as filtration, bioscaffolds and thermal insulators. However, it has been a challenge to maintain desired mechanical strength while maintaining sufficient functional properties such as permeability. Recent work on suspension freeze casting explored that bridges between lamellar pores, created by engulfment of large particles, can significantly improve the compressive strength of freeze-cast solids. An alternate approach is taken here. In this work, bridges between lamellar pores were created via two-stage freeze casting of a preceramic polymer. It has been shown that the bridge density can be controlled by changing the concentration of preceramic polymer in solution. Compressive strength and permeability were investigated to see the enhancement in strength and the influence on permeability by the presence of the bridges. Scanning electron microscopy of the sample after permeability testing is performed to demonstrate the robustness of the bridges.

4:40 PM

(ICACC-S9-033-2018) Multiwall Carbon Nanotube-Silicon Oxycarbide Composite via Freeze-Casting with Preceramic Polymers

C. Kuo*[1]; K. Faber[1]

1. California Institute of Technology, Material Science, USA

For porous ceramics, pore characteristics (such as pore size, pore shape and tortuosity) are closely related to their functionalities. Freeze casting is one of the techniques where a wide range of pore characteristics are easily tunable, which enables engineers to optimize pore characteristics for respective applications. However, despite the functionalities provided by pores, they often serve as crack initiators in materials, which is the main obstacle for a wider utilization of porous materials. In this study, an established method of strengthening ceramics—adding reinforcement fillers—was integrated into freezing casting to strengthen silicon oxycarbide (SiOC) ceramics. Multiwall carbon nanotubes (MWCNTs) were used as reinforcements. Freeze-cast MWCNT-SiOC composites were produced by dispersing MWCNTs in a polysiloxane polymer solution followed by freeze casting of MWCNT suspensions and pyrolysis. The compressive strength was nearly doubled by the incorporation of as little as 1 wt.% MWCNTs. The effects of the MWCNT on microstructure and fracture toughness of freeze-cast structures were also investigated.

S10: Virtual Materials (Computational) Design and Ceramic Genome

Modeling of Performances II

Room: Coquina Salon F

Session Chair: William Weber, University of Tennessee

1:30 PM

(ICACC-S10-016-2018) Image-based modeling – A series of useful tools for designing advanced ceramics (Invited)

G. L. Vignoles*[1]; G. Couegnat[1]; O. Caty[1]; V. Mazars[1]; M. Charron[1]

1. University Bordeaux, LCTS - Lab for ThermStructural Composites, France

This presentation will introduce some image-based modelling approaches that have been developed for the understanding and design of thermostructural materials such as fiber-reinforced ceramic matrix composites, carbon-carbon composites, and reinforced refractory open-cell foams. These tools are based on 3D images, either produced by tomography or by image synthesis. Combinations of original image processing methods and numerical simulation algorithms make it possible to study in detail the structure-property relationships in these classes of composite or porous ceramic materials.

2:00 PM

(ICACC-S10-017-2018) Metal-ceramic composites: Aspects of the numerical material and damage modeling (Invited)

R. Piat*[1]; M. Kashtalyan[2]

1. Darmstadt University of Applied Science, Germany
2. School of Engineering, University of Aberdeen, Centre for Micro- and Nanomechanics, United Kingdom

Metal-ceramic composites offer many advantages over monolithic metals and their alloys such as high specific stiffness and strength, better creep, fatigue and wear resistance, and good thermal properties. One of the recent advances in this area has been made possible thanks to innovative ceramic preforms, fabricated by freezing a water-alumina suspension and subsequent freeze-drying and sintering. As a result these metal/ceramic composites possess a hierarchical lamellar microstructure, with randomly orientated individual regions (domains), in which all ceramic and metallic lamellae are parallel to each other. Metal-ceramic composites with hierarchical lamellar microstructure exhibit pronounced elastic and plastic anisotropy at the domain level. In this paper, the thermo-mechanical properties of these composites were modeled based on the 3D tomographic images of the microstructure. The stress fields in a single-domain sample of metal-ceramic composite containing multiple cracks in the ceramic layer are also investigated. The cracked microstructure for different stage of damage is modeled by analytical and computational approaches. The result obtained by finite elements analysis is consistent with experimental results.

2:30 PM

(ICACC-S10-018-2018) Electronic Origin of Slip Deformation Ability in Ductile Ionic Crystals (Invited)

K. Matsunaga*[1]

1. Nagoya University, Materials Physics, Japan

First-principles calculations of slip deformation in some ionic crystals were performed to reveal physical origins of their ductility at the electronic and atomic level. As an example, slip deformation behaviors in sodium and silver chloride crystals with the rock salt structure were analyzed. A primary slip system of sodium chloride at low temperatures is limited to {110}<110>, whereas silver chloride has several slip systems of {110}<110>, {111}<110> and {100}<110> to be activated, which cannot be explained by the simple ionic nature of bonding. From electronic densities of states during slip deformation, it was also found that slip deformation in silver chloride accompanies unexpected formation of covalent like Ag-Ag interactions across the specific slip planes, which makes multiple slip systems available even at low temperatures. Calculations of the dislocation cores also showed that silver chloride has a much smaller core energy than sodium chloride. This may be closely related to better stability of dislocations in silver chloride, leading to larger plastic deformation.

Modeling of Performances III

Room: Coquina Salon F

Session Chair: Gerard Vignoles, University Bordeaux

3:20 PM

(ICACC-S10-019-2018) Effects of the electronic and nuclear energy loss in molecular dynamics simulations of irradiation (Invited)

E. Zarkadoula*[1]; Y. Zhang[1]; W. J. Weber[2]

1. Oak Ridge National Lab, Materials Science and Technology, USA
2. University of Tennessee, Materials Science and Engineering, USA

During irradiation, energy is transferred from the moving ion to the nuclei of the target material (elastic energy loss) and to the electrons (inelastic energy loss). Understanding the materials' response to radiation includes their response to both the nuclear and electronic energy loss, separately but also combined. Using molecular dynamics simulations, the role of the electronic energy loss in the damage creation and microstructure alterations is investigated. The role of the pre-existing disorder in the energy dissipation in the system is examined and we provide insights into the mechanism of the coupling of electronic and atomic processes. We discuss the synergistic, additive and competing effects of the coupled action of the elastic and inelastic energy loss. Our work highlights the combined effects of the nuclear and electronic energy loss in the energy dissipation and damage production.

3:50 PM

(ICACC-S10-020-2018) Ab initio Molecular Dynamics Simulations of Irradiation Response in Ceramics (Invited)

W. J. Weber*[1]; B. Liu[2]; J. Xi[1]; J. Wang[3]

1. University of Tennessee, Materials Science & Engineering, USA
2. Shanghai University, China
3. Shenyang National Laboratory for Materials Research, China

Ab initio molecular dynamics (AIMD) methods have been used to investigate low-energy atomic recoil events in 3C-SiC, Ti_3SiC_2 and AlN, which are key materials for refractory coatings and composites proposed for nuclear applications and extreme environments. In 3C–SiC, the minimum displacement energies for C and Si atoms are found along the [1 0 0] direction, with values of 20 and 49 eV, respectively. The results demonstrate that significant charge transfer occurs during the dynamics process, and defects can enhance charge transfer to surrounding atoms, which provides important insights into the formation of charged defects. It is found that the C vacancy is a positively charged defect, whereas the Si vacancy is in its neutral state. In Ti_3SiC_2, the threshold displacement energies are shown to be strongly dependent on recoil direction and layer of origin. For Ti and Si atoms in the Ti-Si layer, which exhibit weak mixed bonding, the threshold displacement energies for recoils perpendicular to the basal planes are larger than those parallel to the basal planes, which is obviously related to the layered-structure. In contrast, the threshold displacements energies for the strong covalently-bonded Ti and C atoms in the Ti-C layer are less dependent on recoil direction. In AlN, the displacement energies for Al and N are more than 100 eV along the [0001] direction. Work supported by the U.S. DOE, BES, MSED.

4:20 PM

(ICACC-S10-021-2018) Multi-Phase-Field Modeling of Crack Propagation in Polycrystalline ZrB_2-Based Ceramics

A. Emdadi[1]; M. A. Zaeem[1]; W. Fahrenholtz*[1]; G. Hilmas[1]

1. Missouri University of Science & Technology, Materials Science and Engineering, USA

A multi-phase-field model (MPFM) is presented to simulate intergranular and transgranular crack propagation in polycrystalline ceramics. The MPFM includes elastic anisotropy and grain boundary (GB) energy anisotropy by considering grain orientations and misorientations at GBs, respectively. The model is applied to study crack propagation in ZrB_2-carbon based polycrystalline, and intergranular and transgranular crack propagation are investigated in coarse and fine grain microstructures. The results show that transgranular crack propagation is more favorable in fine grain microstructures, which agrees with experimental and theoretical results. The effect of grain boundary strength on the fracture behavior is also discussed. The results of this study can be used in designing engineered architectures for ZrB_2-based composite ceramics to increase crack deflection and fracture toughness.

4:40 PM

(ICACC-S10-022-2018) Structure and Properties of Pyrophosphate Crystals

P. Adhikari*[1]; W. Ching[1]

1. University of Missouri, Kansas City, Department of Physics and Astronomy, USA

The electronic structure and interatomic bonding in different multi-component pyrophosphates $K_2Mg(H_2P_2O_7)_2 \bullet 2H_2O$, $(NH_4)_2Zn(H_2P_2O_7)_2 \bullet 2H_2O$, $K_8Cu_4(H_2P_2O_7)_8 \bullet 8H_2O$, and $K_2Cu(H_2P_2O_7)_2 \bullet 2H_2O$ are investigated for the first time showing complex interplay of different types of bonding. The existing structures from single-crystal X-ray diffraction are fully optimized before properties calculation. Detailed bond analysis between every pairs of atoms reveals the complexity of various covalent, ionic and hydrogen bonding and their sensitive dependence on structural differences. Quantitative analysis of internal crystal cohesion in terms of total bond order density (TBOD) and partial bond order density (PBOD) gives the relative importance of different types of bonding. The optical and mechanical properties of these crystals are also calculated. These results provide new insights that acidic pyrophosphates could have a variety of unrealized applications in advanced technology.

5:00 PM

(ICACC-S10-023-2018) All Atom Molecular Dynamics Simulation Study; Effects of Functional Group on Polymer Brush Friction

S. Uehara*[1]

1. IMR Tohoku University, Japan

In recent years, concentrated polymer brush(CPB) grafted on ceramics with high density has been developed. CPB swelling in good solvent shows high wear resistance and lubricity. Therefore, CPB has attracted much attention as a lubricant for ceramics. However, the solvent used for CPB such as water and organic liquid evaporates due to the heat of friction, leading to loss of the lubricity. In addition, the use of solvent limits the application of CPB to systems where the use of liquids is prohibited e.g. MEMS. Recently, it was shown that the fluorinated CPB, which contains CF_3 moiety in each monomer, showed low friction in dry state. It is proposed that fluorinated CPB having crystallinity acts as a solid lubricant. However, the details were unknown. Thus, we performed all atom molecular dynamics simulation of friction to reveal the effect of CF_3 moiety. The fluorinated poly(2,2,2trifluoroethylmethacrylate) brush and nonfluorinated poly(ethylmethacrylate) brush were compared. The fluorinated brush showed lower friction force than that of nonfluorinated brush. We found that interpenetration of the fluorinated polymer brush is lower than that of the nonfluorinated brush. Thus, the presence of the CF_3 moiety reduces entanglement of the brushes, which leads to low friction of ceramics. Influence of crystallization of fluorination will be discussed.

5:20 PM

(ICACC-S10-024-2018) Phase Field Simulations of Elastically Anisotropic Heterogeneous Polycrystals

J. Allen*[1]

1. ERDC, USA

In this study, we developed a multi-order, phase field model to compute the stress distributions in anisotropically elastic, inhomogeneous polycrystals and study stress driven grain boundary migration. In particular, we included elastic contributions to the total free energy density, and solved the multicomponent, non-conserved Allen-Cahn equations via the semi-implicit Fourier spectral method. Our analysis included specific cases related to bicrystalline planar and curved systems as well as polycrystalline systems with grain orientation and applied strain conditions. The evolution of the grain boundary confirmed the strong dependencies between grain orientation and applied strain conditions and the localized stresses were found to be maximum within grain boundary triple junctions.

S14: Crystalline Materials for Electrical, Optical and Medical Applications

New Direction

Room: Tomoka C
Session Chairs: Martin Magnuson, IFM; Mariola Ramirez, Universidad Autonoma de Madrid

1:30 PM

(ICACC-S14-042-2018) Advanced oxide thin films prepared by ultraviolet laser-assisted chemical solution processing for electrical and optical applications (Invited)

T. Tsuchiya*[1]; Y. Uzawa[1]; T. Nakajima[1]; I. Yamaguchi[1]

1. National Institute of Advanced Industrial Science and Technology (AIST), Japan

In order to construct the low carbon society in the world, it is necessary to develop a high performances new green device. Metal oxides are expected to be key materials for a new device by controlling metal composition, a crystal structure, orientation or multilayer of the film, a carrier, a spin, etc. In order to fabricate the new devices, their parameters should be controled. In adition, the low cost and power saving process is nesesary for constructing sustainable society in the world. For these purpose, chemical solution process (CSP) would be suitable because it does not require vacuum and high facility investments. In addtion, precise metal composition control is possible when the materials are made from the more than 3 or 4 metal components. However, a heat treatment at greater than 300oC is required for the metal oxide thin film growth, it is diffcult to preare the oxide film on organic materials. To overcome these problems, we have developed the photo-induced chemical solution process such as excimer laser-assisted metal organic deposition (ELAMOD) and photo reaction of nano-particle (PRNP), and photo reaction of hybrid solution(PRHS) at low temprature. In this presentation, we demonstrate 1.epitaxial VO2 film for the IR sensor with high TCR, 2.Flexible ITO film by PRNP method, 3.Flexible luminescent or resistor films by PRHS method.

2:00 PM

(ICACC-S14-043-2018) A Self-Compensating Strain Gage for Use at Elevated Temperatures

M. Ricci*[1]; K. Rivera[1]; C. Dudinski[1]; J. Weigler[1]; O. Gregory[1]

1. University of Rhode Island, Dept. of Chemical Engineering, USA

A self-compensating, nano-composite strain gage has been developed for static strain measurement at temperatures up to 600C. These nano-composite strain gages are comprised of refractory metal (Ni, W, NiCr) nanoparticles embedded in an indium tin oxide (ITO) matrix. The relative amounts were optimized to yield the desired temperature coefficient of resistance and piezoresistive response. These thin film strain gages were sputtered from composite targets prepared using conventional ceramic powder processing techniques and deposited using RF sputtering. After deposition, the films were annealed in an inert atmosphere to remove trapped argon and eliminate point defects. Temperature coefficient of resistance (TCR) and drift measurements were made by heating the devices to temperature and monitoring resistance changes using a four-point probe method. Gage factor (GF) was measured from room temperature to 600C. The most promising nanocomposite films in terms of gage factor and TCR were analyzed using a combination of SEM/EDS/XRD.

2:20 PM

(ICACC-S14-044-2018) Advanced Sensors for CMC Gas Turbine Engine Components

K. Rivera*[1]; O. Gregory[1]; M. Ricci[1]

1. University of Rhode Island, Chemical Engineering, USA

Next generation gas turbine engines used for propulsion purposes will utilize ceramic matrix composite (CMC) components in the engine hot section to achieve higher engine operating temperatures and higher efficiencies. Within the engine hot section, rotating components can experience temperatures above 1000°C and g-forces over 50,000g. Therefore, it is important to monitor temperature, strain, and heat-flux experienced by these CMC components under these harsh conditions, such that structural models can be verified and new designs can be evaluated. However, conventional wire instrumentation is not well suited for such applications since the sensors and leads cannot be welded to the CMC components and the CMC surfaces cannot be altered. Strain, temperature and heat-flux sensors have been developed for SiC-SiC CMC's, which rely on the semiconducting properties of the SiC substrate itself to generate large and reliable piezoresistive and thermoelectric responses, with the added advantage of minimal intrusion, negligible mass, and fast response times. At temperatures >1000°C, oxidation, small diffusional distances, and microstructural changes can lead to signal drift and thus coatings were developed to ensure the integrity of the semiconductor:metal contacts in these sensors. Results of Temperature, strain and heat-flux testing will be presented as well as the fabrication steps used to make the sensors.

2:40 PM

(ICACC-S14-045-2018) Temperature dependent thermal properties of reaction bonded silicon carbide (RBSC) composites

Y. Zhang*[1]; C. Hsu[1]; P. Karandikar[2]; S. Aubuchon[3]; C. Ni[1]

1. University of Delaware, Material Science and Engineering, USA
2. M Cubed Technology, Inc., USA
3. TA Instruments, USA

SiC based composites are utilized in thermal and energy related fields because of their excellent thermal properties and high environmental stability. Reaction bonded silicon carbide (RBSC) composite is favored by industry due to its cost-effectiveness and outstanding properties afforded by the advanced fabrication technology. In this paper, temperature dependent thermal properties of the RBSC with dispersion of 60 wt%, 80 wt%, and 90 wt% SiC were investigated. Thermal conductivity of 90 wt% SiC RBSC reaches 211.4 W/m•K at room temperature, and 55.77 W/m•K at 1173 K. Thermal behavior of RBSC with the temperature profile suggests a structural effect occurred in the composite system. The RBSC microstructure was then thoroughly characterized and showed the existence of amorphous Si near the SiC/Si interface area. Confirmed by TEM in-situ heating test, amorphous Si phase transformation was observed and believed to be responsible for the thermal behavior of RBSC system at elevated temperature. We proposed that the amorphous phase evolution improves the thermal properties of RBSC system. Further investigation also confirmed that thermal conductivity of annealed bulk RBSC, compared with as-prepared specimen, was enhanced by 7 % at room temperature.

3:20 PM

(ICACC-S14-046-2018) Electronic Properties and Bonding in Zirconium Hydride Thin Films Investigated by X-ray Spectroscopy (Invited)

M. Magnuson*[1]

1. Thin Film Physics Division, Linköping University, Department of Physics, Chemistry and Biology, (IFM), Sweden

The variation in local atomic structure and chemical bonding of ZrH_x magnetron sputtered thin films are investigated by X-ray spectroscopies and ab initio electronic structure calculations. For low

hydrogen content, the films consist of a superposition of hexagonal close packed metal (a-phase) and understoichiometric δ-ZrH_x (CaF_2-type structure) phases. For larger hydrogen contents, the films form single phase ZrH_x that largely resembles that of stoichiometric δ-ZrH_2 phase. With increasing hydrogen content, we observe significant reduction of the 4d valence states close to the Fermi level as a result of redistribution of intensity towards the H 1s – Zr 4d hybridization region at ~6 eV below the Fermi level. Chemical shifts at the absorption edges towards higher energy with increasing hydrogen content are observed due to charge-transfer and an ionic or polar covalent bonding component between the Zr 4d and the H 1s states. The change in the electronic structure, spectral line shapes, and chemical shifts as function of hydrogen content and vacancies is discussed in relation to the corresponding modifications of bond lengths, Jahn-Teller distorsions, and charge-transfer from Zr to H that affects the properties by electronic redistribution in the valence band.

3:50 PM

(ICACC-S14-047-2018) Role of interparticle space in hollow silica-alumina composite spheres on their acidic properties and activity for hydrolysis of ammonia borane (Invited)

T. Umegaki*[1]; N. Toyama[1]; R. Ogawa[1]; S. Ohki[2]; M. Tansho[2]; T. Shimizu[2]; Y. Kojima[1]

1. College of Science & Technology, Nihon University, Department of Materials and Applied Chemistry, Japan
2. National Institute for Materials Science (NIMS), Japan

Porous materials with micropores and mesopores have attracted much attention in various fields. The kind of materials showed intrinsic properties compared with other types of materials such as fine particles. This work discussed about role of interparticle space in hollow silica-alumina composite spheres on their acidic properties and activity for hydrolysis of ammonia borane (NH_3BH_3) based on our recent research. The hollow spheres showed much higher activity for hydrogen evolution from aqueous NH_3BH_3 solution than the fine particles. The hollow spheres possessed much higher amount of active Brønsted acidic sites strongly adsorbed with ammonia molecules than the fine particles probably because the acidic sites accumulated in interparticle space formed by the primary particles of the hollow spheres shows unexpected strong Brønsted acid properties. Structure of the interparticle space can be controlled by adding L(+)-arginine and/or cethyltrimethylammonium bromide for the preparation. The hollow spheres prepared using these additives consisted of the shell with well-ordered pore structure including well-dispersed primary particles, and showed high hydrogen evolution rate from aqueous NH_3BH_3 solution than that prepared without these additives probably because the reactants diffused at high rate through well-ordered interparticle space.

S16: Geopolymers, Inorganic Polymers and Sustainable Materials

Sustainable Materials and Composites

Room: Ponce de Leon

Session Chair: Ruy Sa Ribeiro, INPA-National Institute for Amazonian Research

1:30 PM

(ICACC-S16-022-2018) On the Mechanical Behavior of Natural Fibers Reinforced Geopolymers (Invited)

A. C. Trindade[1]; F. d. Silva*[1]

1. Pontificia Universidade Católica do Rio de Janeiro (PUC-Rio), Civil Engineering, Brazil

The increasing use of cementitious materials has created a demand for alternative systems. Portland cement production generates 5% of global CO_2 emissions per year. In contrast, the manufacturing of Al-Si precursors can reduce by up to six times this amount. Geopolymers present a compatible mechanical and durable response, despite still exhibiting a brittle behavior. An interesting sustainable solution is the incorporation of natural fibers. This research presents an experimental investigation regarding the mechanical behavior of natural fibers reinforced geopolymers. Mixtures were produced through metakaolin activation by a sodium silicate and hydroxide solution. Microstructural analyzes were performed, such as: X-ray fluorescence, X-ray diffraction, and SEM observation. Different natural fibers were incorporated in uni and bi-directional forms. The composites were subjected initially to compression, tensile and flexural monotonic loading. Cyclic flexural loading tests were also performed under a deformation-controlled regime. Fiber treatment was carried out, using a polymer solution, to verify mechanical changes due to adhesion. Pull-out tests were performed to comprehend the fibers-matrix stress transfer mechanisms. The results showed that fiber-treated composites presented higher ultimate stresses and greater deformation capacities, and the curauá reinforced geopolymers obtained the best results.

2:00 PM

(ICACC-S16-023-2018) Functional Glass-ceramic Foams from 'Inorganic Gel Casting' and Sintering of Glass/Slag Mixtures (Invited)

A. Rincon*[1]; D. Desideri[1]; E. Bernardo[1]

1. University of Padova, Dipartimento di Ingegneria Industriale, Italy

Glass foams have been recently manufactured according to a new method, based on alkali activation. Soda-lime glass suspensions, in alkaline aqueous solution, undergo progressive gelification at low temperature (80 °C), owing to the formation of hydrated calcium silicate compounds (CSH). Before complete hardening, an extensive foaming may be achieved by vigorous mechanical stirring, with the help of a surfactant. Unlike in conventional glass foams, a sintering treatment, at only 700-800 °C, is applied after the foaming, to stabilize the cellular structures. The present investigation is aimed at showing the flexibility of the approach, by application to more complex suspensions, comprising also an industrial waste (from 10 to 30 wt% of the solid content), consisting of an iron-rich slag from copper metallurgy. After foaming, glass/slag mixtures could be sintered at 800-1000 °C; the mutual interaction caused an extensive crystallization, with precipitation of Ca-Fe silicate and iron oxides (hematite and magnetite), promoting the mechanical properties (up to 2.3 MPa, with a porosity of about 80%). Leaching test confirmed the stabilization of pollutants, from the slag, in the final ceramics. Owing to the separation of iron oxides, particularly magnetite, the newly obtained foams exhibited a ferrimagnetic behavior, that could be exploited in electromagnetic shielding applications.

2:30 PM

(ICACC-S16-024-2018) The effect of partial metakaolin replacement by industrial waste products on geopolymer cements intended for the creation of structural insulated panels (SIPs) (Invited)

L. Oakes*[1]; B. Magee[1]; A. Wilkinson[1]

1. Ulster University, Built environment, United Kingdom

This project used industrial waste products Fly ash (FA), GGBS, Silica Fume (SF) and iron silicate for partial and total replacement of the expensive, mined and fired Metakaolin (MK) in MK geopolymers with varied alkaline reagents to be used in the creation of fibre composite SIPs for fireproof building construction and renovation. The silica / alumina ratio, 7 and 28-day compressive strength and workability of each will be analysed and reported with the creation of contoured ternary blend diagrams enabling performance based specification to help boost use in construction. Binary mixes with suitable flow (>180mm) and compressive strength (>35N/mm^2) for structural use included the 100% MK mix, all GGBS mixes,

the FA mixes at <20%, the SF at 10% and the iron silicate at <40%. The highest performing mix was the (20% GGBS, 80% MK) mix which produced 69.5N/mm^2 compressive strength and a flow >255. 8 new suitable mixes where derived from ternary blends with the binary section of the charts providing another 3 suitable mainly slag blends. Many designs allowed for the use of 100% recycled material without loss of strength and generally with increased flow. The geopolymer cements studied were perfect for use in SIPs providing improved mechanical and fire performance and reduced CO_2 emissions compared to current alternatives.

3:20 PM

(ICACC-S16-025-2018) Development of alkali activated cement panels using different industrial byproducts as raw materials (Invited)

L. Lima Junior*[1]; S. Pianaro[1]

1. State University of Ponta Grossa, Materials Engineering Department, Brazil

The present work aims the obtainment of alkali activated cement panels used as facing materials for building applications. The main part of the research was based on the study of different industrial byproducts, such as glass, foundry and ceramics industry, and their roles as part of alternative cementitious systems able to achieve properties similar to the ones observed for conventional cementitious products originated from ordinary portland cement. The most significant results were obtained when combining the use of waste glass and ceramic tiles, along with a solution of 3.6 mol/l of potassium hydroxide as precursors. The desired properties were listed according to the specifications of the commercial options of similar products found within the market. The research led to the optimization of a cementing system which was used as starting point for the obtainment of composites with the addition of different admixtures. The composite specimen were tested for their flexural strength, water absorption and aging effects. The best results obtained in laboratory scale were chosen to be tested within pilot production lines on real size specimen, delivering a more trustful comparison in between these novel products and commercially available options. The new products showed a lower value of water absorption while maintaining the same values of mechanical strength.

3:40 PM

(ICACC-S16-026-2018) Bone ash, Glass Frit and Saffil Reinforced Geopolymer using MetaMax, Mymensingh Clay and Synthetic Mymensingh Clay

A. W. Bhuiya[1]; M. Hu[1]; D. Ribero[1]; W. M. Kriven*[1]

1. University of Illinois at Urbana-Champaign, USA

Natural bone ash (calcined) of dicalcium phosphate, glass frit, Saffil® alumina fibers and silica sand have been investigated as reinforcements for the enhancement of microstructural integrity and mechanical properties in potassium-based geopolymer (KGP). Reinforcements were individually mixed to form KGP using MetaMax (MT), Mymensingh clay metakaolin (MW) and synthetic Mymensingh clay metakaolin (MW-SYN). Dicalcium phosphate (DCP) and glass frit (GF) at elevated temperature reacted with potassium geopolymer developing an interface with reduced porosity and structural integrity. The geopolymer composites were thermally treated between 1125°C and 1200 °C to investigate the microstructure, thermal resistance and flexure strength. Higher flexure strength was observed 17.12 MPa in KGP(MW), 14.67 MPa in KGP(MW)-7.5DCP-7.5GF, 29.5MPa in KGP(MT)-10SF-7.5DCP compared with 7.64 MPa in KGP(MT), 5.43 MPa in KGP(MT)-7.5DCP-7.5 GF, 4.8 MPa in KGP(MT)-7.5DCP-30SS. Self-glazed geopolymer ceramics was developed in KGP(MW), KGP(MW)-7.5DCP-7.5GF and KGP (MT)-10GF after thermal exposure between 1100 ^{0}C and 1150 ^{0}C. Water absorption studies of KGP(MW)-7.5DCP-7.5GF and KGP (MT)-7.5GF fired between 1100 ^{0}C-1150 ^{0}C exhibited absorption within 1% (dry basis), while KGP(MT)-7.5DCP-7.5GF showed nearly 8% (dry basis).

4:00 PM

(ICACC-S16-027-2018) Low temperature geopolymeric setting for the stabilization of earthen masonry units

E. B. Ojo*[1]; A. I. Katagum[2]; R. S. Teixeira[3]; D. S. Matawal[2]; H. Savastano[3]

1. African University of Science and Technology, Materials Science & Engineering, Nigeria
2. Nigerian Building and Road Research Institute, Nigeria
3. University of Sao Paulo, Brazil

The objective of this study was to evaluate the feasibility of Low Temperature Geopolymeric Setting (LTGS) for the stabilization of argilaceous minerals present in soils for the production of earthen building units. To evaluate the synthesis-composition-property relationship, selected soils from Nigeria and Brazil were stabilized at varying alkaline conditions and curing conditions. Results from the investigation revealed room temperature curing for 3days at 3% alkaline content to be sufficient to produce water stable units. Compressive strengths improved from 4.7MPa to 9MPa by increasing duration of room temperature from 3 days to 45 days demonstrating time-dependence of the geopolymeric reactions. Furthermore, curing at elevated temperatures (60°C and 100°C) for five hours resulted in improved compressive strengths of 6MPa and 13 MPa respectively. Four point bend tests yielded average Modulus of Rupture (MOR) of oven-dry/saturated samples of about 3.0/0.5 MPa at a combined curing of 5hours at 100°C and 20 days at room temperature. Based on the mineral composition and processing conditions, water absorption after immersion in water for 24 hours ranged between 7-20%. These results suggest that with appropriate processing conditions, the LTGS technique may be used for the development of improved building units using natural clays/lateritic soils as precursors.

S17: Advanced Ceramic Materials and Processing for Photonics and Energy

Multifunctional III

Room: Halifax A/B

Session Chairs: Justin Caram, University of California, Los Angeles; J Zapien, The Pennsylvania State University

1:30 PM

(ICACC-S17-041-2018) Promising Nanostructured Cu_2Se Thermoelectrics via High Throughput and Rapid Chemical Synthesis (Invited)

M. S. Toprak*[1]; M. Saleemi[1]; M. Tafti[1]; S. Ballikaya[2]

1. KTH Royal Institute of Technology, Dept. of Applied Physics, Sweden
2. Istanbul University, Department of Physics, Turkey

Bulk nanostructured Cu_2Se, consisting of earth-abundant elements, with high thermoelectric (TE) efficiency have been fabricated via a facile and high yield synthesis route. Starting from readily available materials and by means of rapid and energy-efficient microwave-assisted thermolysis, nanopowders of Cu_2Se were synthesized. SEM revealed the presence of secondary globular nanostructures in the order of 200nm consisting of < 50nm primary particles. HR-TEM revealed crystalline nature of particles with truncated morphology. Pellets with preserved nanostructured grains were obtained through a detailed investigation of different parameters in the compaction such as applied load, heating rate, cooling profiles. Applied load during cooling was demonstrated to have a big impact on the final TE efficiency of the pellets. A very high TE figure of merit (ZT) > 2 was obtained at 900K for SPS-compacted Cu_2Se nanopowders in the absence of the applied load during controlled cooling. The obtained ZT exceeds the state of the art by about 25% improvement at 900K, attributed both to the low thermal conductivity, as low as 0.38 W/mK, and the enhancement in the power factor in nanostructured Cu_2Se. The proposed synthesis as well as the consolidation scheme

could lead to reliable production of large scale TE nanopowders and TE elements/legs for niche device applications.

2:00 PM

(ICACC-S17-042-2018) Emerging Applications of Metal Organic Framework Composites (Invited)

R. Riccò*[1]

1. Graz University of Technology, Austria

Metal-Organic Frameworks (MOFs) are crystalline tridimensional networks of metal nodes and organic ligands, investigated for gas storage, separation, sensing, catalysis, drug delivery, and optoelectronics. This non-exhaustive list arises from their tunable porosity, versatile chemical functionalization, and high accessible surface area. Extensive research is devoted to the preparation of MOF composites encapsulating ceramic, metallic, or polymeric nanoparticles, to obtain unique porous materials with unprecedented magnetic, luminescent, and catalytic properties. The synergy between MOFs and magnetic nanostructures originates composite materials responding to external magnetic stimuli. This promising strategy allows for dynamic positioning of porous crystals, an effective method when precise confinement is needed, especially for miniaturized devices with difficult accessibility (e.g. microfluidics). Another important aspect is the remote induction heating, occurring with superparamagnetic particles exposed to an alternating magnetic, useful to trigger the release of species from MOF cavities or channels, as already demonstrated for drugs and gases. In this communication, the most convenient synthetic routes, salient functional properties, and promising applications of these MOF composites will be outlined, with a focus on our recent progresses in the sequestration of heavy metals from water using magnetic MOF composites.

2:30 PM

(ICACC-S17-043-2018) Advances Ceramics for Aerospace Applications: Current Issues and Future Trends (Invited)

V. M. Castano*[1]

1. Universidad Nacional Autonoma de Mexico, Mexico

The main challenges that ceramics science and technology face in the aerospace industry will be reviewed, with emphasis on the available Roadmaps that both NASA and the Eurpean Space Agency have elaborated. In particular, mechanical strength, radiation resistance and poly-functionality will be analyzed, under the light of the current technologies and their chances to overcome those serious limitations if a new generation of aerospace ceramics and other materials is to be produced. Then, 3 or 4 potential venues, including nanodiamonds and other carbon nanostructures, will be discussed, along with the needs in R&D for producing cost-effective materials.

3:20 PM

(ICACC-S17-044-2018) Electrolyte-gating meets light harvesting: the case of phototransistors working in organic and aqueous media (Invited)

C. Santato*[1]

1. Ecole Polytechnque de Montreal, Canada

Electrolyte gating exploits electrical double layers forming at semiconductor/electrolyte interfaces to modulate, at low voltages (ca 1 V), the electrical conductance of semiconductors. This type of gating can be combined with photogating, where charge carriers are photoproduced upon illumination of the semiconductor with light of suitable energy. We will discuss the proof of principle of metal oxide phototransistors working in quasi-solid state as well as aqueous media. Particular attention will be given to photocatalytic transistors where, once fixed the electrical bias and light conditions, the transistor current is controlled by the concentration of the redox active pollutants present in the aqueous electrolyte, with the semiconductor exposed to sunlight. The miniaturization of the photocatalytic transistors is expected to promote its integration to smart phones thus creating a mobile sensor platform to measure water quality.

3:50 PM

(ICACC-S17-045-2018) Soft Processing (= Green Processing) for 2D Inks: Direct Fabrication of Inks of Graphenes, Their Hybrids, MXene,MoS_2 under Ambient Conditions (Invited)

M. Yoshimura*[1]

1. National Cheng Kung Univ., PCGMR/ME&E, Taiwan

The Fabrication of 2D materials Ink should be free from using excess energies for firing, sintering, melting, vaporizing and/or expensive equipments. Our proposed Soft processing has advantages : (a) simple reaction set up,(b) at ambient conditions, (c) simple procedure (d) direct formation of Ink (dispersed in liquid) and (e) less operating costs and wastes. Present study utilizes "Submerged Liquid Plasma [SLP]", "Electrocemical Exfoliation [ECE], and Sono-chemical reaction[SCR]. SLP resulted Nitrogen functionalized Graphene Nano-sheets of <5 layers from Graphene suspension and/or Graphite electrode in acetonitrile. Products contained unsaturated or high energy functional group (e.g. C = C, C = N and C ≡ N), thus they are electrochemically active. Using pencil rods instead of Graphite rods prodused Nano-clay/Graphene hybrids.. Au Hybrids also realized by SLP. In the ECE, graphite anode is exfoliated electrochemically by H_2O_2-NaOH or Glycine-H_2SO_4 aqueous solutions under ambient conditions, for 5-30 min with +1-+5 volt, into 3-6 layers Graphene Nanosheets, which suspended in solutions can be transformed into N-FG in the same container using $BrCH_2CN$/dioxane, further into Au-Hybridized N-FG by sonification.they showed excellent catalytic performance. Other 2D materials like MoS_2 can also be dispersed in a Solution via SCR for SERS.

4:20 PM

(ICACC-S17-046-2018) In situ electron beam driven reactions as a nanotechnology tool (Invited)

M. Rummeli*[1]

1. Soochow University, School of Energy/SIEMIS, China

A key feature of my research is to develop a transmission electron microscope (TEM) into an atomic-scale nano-chemistry laboratory to fabricate, modify and characterize samples so that crucial structure property studies and synthesis studies can be conducted in high spatial resolution and high temporal resolution. In this presentation I will show a variety of electron beam driven reactions and electron beam engineering techniques in which we can fabricate and manipulate nano-materials. I will also show how one can structure and look at structure-property relationship of graphene devices, which, can be both fabricated and examined in-situ in a TEM.

Honorary Sympoisum: Advancing Frontiers of Ceramics for Sustainable Society Development - International Symposium in Honor of Dr. Mrityunjay Singh

Advancing Frontiers of Ceramics VII -Materials Integration

Room: Coquina Salon E

Session Chairs: Jingyang Wang, Shenyang National Laboratory for Materials Science, Institute of Metal Research, Chinese Academy of Sciences; Monica Ferraris, Politecnico di Torino

1:30 PM

(ICACC-HON-052-2018) Polymer-derived ceramic coatings for metallic substrates (Invited)

W. Krenkel*[1]

1. University of Bayreuth, Germany

Ceramic coatings for metallic substrates can be prepared by the polymer-derived ceramics (PDC) route. Processing consists in the deposition in liquid phase of the coating solution containing a preceramic polymer onto the substrate by simple lacquer methods, followed by a thermal treatment at temperatures above 400 °C. This process overcomes limitations related to high costs and size/shape of parts to be coated, typical of ceramic coating techniques, such as thermal spraying and CVD/PVD. In this work, the PDC route is applied to realize a simple processing of a thermal barrier coating (TBC). $ZrSi_2$ and YSZ were used as active and passive fillers in combination with an (organo)silazane precursor to obtain a coating system with low thermal conductivity and stability up to 1000 °C. The composite coatings were applied onto steel sheets and on the inside of steel pipes (AISI 441), primarely coated with a bond-coat of polysilazane PHPS. Top-coat pyrolysis was conducted in air at 1000 °C. The developed coating system was characterized regarding conversion behavior, microstructure, porosity, adhesion, thermal properties and durability. Coatings with thickness of 50 µm and adequate microstructure for thermal barrier applications were obtained, resulting in adhesion above 20 MPa and thermal conductivity as low as 0.5 $W(mK)^{-1}$.

2:00 PM

(ICACC-HON-053-2018) CMAS attack – a serious challenge for environmental barrier coatings (Invited)

P. Mechnich*[1]

1. DLR - German Aerospace Center, Institute of Materials Research, Germany

Advanced ceramic matrix composites (CMC) promise higher energy efficiency of turbine engines due to substantially higher operation temperatures. The synergistic effect of heat and water-rich exhaust gases, however, is strongly affecting CMC lifetime. Consequently, the development of new, environmental barrier coatings (EBC) minimizing thermochemical degradation of CMC components is a continuing R&D topic. State of the art turbine components, typically Ni-based superalloys with ZrO_2 thermal barrier coatings (TBC) experience severe thermal-chemical degradation by ingested airborne inorganic particles commonly referred to as CMAS. In this context CMAS melt infiltration of TBC is a major reason for premature coating failure. Increasing turbine operation temperatures will aggravate the problem since all kinds of CMAS particles are expected to impinge on turbine components in fully molten, low viscous state. Therefore, new CMC components require EBC materials with much higher CMAS resistance than state-of the art TBC. A promising way to tackle CMAS melt-infiltration is the use of highly reactive EBC materials or alloying elements which can trigger rapid crystallization of CMAS melts, thus enabling the application of EBC-protected CMC also in demanding thermochemical environments. The presentation will give an overview on current developments and directions in this R&D field.

2:20 PM

(ICACC-HON-054-2018) Rare Earth Silicates as Advanced Environmental/Thermal Barrier Coating Candidates for SiC_f/SiC CMCs in Extreme Combustion Environment (Invited)

J. Wang*[1]

1. Shenyang National Laboratory for Materials Science, Institute of Metal Research, Chinese Academy of Sciences, High-performance Ceramics Division, China

SiC_f/SiC CMCs are the best candidates for high temperature structural material for next generation turbine engines. Environmental barrier coating is critically important to protect SiC-based CMCs from high temperature oxidation, water vapor corrosion and CMAS attack. A strategic perspective is to develop advanced environmental/thermal barrier coating (E/TBC) system with optimal property matches and long-term durability in extreme combustion environment. To promote the high throughput design for advanced graded E/TBC, a comprehensive data pool is constructed for the mechanical and thermal properties, as well as high temperature corrosion resistance of RE-silicate candidates. Our works highlighted some novel concepts and/or mechanisms to initiate high-throughput design and selection of E/TBC candidates for advanced SiC_f/SiC CMCs.

2:40 PM

(ICACC-HON-055-2018) Advanced Ceramics Coating as the Strategic Technologies for Future Innovation (Invited)

J. Akedo*[1]

1. National Institute of Advanced Industrial Science and Technology (AIST), ACT, Japan

In recent years, Global market of high-performance ceramic coating, is estimated to be in 2013 about 600 billion yen (5.68 billion dollars), In addition, 7.0 percent of the annual growth rate from 2014 to 2020 (CAGR) is expected, in 2020 about 1 trillion yen to reach (9.07 billion dollars) has been predicted. A coating process such as Aerosol Deposition (AD) method and Cold Spray (CS) method, which is based on collision of particle materials, attracts attention as the advanced coating technology recently. Those coating techniques deferent from conventional thermal spray method, among spray coating may use only mechanical impact pressure to form the material layer. It is expected to reduce energy, cost, difficulty to fabricate the thin or thick film coating with complicate material compositions and the number of processes during fabricating electronic devices and others, as well as to improve their performances substantially. AD process maybe one of the good candidate of the strategic advanced coating for a future innovative technology. In this presentation, The importance of advanced ceramics coating as the strategic process for a future innovative technology including with several successful cases will be introduced.

3:20 PM

(ICACC-HON-056-2018) Ceramic-metal/CNT/G-GO oxide composite coatings with tailored properties (Invited)

S. Seal*[1]

1. University of Central Florida, Mat. Sci. Eng, USA

Carbon nanotubes (CNT) and graphene/graphene oxide (GO) are potential reinforcements for producing metal-ceramic matrix composites and coatings with high strength and toughness. However, the critical issue of avoiding CNT agglomeration and introducing CNT-matrix anchoring has challenged many researchers to improve the mechanical properties of the CNT reinforced nanocomposite. In the current work, dispersed CNTs are grown on or mixed with ceramic and metal powder particles by a variety of techniques. These CNT reinforced coating and composites

show a promising enhancement in hardness and fracture toughness of the plasma-sprayed coating attributed to the existence of strong metallurgical bonding between metal ceramic particles and CNTs. In addition, CNT tentacles have imparted multi-directional reinforcement in securing the splats. High-resolution transmission electron microscopy shows interfacial fusion between metal-ceramic and CNT. Various strengthening mechanisms are discussed. A few examples of ceramic-metal-GO composites are also presented.

3:40 PM

(ICACC-HON-057-2018) Interfacial Issues in Brazing of Advanced Ceramics for Structural and Multifunctional Applications (Invited)

R. Asthana*[1]; N. Sobczak[2]; J. J. Sobczak[2]; M. Singh[4]

1. University of Wisconsin-Stout, Engineering and Technology, USA
2. Foundry Research Institute, Poland
4. Ohio Aerospace Institute, USA

Advanced ceramics are brazed using metallic interlayers for a variety of applications including heat exchangers, fuel cells, thermoelectric systems, and advanced sensors. High-temperature wetting of ceramics and the resulting interface formation govern the bonding in brazed assemblies in such applications. Investigations of the classical wetting behavior and interface characterization thus assume central importance in context of brazing. In this presentation, some results from high-temperature wettability studies using specially developed sessile drop and capillary purification test procedures (e.g., in-situ interface opening by droplet pushing) will be presented. In particular, the effect of oxide layer, ceramic dissolution and roughening, recrystallization and surface modification ahead of the wetting front, and capillary infiltration of substrate porosity will be discussed with reference to the brazing of oxide, carbide and nitride ceramics. High-temperature contact angle measurements on pure metals and alloys containing active elements in contact with ceramics will be presented together with the observations on interface characterization using OM, SEM, EDS and TEM. Implications of lab-based wettability test outcomes to industrial brazing of ceramics will be highlighted.

4:00 PM

(ICACC-HON-058-2018) Research on joining at Politecnico di Torino: Everything started more than 20 years ago (Invited)

M. Ferraris*[1]

1. Politecnico di Torino, Department of Applied Science and Technology, Italy

Origin, results and perspectives of 20+ years of research activity on joining at Politecnico di Torino, Italy, will be summarized and discussed. This talk will give an overview on our joining materials and techniques, on components we obtained by joining similar and dissimilar materials, our attempts to develop a reliable shear strength tests for joined materials, our industrial collaborations and international links, together with some news on our recently funded Centre on Advanced Joining Technology (J-TECH@POLITO).

4:20 PM

(ICACC-HON-059-2018) Non-conventional joining of a ceramic solid oxide fuel cell to a metallic interconnect (Invited)

R. Muccillo*[2]; E. N. Muccillo[1]

1. IPEN, Brazil
2. IPEN-UFABC, Brazil

Co-generation of energy (heat and electric power) with ceramic solid oxide fuel cells requires consolidation of anode-electrolyte-cathode single cells with a dense electrolyte and porous anode and cathode. The consolidation is usually achieved by sintering the three-layer single cells according to special procedures to produce flat square shape cells. Non-conventional consolidation of several ceramic materials used in energy-conversion devices has been done with success in several ceramic materials by electric field-assisted pressureless sintering, producing porous or dense components. The same approach has been tried in a flat single solid oxide fuel cell with yttria-stabilized zirconia solid electrolyte, lanthanum strontium manganite cathode and yttria-stabilized zirconia/nickel oxide anode with promising results, opening new routes for future low cost production of these devices. There is still a challenge in using that technique for joining ceramic single cells to metallic interconnector, one of the research areas of Dr. Mrityunjay Singh. Our efforts now are directed to carry out experiments aiming the joining of ceramic single cells to metallic interconnects by application of electric fields.

4:40 PM

(ICACC-HON-060-2018) Novel single crystals for electro-optical applications (Invited)

K. Shimamura*[1]; V. Garcia[1]

1. National Institute for Materials Science, Japan

Optical technology progress in a wide range of applications, and still demands the further development. Here, novel single crystals with advantageous characteristics will be introduced. $Tb_3(Sc_{1-x}Lu_x)_2Al_3O_{12}$ (TSLAG), CeF_3 and PrF_3 single crystals have been designed and grown for high-power laser machinery. They showed a higher visible-UV transparency and a larger Faraday rotation than $Tb_3Ga_5O_{12}$ (TGG). They are therefore very promising material in particular for new magneto-optical isolator applications in the UV-VIS-NIR wavelength. A new concept of high-brightness white LEDs based on Ce:YAG single crystal phosphors (SCPs), which can overcome the conventional temperature- and photo-degradation problems, is proposed. SCPs demonstrated excellent thermal stability with no temperature quenching, high values of luminous efficacy and increased quantum efficiency. Other single crystals investigated so far will also be introduced. Authors would like to thank to Tamura Corp., Koha Co., Ltd., Shinko Manufacturing Co., Ltd., and Fujikura Ltd., for the collaboration.

5:00 PM

(ICACC-HON-061-2018) Challenges and prospects for advanced laser ceramic processing

Y. Wu*[1]

1. New York State College of Ceramics, Alfred University, Department of Materials Science and Engineering, Kazuo Inamori School of Engineering, USA

Polycrystalline transparent ceramics are emerging as a highly promising alternative to single-crystal materials for potential utilization in a wide range of optics and photonics applications, most notably being ceramic lasers and scintillators. In active pursuit of successfully processing optical quality materials, crystallographic cubic ceramics have been studied, however, processing anisotropic transparent ceramics presents significant challenges due to their inherent birefringence. In attempt to process transparent non-cubic ceramics to achieve nanostructure grain size, a field-assisted sintering method is studied in which the ceramic samples can be quickly densified without significant grain growth. Meanwhile, laser ceramics could be made by dry and wet forming techniques. The gel-casting is a near-net shaping process for simple and complex shapes of ceramic fabrication using some of organic materials for the gelling reaction. The process has some disadvantages associated with toxicity, rigid conditions for reaction and high amount of organic addition. A newly developed gelling system without using toxic organic compounds has been investigated to develop transparent complex-shaped ceramics. This novel process is promising to fabricate large-size and complex-shaped transparent ceramics for optical and photonic applications.

5:20 PM

(ICACC-HON-062-2018) Advanced Ceramics, Today and Tomorrow in Japan (Invited)

T. B. Yano[1]; H. Sato*[1]

1. Japan Fine Ceramics Association, Japan

Japan Fine Ceramics Association (JFCA) has 90 members including major Japanese manufacturers and users of advanced ceramics and foreign subsidiaries in Japan. According to an industrial trend survey, the total domestic production of advanced ceramics in Japan reached approximately $ 250 million in 2016. To meet our members' needs, we strive to develop the root technologies for the future of advanced ceramics and offer a variety of projects which include developing international standards, hosting seminars and developing industrial roadmaps. Concerning the international standards development, JFCA serves as the international secretariat for ISO/TC206 of Fine Ceramics in addition to its role as the Japanese secretariat for ISO/TC206 and ISO/TC150 of Implant for Surgery. In this presentation, we will talk about our activities on R&D and international standards development and key points from the roadmap 2050 for advanced ceramics which we developed last year.

FS1: Bio-inspired Processing of Advanced Materials

Bio-inspired Processing III

Room: St. John

Session Chair: Joaquin Ramirez-Rico, Universidad de Sevilla

1:30 PM

(ICACC-FS1-019-2018) Composite Structural Modeling and Mechanical Behavior of Nanocarbon-based Composites with Nacre-like Structures (Invited)

Y. Su*[1]; Z. Li[1]; Q. Guo[1]; D. Xiong[1]; D. Zhang[1]

1. Shanghai Jiao Tong University, Materials Science and Engineering, China

Currently, how to effectively establish the relationship between composite structures and mechanical properties of nanocarbon-based composites has become one of the most important tasks. The present work aims to study the typical nanocarbon-based composites with nacre-like structures. On account of the statistical structural description of nanocarbon (e.g. CNT and graphene), the irregular morphologies of nanocarbon and composite structures of nanocarbon-based composites can be constructed, while the interfacial property and failure behaviour can also be considered in the nanocarbon-based composites. Meantime, the mechanical deformation of nanocarbon-based composites can be predicted and verified by experimental results. Based on the basic research work above, a common research framework can be built to indicate the mechanical behaviour of nanocarbon-based composites, from initial characterizations, to structure reconstructing, to interfacial regulations, to property prediction, which can provide us an important theoretical basis and design guidance for preparing, processing and/or optimizing nanocarbon-based composites.

1:50 PM

(ICACC-FS1-020-2018) Bio-inspired graphene-based nanocomposites (Invited)

Q. Cheng*[1]

1. Beihang University, School of Chemistry, China

With its extraordinary properties as the strongest and stiffest material ever measured and the best-known electrical conductor, graphene could have promising applications in many fields, especially in the area of nanocomposites. However, processing graphene-based nanocomposites is very difficult. So far, graphene-based nanocomposites exhibit rather poor properties. Nacre, the gold standard for biomimicry, provides an excellent example and guidelines for assembling two-dimensional nanosheets into high performance nanocomposites. The inspiration from nacre overcomes the bottleneck of traditional approaches for constructing nanocomposites, such as poor dispersion, low loading, and weak interface interactions. Herein, we summarizes recent research on graphene-based artificial nacre nanocomposites and focuses on the design of interface interactions and synergistic effects for constructing high performance nanocomposites.

2:10 PM

(ICACC-FS1-021-2018) Achieving High Strength and High Ductility in Metal Matrix Composites Reinforced with Discontinuous Three-Dimensional Graphene-Like Network (Invited)

C. He*[1]; X. Zhang[1]

1. Tianjin University, School of Materials Science and Engineering, China

In this work, we have developed an innovative in-situ processing strategy for the fabrication of metal matrix composites reinforced with discontinuous 3D graphene-like network (3D GN). The processing route involves the in-situ synthesis of encapsulation structure of 3D GN powders tightly anchored with Cu nanoparticles (NPs) (3D GN@Cu) to ensure mixing on the molecular level between graphene-like nanosheets and metal, coating of Cu on the 3D GN@Cu (3D GN@Cu@Cu), and consolidation of the 3D GN@Cu@Cu powders. This process can produce GN/Cu composites on a large scale, in which the in-situ synthesized 3D GN not only maintains perfect 3D network structure within the composites, but also has robust interfacial bonding with metal matrix. As a consequence, the as-obtained 3D GN/Cu composites exhibit exceptional high strength and superior ductility (the uniform and total elongation to failure of the composite are even much higher than the unreinforced Cu matrix).

2:30 PM

(ICACC-FS1-022-2018) Bio-inspired Ultra-black Coating with Hollow Carbon Microspheres

L. Pan*[1]

1. Harbin Institute of Technology, China

The reflective spectra and structures of several black butterfly's wings were investigated. The enhancement of optical absorption raised by the microstructures of black butterfly's wings was universally observed. The general mechanism of this enhancement was proposed, based on which a bio-inspired ultra-black coating was fabricated with carbon hollow microspheres as absorbent. Through optimizing the coating formulation, the highest solar absorption of the bio-inspired ultra-black coating is 0.983, which is evidently higher than that of traditional black coating with similar chemical ingredient.

3:10 PM

(ICACC-FS1-023-2018) Bioinspired optical structure for enhancement infrared absorption (Invited)

W. Zhang*[1]; D. Zhang[1]

1. Shanghai Jiao Tong University, State Key Lab of Metal Matrix Composites, China

Recently, an increasing number of researchers have directed their attention to the wings of lepidopterans (butterflies and moths) because of their dazzling colors. According to one previous study, these iridescent colors are caused by periodic structures on the scales that make up the surfaces of these wings. These materials have recently become a focus of multidiscipline research because of their promising applications in the display of structural colors, advanced sensors, and solar cells. This work will provide a broad overview of the research into these wings. Specifically, the review focuses on characterization and simulation of bioinspired optical materials templated from lepidopteran wings scales.

3:30 PM

(ICACC-FS1-024-2018) Electrochemical Fabrication of "Anole Skin"

N. Li*[1]; Y. Li[2]; J. Zhao[1]; H. Xu[1]; L. Pan[1]

1. Harbin Institute of Technology, School of Chemistry and Chemical Engineering, China
2. Harbin Institute of Technology, Center for Composite Materials and Structure, China

Fabrication of supported metal nanoparticles with desired properties and high stability has been an important topic in the field of nanotechnology. Traditional methods have been suffering from several challenges, including low reproducibility, poor control over the morphology of single unit, low stability because of the weak interaction between nanoparticles and substrate, and so on. In this work, an electrochemical deposition method has been developed to prepare ITO-supported Ag films with tunable optical properties and high stability. The Ag film is composed of Ag nanoparticles with controlled morphology. The as-obtained Ag film can be used as an effective sensor for the detection of different solvents, as the surrounding solvents changed, the color of the film will be changed as "Anole Skin" The sensor is highly stable and can be used for more than 100 times without any performance deterioration. This work can not only provide a new pathway for the preparation of supported metal nanoparticles with controllable morphology and desired property, but also pave the way for the application of such nanoparticles.

3:50 PM

(ICACC-FS1-025-2018) Tunable color of VO_2 films by a bionic micro-structure

S. Dou*[1]; Y. Li[1]

1. Harbin Institute of Technology, School of Astronautics, China

VO_2 film has an ungly color for smart window, here, inspired by the bright colors of butterfly wings, we used structural colors to regulate the colors. a monolayer SiO2 microphere was introduced into VO2 film, and the scattering of visible light lead to tunable vivo color.

4:10 PM

(ICACC-FS1-026-2018) Bio-inspired Bragg reflector made of silk-titanates nanocomposite as platform for humidity sensing

E. Colusso[1]; F. Omenetto[2]; A. Martucci*[1]

1. University of Padova, Industrial Engineering, Italy
2. Tufts University, USA

In this work, we report a structurally-colored multilayer film, which can be considered a Bragg reflector, with humidity-responsive optical properties, inspired to the cuticle of Hoplia coerulea. This beetle is able to modify its color in the presence of moisture, which affect the thickness and refractive index of the cuticle. In the same way, our multilayer structure responds to changes in the environmental humidity with a reversible color change. For the generation of interference color, we combined the regenerated silk fibroin, which has a refractive index of 1.55, with a nanocomposite made of silk and titanate nanosheets, a novel 2D material that presents a high refractive index. The multilayer film was fabricated through a layer by layer deposition from the respective water-based solutions by spin coating. The final structure exhibits an interference peak in the transmittance spectrum centered at 400 nm, whose position responds to changes in the relative humidity of the environment. The stimuli-responsive properties of the film were characterized and a simple optical model for the sensing mechanism was proposed. Specifically, the silk-based multilayer showed a reversible color change when exposed to different relative humidity, and good performances in term of reproducibility and stability over time.

FS2: Tomography and Microscopy based Modeling of Ceramics

Strain Characterization by Digital Image Correlation Technique

Room: Coquina Salon H
Session Chairs: Benoit Rousseau, LTN UMR CNRS 6607;
Tobias Fey, Friedrich-Alexander University Erlangen-Nürnberg

1:30 PM

(ICACC-FS2-001-2018) In-situ bending behavior and failure characterization of 3D needle-punched C/SiC composite (Invited)

Y. Dong*[1]; X. Shi[2]; Z. Zhang[1]; B. Pan[1]

1. Beihang University, China
2. Institute of Metal Research, Chinese Academy of Sciences, China

In-situ failure characterization of three-dimensional (3D) needle-punched C/SiC composites under three-point bending tests were experimentally investigated. The non-linear macroscopic load-displacement curves were obtained. The evolution of the non-uniform displacement and strain fields during loading were reproduced by stereo-digital image correlation (stereo-DIC). Macro-damage and fracture surface after the three-point bending test was investigated by using optical microscopy and scanning electron microscopy (SEM). The results show that the specimen had a quasi-ductile fracture behavior with a flexural strength of 352 MPa exhibiting a step-like load-displacement curve. The delamination and through-thickness cracking were observed, with occurrence of delamination taking precedence over the generation of through-thick cracking. The microstructure results show the crack deflection, fiber pullout, fiber shearing fracture, fiber/matrix interfacial deboning, matrix cracking are the mainly failure mechanisms.

2:00 PM

(ICACC-FS2-002-2018) Advanced digital volume correlation for large non-uniform deformation measurement of high-resolution volume images (Invited)

B. Pan*[1]; B. Wang[1]

1. Beihang University, China

Since it was developed in 1999, Digital volume correlation (DVC) has experienced great improvements in both accuracy and efficiency. However, DVC still faces challenges in practical applications, such as massive memory requirement when processing high-resolution volume images and serious decorrelation effect occurring during large deformation measurement. To address these issues, we propose a flexible, robust and accurate DVC approach that can be implemented in personal computers with limited random-access memory and applicable to high-resolution volume images subjected to large non-uniform deformation. By combining several improvements, accuracy, precision, efficiency and flexibility in processible image size of DVC measurement were enhanced and then demonstrated by analyzing high-resolution volume images undergoing large deformation in numerical tests. Finally, by combining X-ray tomographic imaging and the presented DVC technique, in-situ stepwise uniaxial compression tests of pomelo peel samples were performed, and then internal full-field large non-uniform deformation response and morphological evolution under compaction were explored.

2:30 PM

(ICACC-FS2-003-2018) In-stite observation and strain measurement at high temperature by cutting off thermal radiation (Invited)

H. Kakisawa*[1]

1. National Institute for Materials Science (NIMS), Japan

In-stite observation and strain measurement at high temperature system was developed. A bandpass filter or confocal optical system is used to cut off thermal radiation which causes difficulty in optical observation at high-temperature, specifically over 1273 K while the blue or ultraviolet illumination is done on a sample. The systems were proven to be effective up to 1673 K for obtaining microscopic images without degradation of contrast by thermal radiation. The constant contrast of obtained images at various temperatures enables continuous strain measurement during heating or cooling process using digital image correlation. The configuration of the systems developed and some examples of obtained images and strain measurement for ceramics and ceramics matrix composites are presented. Production of heat resistant microscopic speckle pattern is also shown.

Characterization Technologies

Room: Coquina Salon H

Session Chairs: Ulf Betke, OvGU Magdeburg; Tobias Fey, Friedrich-Alexander University Erlangen-Nürnberg

3:20 PM

(ICACC-FS2-004-2018) Fabrication and characterization of carbon monolith

R. Inoue*[1]; Y. Kaneda[1]; Y. Kogo[1]

1. Tokyo University of Science, Japan

We developed porous carbon material with interconnected structure by carbonization of phenol monolith. Phenol monolith was fabricated by polymerization-induced phase separation (PIPS). Benzenenesulfonyl chloride, sodium carbonate, and ethylene glycol were used as raw materials. The effect of composition of resin mixture, curing time and viscosity of phenol resin on the pore structure was examined by conventional procedures, such as scanning electron microscopy (SEM), mercury intrusion porosimetry (MIP). In addition, three dimensional microstructures of developed materials were reconstructed using X-ray computed and focused ion beam/ scanning electron microscope (FIB-SEM) tomography. Phenol monolith with different pore size was obtained by changing composition of resin mixture, curing time and viscosity of phenol resin. Process parameters for controlling pore size was successfully optimized. Based on experimental results and analysis, the relation among process parameters, morphologies, and physical properties of carbon monolith will be presented.

3:40 PM

(ICACC-FS2-005-2018) Assessment of Thermal Fatigue Behavior of Active Metal Brazing Substrates during Temperature Cycling (Invited)

Y. Zhou*[1]; H. Miyazaki[1]; H. Hyuga[1]; S. Fukuda[1]; K. Hirao[1]

1. National Institute of Advanced Industrial Science and Technology (AIST), Japan

Metalized ceramic substrates for high power electronic devices are generally fabricated by brazing ceramics with conductive metal at an elevated temperature. The difference of coefficients of thermal expansion between ceramic and metal generates large thermal stresses in both constituent materials during brazing process as well as in use, which may generate cracks at the interface or in the ceramic body. In this investigation the effect of temperature cycling from -40 oC to 250 oC on Si_3N_4 based active metal brazing substrates was examined to evaluate their reliability in harsh environment. The extent of damage of bonded interface, in particular crack generation and propagation in the ceramic part, during temperature cycling was evaluated by means of scanning acoustic microscopy, micro-focus X-ray computed tomography, and measurement of residual bending strength. This work was supported by Council for Science, Technology and Innovation (CSTI), Cross-ministerial Strategic Innovation Promotion Program (SIP), "Next-generation power electronics/Consistent R&D of next-generation SiC power electronics" (funding agency: NEDO).

Analysis of Sintering and Solidification

Room: Coquina Salon H

Session Chairs: Hideki Kakisawa, National Institute for Materials Science (NIMS); Satoshi Tanaka, Nagaoka University of Technology

4:10 PM

(ICACC-FS2-006-2018) Micro CT observation of macroscopic pore evolution in alumina ceramics during sintering (Invited)

S. Tanaka*[1]

1. Nagaoka University of Technology, Materials Science and Technology, Japan

Engineering ceramics contains multi-scale pores from submicron to several tens microns. Coarse pores, particularly, limits their application. Recently, the technique of micro focus x-ray computed tomography (micro- CT) has progressed, and we can use it easily to detect coarse pores in ceramics without fracture. In this study, we demonstrate the coarse pores evolution during sintering sequentially using a micro-CT. For experimental, two types of alumina granules were used. One grade was prepared from a slurry without a binder, another included a binder. The binder was segregated on the granule surface. Size of primary particles in granules were 0.1 μm. Powder compacts were formed by uniaxial pressing at 40 MPa and subsequent cold isostatic pressing at 200 MPa. The structure in a sample with size $1mm^3$ during sintering was observed sequentially by micro- CT. The sample was heated to an objective temperature and was picked up from the furnace. The structure in the sample was observed by the micro-CT. After observation, the sample was heated in the furnace to next temperature. The same region in the sample was always observed. As a result, the evolutions of coarse pores were succeeded to be observed sequentially. Some clusters of pores with several micron formed at the interstices between granules in the powder comact became larger coarse pores with several tens micron with sintering.

4:40 PM

(ICACC-FS2-007-2018) Distinction of stages of sintering from 3D visalization of microstructure in sinteting by using X-ray microtomography (Invited)

G. Okuma*[1]; S. Tanaka[2]; F. Wakai[1]

1. Tokyo Institute of Technology, School of Material and Chemical Technology, Japan
2. Nagaoka University of Technology, Materials Science and Technology, Japan

Sintering is a common process during which nanoparticles and microparticles are bonded, leading to the shrinkage of interstitial pore space. Recent advances in X-ray microtomography revealed that the 3D microstructural evolution during sintering is far more complicated than the simplified model. This limits the applicability of classical models in real situations. The direct measurement of a 3D structure, which is now readily available from X-ray tomography, is the first step to understand the realistic property-microstructure relationship during sintering. We presented a topological model of sintering to understand the morphological evolution during sintering through microstructural images obtained from X-ray tomography. In the continuum model of sintering, shrinkage rate is a mechanical response of porous body to the thermodynamic

driving force, sintering stress. It is recognized that the most appropriate method for determining sintering stress should be selected depending on the sintering stages. However, no theoretical criterion exists for distinguishing stages of sintering by using relative density. Here, we discuss about the quantitative knowledges on topological properties, which provide insights for distinguishing stages of microstructural evolution in sintering. The result is general, and it provides criteria to distinguish the stages of sintering.

5:10 PM

(ICACC-FS2-008-2018) Analysis of Densification Behavior Depending on Grain Growth for Zirconia

B. Kim*[1]; K. Morita[1]; H. Yoshida[1]; J. Li[1]; H. Matsubara[2]

1. National Institute for Materials Science (NIMS), Japan
2. Tohoku University, Japan

The densification behavior of powder compacts can be characterized by determining the activation energy, grain-size exponent and unspecified density function. Several methods have been proposed to determine the activation energy and the grain-size exponent, but no method for the density function. In this study, a method is proposed to determine the unspecified density function during isothermal sintering of Y_2O_3-stabilized zirconia. An interesting point is that the relationship between the grain size G and the relative density D is not constant but dependent on the temperature, though a constant relationship has widely been recognized. With increasing temperature, the density-dependence of the grain size decreases in the intermediate stage, whereas it increases in the final stage. The grain-size exponent evaluated is 2.75 and 1-2 in the intermediate and final stage, respectively. The grain-size exponent of 1-2 in the final stage was not expected, because almost all theoretical models have predicted an exponent of 4 for a mechanism of grain-boundary diffusion. In our experiments, however, the grain-size exponent of 4 apparently yields a large deviation from the experimental densification kinetics. With the grain-size exponent and the density function determined experimentally, the existing theoretical models are evaluated and the sintering mechanism is discussed.

5:30 PM

(ICACC-FS2-009-2018) Non-destructive analysis and strength prediction of ceramics using optical coherence tomography (Invited)

J. Tatami*[1]; F. Sakamoto[1]; T. Takahashi[2]; M. Iijima[1]

1. Yokohama National University, Japan
2. Kanagawa Institute of Industrial Science and Technology, Japan

In order to improve the reliability of ceramics, it is necessary to predict their strength based on detection of the position and geometry of defects by non-destructive analysis. Optical coherence tomography (OCT) is an optical imaging technique to observe internal structure of opaque objects based on the interference of lights. The OCT is characterized by three dimensional and non-destructive imaging with high speed and high resolution. Although this technique has been applied to the medical application, non-destructive analysis of ceramics using OCT has not been investigated. In this study, optical coherence tomography (OCT) was applied to the non-destructive analysis and strength prediction of ceramics. Artificial defects in ceramics were formed using spherical carbon particles and graphite particles, SiO_2 ceramics and Al_2O_3 ceramics prepared by conventional powder processing were used as the matrix. The OCT observation showed that spherical pores and crack-like flaws were uniformly dispersed in the ceramics. The strength predicted using the size and position of the defects and the fracture toughness was found to be good agreement with the strength measured by 3-point bending test. Consequently, it was shown that the OCT observation is very effective in the nondestructive analysis and strength prediction of ceramics.

FS3: Chemical Processing of Functional Materials: Understanding the Conversion of Molecular Structures to Solid-State Compounds

Materials Processing I

Room: Coquina Salon A

Session Chairs: Philippe Miele, Ecole Nationale Superieure de Chimie de Montpellier; Zhaoju Yu, Xiamen University

1:30 PM

(ICACC-FS3-009-2018) Nanostructured Ceramic Coatings Synthesized by Metal-Organic Chemical Vapor Deposition (Invited)

A. Ito*[1]

1. Yokohama National University, Japan

We have demonstrated formation of unique nanostructures in various ceramic coatings synthesized by chemical vapor deposition using metal-organic precursors (MOCVD). Functionalization of coatings made with traditional engineering ceramics, such as Al_2O_3, ZrO_2 and SiC, with the control of orientation and nanostructure is of great interest in both of academic and industrial aspects. This talk will introduce you to recent topics on nanostructure formation in Al- and Si-based coatings via MOCVD process with a significant deposition rate—orientation control of α-Al_2O_3 films, feather-like structures in β-Al_2TiO_5 films, and nanodendrite formation in Al_2O_3–ZrO_2 nanocomposite films. Polytype formation between 3C and 2H in SiC coatings by different MO precursors will be discussed. In addition, development of Yb–Si–O environmental barrier coatings for SiC-reinforced SiC matrix composite for a next generation gas turbine engine might be presented. This work was supported in part by KAKENHI, Japan Society for the Promotion of Science, and Cross-ministerial Strategic Innovation Promotion Program (SIP), "Structural Materials for Innovation, SM[4]I (Funding agency: JST)".

2:00 PM

(ICACC-FS3-010-2018) Smarts molecular precursors for chemical processing of functional nanomaterials (Invited)

S. Daniele[1]; S. Mishra*[1]

1. IRCELYON, University Lyon 1, France

Chemical solution routes (i.e. Sol-gel, MOD) are usually coupled to the design of tunable molecular precursors and the industrial development of such techniques is strongly dependent of and associated to the inorganic molecular engineering. Indeed, they are usually the starting point of the processes and influence the micro-, nano-structure and finally the nature and the properties of the materials. Furthermore, the use of well-defined precursors also allows establishing relationships between precursors and the final material and getting some insight in their transformation. The aim is then to design homo or heterometallic ("single-source") precursors which will be converted into materials by "bottom-up" approaches by having (i) a formulation matching that of the materials, especially stoichiometry between metals for multimetallic materials, (ii) appropriate physicochemical properties (solubility, thermal stability), (iii) the required chemical functionalities (coordination sphere with an appropriate set of ligands), and (iv) a clean, low temperature conversion into materials and minimize undesired residues in them. Such concepts will be addressed in this presentation about well-defined metal-organics precursors for up-converting ($NaGdF_4$ co-doped Ln^{3+}, Ln = Eu, Tb, Yb), down-converting (ZnO/polymer) and thermoelectric ($Sn_xTi_{1-x}O_2$ doped with M^{5+}, M= Nb, Ta) nanomaterials.

2:30 PM

(ICACC-FS3-011-2018) "Free" Carbon in Silicon Oxycarbide Ceramics: Multiple Stages of Structure Formation

P. Kroll*[1]

1. UT Arlington, USA

Combining structure modeling, ab-initio molecular dynamic simulations, million-atom-simulations, experimental as well as computational NMR studies we explore "free" carbon and its impact on structures of SiCO ceramics. We observe distinct stages of structure formation within the "free" carbon phase. Isolated carbon units of a molecular precursor are initially well dispersed throughout the material. Upon annealing they combine to larger but finite segregations of single-layered carbon sheets. These carbon segregations separate the surrounding SiCO glass matrix, essentially containing it in small domains. Continuity and sizes of these domains are related to the amount of free carbon and to the composition of the material. Further annealing yields bending of single layers towards tubular carbon structures. Formation of tubes depends on the viscosity of the surrounding SiCO glass phase, which in turn is determined by composition. Ultimately, tubular structures convert into graphite segregations.

Materials Processing II

Room: Coquina Salon A
Session Chairs: Maarit Karppinen, Aalto University; Shashank Mishra, University of Lyon 1

3:10 PM

(ICACC-FS3-012-2018) Functional Nanoscaled Silicon-Based Ceramics and Ceramic Composites via Molecular Design (Invited)

R. Riedel*[1]

1. TU Darmstadt, Materials Science, Germany

This presentation is devoted to concepts for the synthesis of novel multifunctional ceramics and ceramic-based composites with a tailor-made nanoscaled structure. Industrial demands on future technologies have created a need for new material properties which exceed by far those of materials known today and which can only be produced by designing the respective microstructure at a nanoscale. In particular, molecularly derived amorphous and polycrystalline ceramics have to be considered as novel material classes and are produced by means of cross-linking routes in various states of condensation. Possible fields of application for materials produced at a nanoscale are key technologies of the 21st century such as transport systems, information technology, energy as well as environmental systems and micro- or nano-electromechanical systems. In particular nanostructured polymer-derived ceramics for applications in Li-ion batteries, high-temperature resistant nano-sized carbides and nitrides with a variety of integrated functional (sensing, electrical and thermal conductivity, piezoresistivity, etc.) and mechanical (ultra-high hardness, creep resistance, etc.) properties suitable for e.g. environmental and thermal barrier coatings (EBC and TBC) will be highlighted and discussed.

3:40 PM

(ICACC-FS3-013-2018) Design of Metal Oxide Nanoparticles: Toward the Control of the Electrochemical Properties

S. Cassaignon*[1]; O. Durupthy[1]; D. Portehault[1]

1. Sorbonne University, UPMC, LCMCP, France

Emergence of nanotechnology increasingly shows examples of material potentiality, which has at least one dimension lower than 100 nm. The preparation of nano-objects by soft chemistry in aqueous solution with crystal structure, size and morphology perfectly controlled is based on the use of molecular precursors and adjustment of the physico-chemical parameters throughout precipitation of the solid. The growth of nanocrystals can be limited or on the contrary favored in some crystallographic directions. It is also possible to control other parameters such as the nature of the precursor, the presence of ions or molecules in the reaction medium and also to involve redox processes in addition to the acido-basic reactions. This last point can significantly enhance the potentiality of this chemistry in the design of particles. Along with the study of formation mechanisms, our attention focused on the development of higher complexity objects that results in the synthesis of nano-textured objects for various applications. Hierarchical materials with multiple scales of organization and often formed from the assembly of nanoparticles, raises a growing interest, mainly thanks to their ability to combine the functions of the various elementary units. This allows to consider interesting applications of these systems in various fields and especially in the field of the Energy and Environment.

4:00 PM

(ICACC-FS3-014-2018) Developments of nanostructured metal oxide thin films for photoelectrochemical water splitting

S. Bera*[1]; S. Kwon[1]

1. Pusan National University, School of Materials Science and Engineering, Republic of Korea

Different nanostructured films of metal oxides having hierarchical arrangement are the most exciting prospect in the material science for optoelectronic application. The hierarchical arrangement represents a critical building block of the nanomaterials which greatly enhances the novel optical, electronic and mechanical properties of the films. In this respect, researchers have investigated several bottom up approaches on solution and gas phase methods for the synthesis of hierarchical nanostructured thin films. However, the ability to fabricate the large area nanostructured thin films with tunable control of orientation, surface morphology, repeatability is a challenging task in the scientific community. However, atomic layer deposition (ALD) assisted precisely controlled solution process can be the efficient approach for the growth of hierarchical thin films on wide range of substrate. Moreover, ALD can significantly control the film thickness at atomic scale. Herein, we present a simple solution or ALD assisted solution processed hierarchical metal oxide thin films. We propose to use the nanostructured film for photoelectrochemical water splitting and found enhanced photoelectrochemical performance. We demonstrate that the hierarchical morphologies are advantageous to enhance light harvesting and also provide direct conduction pathway to hamper the charge recombination.

4:20 PM

(ICACC-FS3-015-2018) Synthesis, microstructure and advanced functional properties of SiCN-based nanocomposites (Invited)

Z. Yu*[1]

1. Xiamen University, College of Materials, China

Silicon carbonitride (SiCN) based ceramic nanocomposites modified with incorporated carbon nanophase are effectively synthesized via the polymer-to-ceramic transformation technique starting from suitable single-source-precursors. In this lecture, the single-source-precursor molecular structure, complex microstructure, and dielectric properties of the obtained SiCN-based nanocomposites will be discussed. The developed single-source-precursor approach allows to homogeneously disperse nanocarbon phase in ceramic matrices, which exhibits significant advantage over the physical blending method. The carbon nanophase plays a critical role in their dielectric properties. The relationship between the obtained nano/microstructure of the synthesized SiCN-based nanocomposites and their property features will be highlighted. Moreover, due to the significantly enhanced dielectric properties, our SiCN-based nanocomposites possess outstanding electromagnetic (EM) performance and versatile designability from EM absorbing to shielding behavior, which shows substantial progress beyond the state of the art.

4:50 PM

(ICACC-FS3-016-2018) Probing local environments in PDCs with solid-state NMR combined with ab-initio calculations

C. Gervais*[1]; C. Salameh[1]; M. Schmidt[2]; A. Viard[2]; S. Bernard[2]; F. Babonneau[1]; P. Miele[2]

1. UPMC, LCMCP, France
2. IEM, France

In the polymeric route to ceramics, the polymer architecture may strongly influence the structure of the final ceramic. It is therefore essential to get a precise description of its structure, and then be able to follow step-by-step the polymer-to-ceramic conversion. High resolution solid state magic angle spinning nuclear magnetic resonance (MAS NMR) appears therefore to be extremely useful to characterize the different intermediates. This technique is sometimes challenging because of the lack of sensitivity of the isotopes in natural abundance (^{15}N) or a lack of resolution of the spectra due to the quadrupolar character of the isotopes (^{11}B or ^{27}Al). However, an appropriate combination of techniques such as double resonance experiments, multiple-quantum (MQ) MAS sequence for quadrupolar nuclei, can help to get a better description of the various environments present in the structures. Nonetheless, even using a large panel of specific sequences, interpretation of the spectra may be difficult due to small chemical shift ranges resulting in overlapping signals. Moreover, observed signals may not be in full agreement with expected structures of the PDCs that are often very complex. In this context, ab- initio calculations of NMR parameters can be extremely useful in helping to the understanding of the spectra and for the assignment of the sites present in the polymers and the ceramics.

5:10 PM

(ICACC-FS3-018-2018) Low Temperature Joining of Borosilicate Glass

E. Muskovin*[1]; W. Fahrenholtz[1]; R. Brow[1]; J. Buckner[2]

1. Missouri University of Science & Technology, Material Science and Engineering, USA
2. Applied Technology Associates, USA

Quaternary ammonium hydroxides were used to join borosilicate glass at temperatures below 200°C. Silica exhibits high solubility in quaternary ammonium hydroxides, which can be exploited to form high strength organic-inorganic hybrid joints that are stable at elevated temperatures. Solidification of the joints occurred at temperatures below 200°C in inert environments. The joining layer was examined using thin film x-ray diffraction, x-ray photoelectron spectroscopy, scanning electron microscopy and energy dispersive spectroscopy.

Friday, January 26, 2018

S10: Virtual Materials (Computational) Design and Ceramic Genome

Modeling of Functional Ceramics I

Room: Coquina Salon F
Session Chair: Per Eklund, Linkoping University

8:30 AM

(ICACC-S10-025-2018) Functional electronic ceramics material research combining atomic level structure analysis and theoretical calculations (Invited)

H. Moriwake*[1]

1. JFCC, Japan

In this talk, I will try to explain our recent achievements of the functional electronic ceramics material research combining atomic level analysis and theoretical calculations (e.g., Li ion battery materials, ferroelectric materials, and so forth). For one of these example, Li-ion battery material, solid-state Li ion conductor (La,Li)TiO_3 (LLTO). The atomistic structure of 90° domain boundaries in LLTO has been determined using HAADF-STEM observations. Important finding is that at 90° domain boundary, each cells connected by La layer. The calculated Li conduction activation energy for LLTO bulk shows exceptionally small value (E_a = 0.2 eV). However, this theoretical E_a was quite small to compare with experimentally reported value (E_a = 0.4 eV). To reveal the origin of this E_a discrepancy between theory and experiments, secondary, we examined Li-conduction energies at 90° domain boundary. Li conduction through La layer shows very high E_a of 3.6 eV. However, if we assume the La vacancy at La layer, conduction energy significantly decreased to 0.6 eV. This conduction energy well corresponds to experimental value. Our calculations reveal that Li conduction in LLTO should be strongly influenced by 90° domain boundaries in this system.

9:00 AM

(ICACC-S10-026-2018) Electronic Structure and Chemical Bonding in MXenes and MAX phases Investigated by Density Functional Theory and X-ray Spectroscopy (Invited)

M. Magnuson*[1]

1. Thin Film Physics Division, Linköping University, Department of Physics, Chemistry and Biology, (IFM), Sweden

The electronic structures and chemical bonding in the 2D ceramic materials MXenes and MAX-phases are investigated by ab initio electronic structure calculations in comparison to X-ray spectroscopy. For MXenes, the role of functional –OH, –O and –F temination groups at the interfaces and their local symmetries at different adsorption sites are discussed. Experimental spectroscopic studies are important tests of state-of-the-art electronic structure density functional theory. Calculated spectra using density-functional theory (DFT) including core-to-valence dipole matrix elements are found to yield consistent spectral functions of experimental data. By varying the constituting elements and structures in MXenes and MAX-phases, a change of the electron population is achieved causing a change of covalent bonding between the laminated layers, which enables control of the macroscopic properties of the materials. Synchrotron radiation techniques such as bulk-sensitive soft X-ray absorption and emission spectroscopy are shown to be particularly useful for detecting detailed symmetry in the electronic structure and yield anisotropy information about internal monolayers and termination groups at the interfaces. Angle- and polarization-resolved measurements reveal differences in the occupation of the orbitals across and along the laminate basal plane.

9:30 AM

(ICACC-S10-027-2018) Li storage in SiCO anode materials (Invited)

P. Kroll*[1]

1. University of Texas, Arlington, USA

The high reversible Li storage capacity of amorphous SiCO – two-fold higher per gram of material than that of graphite – is supported by multiple experimental studies and persists even after 1000 cycles of charging and discharging. If "free" carbon would be the only storage site in SiCO, then its capacity would be 1 Li for every 1 "free" C – six-fold in comparison to graphite, which reaches its limit with the composition LiC_6. In our continuous efforts to understand this unique effect, we present atomistic models of amorphous SiCO and show our results of density functional theory calculations. Li always prefers bonding to O and is found as cationic Li^+, while the surplus electron is promoted to unoccupied states. We find that the SiC content of the SiCO glass phase impacts the chemical potential of unoccupied states and promotes favorable insertion. We discuss the impact of "free" carbon with respect to the formation of nano-domains separated by graphitic segregations. Li migrating through the interfacial region react with the top layer creating a partially reduced LiSiO phase, which adds significantly to the storage capacity of the material. The picture we deliver provides motivation for new experimental studies.

Modeling of Functional Ceramics II

Room: Coquina Salon F
Session Chair: Peter Kroll, UT Arlington

10:20 AM

(ICACC-S10-028-2018) Visualization of Energy-Structure Relationships of Mn^{4+} in Oxides with Local Symmetries Specified by Three or Four Structural Parameters (Invited)

K. Ogasawara*[1]

1. Kwansei Gakuin University, Department of Chemistry, Japan

Mn^{4+}-doped phosphors are drawing attention as red phosphors to improve the color rendering properties and production cost of white LEDs. Although Mn^{4+}-doped fluoride phosphors are already commercially used, Mn^{4+}-doped oxide phosphors are more desirable considering the stability against high temperature and high humidity. However, in the case of Mn^{4+} in oxides, the shortest emission wavelength reported so far is still longer than the required wavelength. In order to provide guidelines to control the emission wavelength of Mn^{4+}-doped oxides, we have recently constructed various energy maps of Mn^{4+} in oxides in terms of structural parameters such as bond lengths or bond angles by performing first-principles multiplet calculations using MnO_6 clusters with various symmetries such as D_{4h}, and D_{3d}. However, for lower symmetries with three or more structural parameters, construction of similar maps is not straightforward. In this work, in order to visualize the energy-structure relationships of Mn^{4+} in oxides with D_3 and C_{4v} symmetries, three-dimensional energy maps were constructed by performing first-principles configuration-interaction calculations using MnO_6 clusters with various combination of the structural parameter values. The energy-structure relationships for these symmetries were clarified by the combination of these energy maps.

10:50 AM

(ICACC-S10-029-2018) Novel layered thin-film materials for contacts and thermoelectrics by integrated theoretical-experimental studies (Invited)

P. Eklund*[1]

1. Linkoping University, Dept. of Physics, Chemistry, and Biology, Sweden

The large class of layered ceramics encompasses both van der Waals (vdW) and non-vdW solids and find a wide range of applications. In this invited talk, I will present examples of our work on layered ceramics from experimental and theoretical investigations of oxide, nitride, and carbide thin film systems. Alloying approaches for thermoelectrics in CrN- and ScN-based systems are discussed as well as layered flexible oxide thermoelectrics. Furthermore, I will discuss our results on Au- and Ir-based MAX phases of formation of Ti_3AuC_2 and $Ti_3Au_2C_2$ phases with up to 31% lattice swelling by a substitutional solid-state reaction of Au into Ti_3SiC_2 single-crystal thin films with simultaneous out-diffusion of Si. These phases form Ohmic electrical contacts to SiC and remain stable after 1000 h at 600 C in air.

11:20 AM

(ICACC-S10-030-2018) High-throughput dopant screening for low power-consuming and fast phase change memory material

H. Choi*[2]; M. Choi[1]

1. Hanyang University, Republic of Korea
2. Virtual Lab Inc., Republic of Korea

Achieving high speed and low-power consumption is the ultimate goal of materials engineering for non-volatile and neuromorphic devices. A new material design has been done with density functional theory (DFT) screening method, which provides 1000 times cheaper cost and 150 times faster development period for nonvolatile phase change memory material. We screened doping elements systematically and investigated for In_3SbTe_2 (IST) phase change material being initiated by our previous fundamental study on the lattice distortion induced amorphization of IST [M. Choi, et al. Sci. Rep. 5 (2015) 12867]. Among the 40 candidate dopants, it is predicted that yttrium (Y) is the ideal element because the Y can provide energetically stable doping on In-site and the largest lattice distortion among In-substituting dopants. This distortion effect is important factor to discover a new phase change material as well as high-throughput screening method with the least error. Experimentally, Y-doped IST devices show that the 'set' and 'reset' speed quite well improved, which lowers the power consumption in Y-doped IST phase change memory and neuromorphic devices.

11:40 AM

(ICACC-S10-031-2018) First-Principles Calculations on Impurity Poisoning of Pt Alloy Anode Catalyst in PEFC

K. Kuranari*[1]; J. Xu[1]; Y. Ootani[1]; N. Ozawa[1]; M. Kubo[1]

1. Institute for Materials Research, Tohoku University, Japan

Platinum catalysts are typically used as anode catalysts for polymer electrolyte fuel cell (PEFC). The fuel for PEFC contains a small amount of impurities such as CO, NH_3 and H_2S. These impurities degrade the hydrogen oxidation reaction (HOR) activity of the anode catalyst. Therefore, the development of the high impurity tolerant anode catalyst is required. It was experimentally found that Pt-Fe alloy nanoparticle with two atomic layers of Pt-skin(Pt_{2AL}-PtFe) has high CO tolerant HOR activity. Moreover, the high CO tolerant catalyst has been developed by adding SnO_2 nanoparticles to the Pt/C catalysts. However, NH_3 and H_2S tolerance of these catalysts is not clear. In this study, we calculated the adsorption energy of CO, NH_3, and H_2S on Pt_{2AL}-PtFe and Pt/SnO_2 and analyzed the electronic states to investigate the impurity tolerance of these catalysts using first-principles calculation. The adsorption energies of CO, NH_3 and H_2S on Pt_{2AL}-PtFe(111) are lower than those on Pt(111). Thus, it was found that the adsorption of not only CO but also NH_3 and H_2S on

Pt_{2AL}-PtFe is suppressed compared to pure Pt. We will explain about the effect of the addition of SnO_2 nanoparticles on the impurity tolerance and the electronic states analysis at the conference.

Honorary Sympoisum: Advancing Frontiers of Ceramics for Sustainable Society Development - International Symposium in Honor of Dr. Mrityunjay Singh

Advancing Frontiers of Ceramics VIII -Novel Processing 2

Room: Coquina Salon E

Session Chairs: Roger Narayan, NC State University; Richard Sisson, Worcester Polytechnic Institute

8:30 AM

(ICACC-HON-063-2018) Bioceramic Substrates for Engineering Applications: Particles Filtration in Diesel Engines (Invited)

P. Orihuela Espina[2]; A. Gómez Martín[1]; J. Ramirez-Rico[1]; J. M. Fernandez*[1]; R. Chacartegui Ramírez[2]; M. Singh[3]

1. UNIVERSIDAD DE SEVILLA, FÍSICA MATERIA CONDENSADA, Spain
2. UNIVERSIDAD DE SEVILLA, INGENIERÍA ENERGÉTICA, Spain
3. Ohio Aerospace Institute, USA

Biomorphic SiC ceramics are produced by a low-cost and eco-friendly route with near-net shape potential and amenable to rapid prototyping. Several applications have been proposed for these materials, owing to their good thermomechanical properties and characteristic microstructure. In this work, the suitability of biomorphic Silicon Carbide (bioSiC) as substrate for Diesel Particulate Filters (DPF) has been assessed. Compared to the standard SiC used in commercial DPF, bioSiC has an anisotropic, hierarchical microstructure that may be tailored in permeability, porosity and pore size. Comprehensive characterization study was performed over a number of bioSiC samples made from different precursors. Then, two experimental campaigns were carried out, one with a thermal oil boiler and another one with a diesel automotive engine, in order to study the filtration efficiency and the pressure drop of some simple-shaped bioSiC filter samples. Finally, a small-scale wall-flow filter was designed and built, and an accurate measurement of its pressure drop and filtration efficiency was done under controlled conditions with a laboratory-generated soot laden gas stream. This study has shown that bioSiC may be a good candidate for manufacturing particle filters, complying with the current emission limits, and providing with some independency between particle filtration efficiency and pressure drop.

9:00 AM

(ICACC-HON-064-2018) High-porosity geopolymer components by direct foaming and direct ink writing (Invited)

P. Colombo*[1]; C. Bai[1]; G. Franchin[1]; A. Conte[1]; A. Milan[1]; P. Scanferla[1]

1. University of Padova, Industrial Engineering, Italy

High-porosity metakaolin-based geopolymer foams were fabricated by a gelcasting technique using hydrogen peroxide (foaming agent) in combination with Tween 80 (surfactant). Slurries processed in optimized conditions enabled to fabricate potassium based geopolymer foams with a total porosity in the range of ~67 to ~86 vol% (~62 to ~84 vol% open), thermal conductivity from ~0.289 to ~ 0.091 W/mK, and possessing a compressive strength from ~0.3 to ~9.4 MPa. Moreover, parameters influencing the compressive strength, the porosity, the thermal conductivity, and the cell size distribution were investigated. The results showed that the cell size and size distribution can be controlled by adding different content of surfactant and foaming agent. The foamed geopoymer can also be used as adsorbents for the removal of copper and ammonium ions from wastewater. The foams, due to their low thermal conductivity, could also be used for thermal insulation. It was also possible to produce geopolymer formulations that could be printed using additive manufacturing technology (Direct Ink writing), which enabled to produce components with non-stochastic porosity.

9:20 AM

(ICACC-HON-065-2018) Biohybrid Sol-Gel Materials for Renewable Fuel Production (Invited)

K. Johnson*[1]; M. Longo[1]; S. H. Risbud[1]

1. University of California, Davis, Chemical Engineering and Materials Science, USA

Hydrogen fuel has become a popular option for a future fuel source to replace dwindling natural resources. Despite this push, the bulk of today's hydrogen fuel is still produced from fossil fuels, tethering production to non-renewable sources and further release of greenhouse gases. The use of photocatalytic semiconductors for solar-driven water-splitting offers a clean solution to hydrogen fuel production. Titanium dioxide sensitized to visible light represents a simple and promising system for application in such an endeavor. Sol-gel synthesis methods allow for the development of titanium dioxide with highly tunable properties while providing a simple method for inclusion of sensitizing agents without the use of addition fixing agents or lengthy adsorption stages. Rather than using conventional dyes to sensitize the titanium dioxide for visible light photocatalysis, the creation of a biohybrid material through the encapsulation of proteins enables us to take advantage of the natural functionality of such protein to further improve on the photocatalytic efficiency of titanium dioxide. By harnessing naturally occurring proteins from our environment and sol-gel chemistry, we are capable of fabricating novel materials in order to address the mounting need for clean, renewable fuel production.

9:40 AM

(ICACC-HON-066-2018) Hydrogen absorption properties of amorphous Al(Si)CN derived from Al carbodiimde-based polymeric precursors (Invited)

Y. Iwamoto*[1]; K. Mizutani[1]; S. Tada[1]; S. Bernard[2]; E. Ionescu[3]; G. Mera[3]; Y. Daiko[1]; S. Honda[1]; R. Riedel[3]

1. Nagoya Institute of Technology, Japan
2. CNRS, Research Institute on Ceramics (IRCER), European Ceramic Center, France
3. Technische Universität Darmstadt, Germany

Recently, various AlN nanostructures have been successfully synthesized, and a great deal studies on their properties have been perfomed both experimentally and theoretically. In contrast to bulk AlN having four-fold coordination, Al atoms on the surface of the AlN nanostructures have three-fold coordination. The unsaturated sites on the Al are expected to provide the desirable adsorption sites for H_2 molecules. Such Al sites may be created in-situ within the polymer-derived ternary or quaternary amorphous Al(Si)CN. In this study, $AlCl_3$ was reacted with bis(trimethylsilyl)carbodiimide to afford poly(Al-cabodiime). The synthesized polymer was converted to X-ray amorphous Al(Si)CN by pyrolyzed at 873 K in Ar followed by an addtional heat treatment at 773 to 873 K in 10%H_2/Ar. H_2 adsorption properties of the synthesized Al(Si)CN was investigated by heat treatment under H_2 flow at 823 K followed by measuring the TPD profile in Ar. The TPD measurement resulted in the detection of a broad TCD signal at around 623 to 823 K. The desorbed gas was identified as H_2 by the simultaneous MS analysis. Further study on the detemination of activation energy for the H_2 desorption, and the local structure analysis are under investigation, and the results will be shown and discussed from a viewpoit to develop novel H_2 strage materials through the polymer-derived ceramics (PDCs) route.

10:20 AM

(ICACC-HON-067-2018) Additive Manufacturing of Ceramics for Structural and Functional Applications (Invited)

J. Liang[1]; A. M. Peterson[2]; R. D. Sisson*[1]

1. Worcester Polytechnic Institute, Materials Science and Engineering, USA
2. Worcester Polytechnic Institute, Chemical Engineering, USA

Additive manufacturing processes for polymers and metals are becoming well established for many applications. Additive manufacturing of ceramics for structural and functional applications appears to be lagging behind in process development and acceptance. In this presentation, the currently available processes for ceramics will be presented and discussed in terms of capabilities and limitations. In addition, the results of our preliminary work on 3D printing of polymer - ceramic blends, the post processing debinding heat treatment and the resulting microstructures and properties will be presented and discussed.

10:40 AM

(ICACC-HON-068-2018) Additive Manufacturing of Medical Devices (Invited)

R. Narayan*[1]

1. NC State University, USA

Additive manufacturing, which also known as 3D printing and rapid prototyping, is an technology that involves processing of three-dimensional materials and/or multicomponent systems using a layer-by-layer approach. Several additive manufacturing methods have been developed over the past three decades that involve processing of filamentous, powder, or liquid precursors. In recent years, additive manufacturing methods have been used to create medically-relevant structures that are difficult to create using conventional methods. The additive manufacturing method known as two-photon polymerization involves excitation of photoinitiator molecules to initiate reactions between photoinitiator molecules and monomers, leading to photopolymerization of a photosensitive material. Two-photon absorption demonstrates a nonlinear relationship with the incident light intensity. Polymerization occurs at sites where energies exceed the excitation threshold of the photoinitiator. The nonlinearity of two-photon absorption enables solidification of material and formation of structures well below the diffraction limit. Two-photon polymerization was used to prepare medical devices with small-scale features out of polymers and organically-modified ceramic materials. This talk will examine the use of two-photon polymerization of medically-relevant structures, including drug delivery devices, sensors, and tissue engineerind scaffolds, with small-scale features.

11:00 AM

(ICACC-HON-069-2018) Engineered strategy for pore configurations in cellular ceramics

M. Fukushima*[1]; H. Hyuga[1]; T. Ohji[1]; Y. Yoshizawa[1]

1. National Institute of Advanced Industrial Science and Technology (AIST), Japan

Various pore configurations have been widely found in nature, because structures of animals and plants can permit efficiency in mechanical properties - porosities. Fabricated porous ceramics have great advantages with high temperature stability and corrosion resistance. When numerous engineering applications are considered, high porosity should be desired, in terms of insulation, filtration, fluid permeability, absorption, reaction and weight. Although there is still big issue to achieve the optimized balance between porosity and properties, tailored pore configurations can have some possibilities to overcome the problem. In this presentation, we will review our recent progress on advanced processing strategies to control pore configurations at different length and volume scales. The proposed versatile approaches can be used to tailor pore configurations, and fabricate macroporous monoliths with various distinct characteristics.

11:20 AM

(ICACC-HON-070-2018) New development of ceramics process by fusion of nanosecond technology and nanotechnology

T. Nakayama*[1]; K. Niihara[1]; H. Suematsu[1]; T. Suzuki[1]

1. Nagaoka Univ of Tech, Japan

The thermal management (thermal discharge) in compact devices has become an urgent issue as these devices become increasingly more compact. To overcome this problem, research is being conducted in materials that promote heat radiation. Consequently, manufacturers need to find inorganic substances that discharge heat to serve as next-generation heat-dissipating materials. Inorganic materials such as aluminum nitride and boron nitride have higher heat conductions than aluminum and copper and they also have the advantage of being electrical insulators". However, these inorganic materials are extremely brittle, which causes reliability problems when they are used in portable devices. Because of this, attention is currently focused on heat-dissipating materials that are composites of organic and inorganic materials (thermal interface materials; TIMs), since they provide a way to overcome the problem of reliability. These inorganic heat-dissipating materials are used as fillers and are dispersed within polymers. They are both pliant and maintain the thermal conducting and electrical insulating properties of the material. We have proposed a new method for realizing orientation that employs a nanosecond pulse power supply as a new way to achieve anisotropic structure control. This method permits characteristic structure control that cannot be realized using other orientation methods.

11:40 AM

(ICACC-HON-071-2018) Development of Ultra Low Density Refractory Granules(ULDRG) for Kilns (Invited)

L. K. Sharma*[1]; D. P. Karmakar[1]

1. CSIR-Central Glass & Ceramic Research institute, India

Kiln is a basic need of ceramic industry which has 30-60% of the product cost through thermal energy depending on the type of the product. There are so many parameters like Low Thermal Mass Kiln Car Furniture, Low Thermal Mass Kiln Car Base, Flue Gas Analysis for Efficient Fuel Burning, Preventing Heat Losses through kiln Walls, Optimum Wares loading Density on Kiln Cars, Reducing Kiln Cars pushing time & Optimization of Firing Cycles etc for energy saving. Research was carried out on development of Ultra Low Density Refractory Granules to reduce the dead mass of the Kiln Cars Base. Refractory granules of bulk density less than 0.80 gm/cc with apparent porosity in the range of 60-70% were developed by using Indian Raw Materials by extrusion process. Granules have whiteness of approx 90%, life estimate of 8-10 years, Loose Packing Density (LPD) of 1.0 gm/cc, Packing Density(PD) of 1.20 gm/cc. Service temperature of this ULDRG is 1250 degree C. Thermal Conductivity was 0.20-0.25 W/mk at 1000 deg C. This ULDRG is very much Eco friendly and does not have and health or safety issues like ceramic fiber. 2000 kilograms ULDRG was produced and used in regular kiln cars of the Tunnel & shuttle kilns as a part of Industrial research. It contributed the mass reduction of 30-40% with fuel saving of 8-15%.

FS2: Tomography and Microscopy based Modeling of Ceramics

Influence of Inhomogeneity on Physical Properties

Room: Coquina Salon H

Session Chairs: Ryo Inoue, Tokyo University of Science; You Zhou, National Institute of Advanced Industrial Science and Technology (AIST)

8:30 AM

(ICACC-FS2-010-2018) Micro CT structure analysis and process design of organic and inorganic hybrid materials (Invited)

T. Nakayama*[1]; H. Cho[2]; S. Tanaka[1]; T. Fujihara[3]; H. Triet[1]; S. T. Nguyen[1]; H. Suematsu[1]; T. Suzuki[1]; K. Niihara[1]

1. Nagaoka Univ of Tech, Japan
2. Hanyang University, Republic of Korea
3. National Institute of Technology, Anan College, Japan

The controlled assembly of micro- and nano-ceramic fillers in polymer nanocomposites provides robust properties such as wetting, adhesion, thermal conductivity, electrical insulation and optical activity, and enable the extended application of these hybrid materials as thermal interfacing materials in microelectronics and for energy conversion. However, the required properties can only be obtained either by homogeneous mixing or by anisotropic orientation of a large amount (>50 vol.%) of expensive fillers, which is economically inefficient. Here we propose a strategy for tuning the orientation and assembly of ceramic boron nitride nanofillers in a polymer nanocomposite using a small amount (<5 vol.%) of filler to enhance thermal conduction. The texture of the BN fillers is tuned by application of a nanosecond pulse electric field and a superconductor magnetic field (10 T); the three-dimensional structure of the products was analyzed using micro X-ray CT scanning. The enhanced anisotropic orientation and thermal properties of the products were assessed as a function of the structural variation of the boron nitride fillers in the polymer.

9:00 AM

(ICACC-FS2-011-2018) X-ray μ-tomography: Actual and new developments for the characterization of the radiative properties of cellular ceramics up to very high temperatures

B. Rousseau*[1]

1. Laboratoire Thermique et Energies de Nantes, France

Knowledge of thermal radiative properties (TRPs) is mandatory for engineering systems and processes where the energy contribution of thermal radiation is important. A first group of TRPs can be directly measured (reflectance, transmittance, emittance) trough experimental facilities until homogenous conditions of heating are ensured. Another second group (absorption and scattering coefficient, scattering phase function) is needed to solve the Radiative Transfer Equation (RTE) that is unavoidable to solve the generalized heat equation. Properties of the second group can be deduced from the first one by applying inverse methods. Nevertheless exact direct 3D modelling for solving RTE are uncommon making that geometrical constraints are often operated to study the materials. For cellular ceramics, the probed volumes can be below those required to get a Representative Elementary Volumes for the estimation of TRPs. The introduction of X-ray μ-tomography over the past 12 years has opened new doors (i) to compute all the TRPs at room temperature as well as high temperatures (ii) to understand the main length scales which govern the TRPs on real samples (iii) to validate material generation algorithms. Key results will be recalled and new challenges will be given especially up to very high temperatures.

9:20 AM

(ICACC-FS2-012-2018) A New Differential Thresholding-Based Binarization Approach for the Tomographic Characterization of Cellular Ceramics (Invited)

U. Betke*[1]; S. Dalicho[1]; S. Rannabauer[1]; A. Lieb[1]; M. Scheffler[1]

1. OvGU Magdeburg, Nachwuchsforschergruppe NEOTHERM, Germany

The manufacturing of cellular ceramics can be performed by the sponge replication technique established by Schwartzwalder and Somers in 1961. This process makes use of ceramic slurries, which are coated onto a polymer foam template; after sintering of the ceramic phase a replica of the template is the result. For the control of the ceramic foam characteristics the rheological properties of the ceramic slurries used are of immediate importance. In this study, 45 ppi alumina foams were prepared by a standardized Schwartzwalder technique using slurries of varying compostion. The yield stress and high-shear viscosity of the slurries were determined as a function of the slurry composition. The ceramic foams were characterized by computed tomography (CT) and morphometric analysis of the binarized reconstruction volume. The main scope of the work involved the development of a procedure to reliably define the binarization threshold during these morphometric calculations. This process is based on the analysis of the differential course of the total porosity results from calculations performed at varying binarization thresholds (differential thresholding). A very good match of the obtained CT porosity results with the experimental data was achieved. The CT evaluation results were finally correlated to the rheological properties of the slurries used in foam manufacturing.

9:50 AM

(ICACC-FS2-013-2018) Simulation and Permeability and tortuosity of ceramics foams based on μCT

T. Fey*[1]

1. Friedrich-Alexander University Erlangen-Nürnberg, Department Material Science and Engineering, Germany

Cellular materials offer a wide spectrum of applications such as catalyst support structures, lightweight materials, energy adsorption, energy storage materials and filters. For filtration purpose an open-cell structure is needed. The cell window size is of particular interest for permeability of liquids and gases. Also cell interconnectivity and resulting pore percolation network is strongly affected by the cell window size. Due to the fact that experimental permeability measurements are limited in pressure and temperature range as well as type of gas or liquid (e.g. molten metal), simulation of permeability is in particular focus of interest. Based on micro computertomography (μCT) data the pore network is extracted for permeability simulations and tortuosity calculations. Darcian (linear) and Forchheimer (non-linear) permeability can be calculated from the simulated pressure drop in a defined volume of a cellular ceramic foam. A certain pore path within this volume for e.g. minimum pressure drop can be expressed by calculation of tortuosity.

10:10 AM

(ICACC-FS2-014-2018) Microstructure characterisation and FEM-simulation of porous ceramic structures

T. Fey*[1]

1. Friedrich-Alexander University Erlangen-Nürnberg, Department Material Science and Engineering, Germany

Porous ceramic foams are used in a wide field of applications as catalyst support structures, lightweight materials, filtration, or energy storage materials. Beside this porous ceramic scaffold for bone replacement are a promising material in future to ensure a faster medical healing. All applications in common is the need to know which pore morphology, surface, pore connectivity and tortuosity can be achieved with the given microstructure. Beside light microscopy approaches in the past on several cross cut sections nowadays

the use of microcomputer tomography (μCT) gives a full 3D-Model. In this paper we show the way from CT-scanning to simulation of structural and physical properties based on CT-data. Therefore different porous ceramic foams and periodic cellular structures and composites are analysed by μCT-measurements providing 3D-volume data for microstructural evaluation. Starting from reconstructed 2D-slices structural parameter can be calculated. Especially pore connectivity, pore network structure with their branch nodes and tortuosity are calculated from the pore volume data after thresholding. The segmented data is transferred into a FEM-mesh suitable for certain physical properties calculations. After verification of the simulation by experimental measured data new findings e.g. on localized properties can be determined to improve the physical properties of porous ceramic.

FS3: Chemical Processing of Functional Materials: Understanding the Conversion of Molecular Structures to Solid-State Compounds

Nanostructured Materials

Room: Coquina Salon A

Session Chairs: Emanuel Ionescu, Technical University Darmstadt; Gunnar Westin, Uppsala University

8:30 AM

(ICACC-FS3-019-2018) Polymer-derived ceramic nanocomposites for electrochemical energy storage (Invited)

G. Singh*[1]

1. Kansas State University, Mechanical and Nuclear Engineering Dept., USA

Molecular precursor derived ceramics (PDCs) have garnered intense research interest as potential standalone as well as composite electrode materials for rechargeable alkali metal-ion batteries and supercapacitors. PDC based electrodes offer high surface area, improved electrical conductivity and mechanical strength along with added value of mass production. Here, we will present data on recent success in synthesis of composites molecular precursor-derived silicon oxycarbide (SiOC), and chemically modified graphenes. We will show that interfacing PDCs with graphene derivatives is an effective strategy in improving PDC's Li-ion electrochemical capacity, first cycle efficiency, and long- term cyclability. Flexible, lightweight, and mechanically robust nanostructured electrodes deliver Li- capacity of approximately 550 mAh/g (total electrode weight) with nearly 100 % coulombic efficiency for over 1000 cycles. In addition, we will discuss the role of thermal annealing on electrical conductivity and capacitance effect in SiOC electrodes.

9:00 AM

(ICACC-FS3-020-2018) Disclosing the role of mixed Si-O-C units on the high Li-reversible capacity of silicon oxycarbide glasses

G. Soraru*[1]; M. Graczyk-Zajac[2]; D. Vrankovic[2]; V. Pradeep[2]; P. Waleska[2]; C. Hess[2]; H. Kleebe[2]

1. University of Trento, Industrial Engineering, Italy
2. Technical University Darmstadt, Germany

Graphite-based anode materials for Li-ion batteries provide stability and safety but the capacity does not exceed the theoretical value of 372 mAh g^{-1}. Silicon oxycarbide glasses, SiOCs, are emerging as the promising Li-ion storage host. SiOC glasses recover capacities up to 900 mAh×g^{-1} and maintain a good stability even when charged/discharged at very high currents in line with high stability over prolonged cycling. The reason for high capacities/stabilities and the precise description of the Li-ion storage sites are still a matter of debate. Many hypothesis have been proposed: Li intercalation between the sp^2 C layers or at the edges and in micropores of neighboring graphene layers or directly in the Si-O-C glass phase, and, in particular, in the mixed silicon oxycarbide units. In this paper we synthesized, starting from the same pre-ceramic polymer, two silicon oxycarbide glasses with similar microstructure and chemical composition. However, in one case all the mixed Si-O-C units have been converted into SiO_4 units. The comparison of the electrochemical behavior of these two glasses unveil the dominant role of mixed Si-O-C units on the Li-storage capacity of SiOCs.

9:20 AM

(ICACC-FS3-021-2018) Atomic/Molecular Layer Deposition of Inorganic-Organic Carboxylate Network Thin Films for the Application in Microelectronics

J. P. Penttinen*[1]; M. Nisula[1]; M. Karppinen[1]

1. Aalto University, Department of Chemistry and Materials Science, Finland

We introduce novel layer-by-layer deposition processes for the fabrication of crystalline inorganic-organic coordination network thin films by combined atomic/molecular layer deposition (ALD/MLD) technique with s-block elements and different aromatic polycarboxylates. The deposition processes for Li, Na, K, Mg, Ca, Sr, and Ba based carboxylate thin films fulfill the basic principles of ALD type growth including the sequential self-saturated surface reactions. Furthermore, we investigated the stability of the films in heat and humidity treatments. Some of the coordination network thin films reversibly absorb water molecules forming well-defined crystalline water-derivative phases. In our ALD/MLD processes we employed metal-thd complexes as metal precursors and different carboxylic acids, e.g., terephthalic acid, 3,5-pyridinedicarboxylic acid, as the organic precursors. Crystalline coordination network structures have large internal areas that can be used for various surface reactions, and they also have a stable porosity capable of storing guest species of various sizes. Our work thus underlines the strength of the ALD/MLD technique in discovering new exciting coordination network thin-film materials that may ultimately be potential material candidates for the next-generation application in, e.g., electronics, sensors, and other high-technology products.

9:40 AM

(ICACC-FS3-022-2018) Surface Modification of Oxide Nanomaterials and Application of Modified Nanomaterials to Polymer-Based Hybrids (Invited)

Y. Sugahara*[1]

1. Kagami Memorial Research Institute for Materials Science and Technology, Waseda University, Department of Applied Chemistry, School of Advanced Science and Engineering, Japan

Oxide nanomaterials have been attracting increasing attention, and the surface modification is one of the key techniques for oxide nanomaterials. Although silylation with silane coupling agents is the most common technique for surface modification of oxide nanomaterials, surface modification with phosphorus coupling agents, such as organophosphonic acids and phosphate esters, is also very suitable in particular for transition metal oxide nanomaterials. Their advantages over silane coupling agents include suppression of homocondensation under mild conditions and stability of metal-oxygen-phosphorous bonds formed. The surface modification with alcohols is also attractive because of the variation in their structures. We have developed surface modification of oxide nanomaterials, in particular nanoparticles and nanosheets, mainly using phosphorus coupling agents and alcohols. Titania nanoparticles and nanosheets prepared from potassium hexaniobate, $K_4Nb_6O_{17}$, and ion-exchangeable layered perovskite, $HLaNb_2O_7$, were mainly employed. One of their promising applications is their use as nanofillers of polymer-based hybrids, since surface-modified oxide nanomaterials are suitable to achieve high dispersibility in polymer matrices. Properties of polymers, for example optical and mechanical properties, were improved by incorporation of surface-modified oxide nanomaterials.

10:10 AM

(ICACC-FS3-023-2018) From colloidal solutions to superconducting nanocomposite epitaxial ceramic layers of HTSC-YBCO (Invited)

S. Ricart*[1]

1. Institut de Ciencia de Materials de Barcelona (CSIC), Spain

Achieving high current superconducting wires for large scale applications and magnets has been one of the most challenging objectives which has delivered to the so called, coated conductors. In the particular case of ceramic ReBCO-HTSC (High Temperature Superconductors) the challenge was to enhance vortex pinning at high temperatures and high magnetic fields through the generation of artificial pinning centers in the layer. For this purpose two low cost chemical solution deposition processes based on spontaneous segregation of second non-superconducting nano-phases during thermal processing of metalorganic precursors and preparation of colloidal solutions of preformed nanoparticles adapted to form stable solutions with Y, Ba and Cu salts. This talk will present different approaches to YBCO nanocomposites using different standard and colloidal precursor solutions containing different amounts of salt precursors or nanoparticles of metal oxides (Ferrites, CeO2, ZrO2, BaZrO3, BaHfO3). In both cases high performance epitaxial nanocomposite layers upon a CSD growth process are obtained. Compatibility of these low cost methodologies to scalable ink jet printing deposition of thick epitaxial nanocomposite layers and development of. Low-cost nanocomposite CSD crystallization through a transient-liquid assisted growth (TLAG) process will be discussed.

Precursor Chemistry

Room: Coquina Salon A

Session Chair: Thomas Fischer, University of Cologne

10:40 AM

(ICACC-FS3-024-2018) A new chemical approach to transform natural wood into bone scaffolds with superior mechanic and biologic performance

S. Sprio*[1]; A. Ruffini[1]; A. Ballardini[1]; M. Montesi[1]; S. Panseri[1]; A. Tampieri[1]

1. National Research Council of Italy, Institute of Science and Technology for Ceramics, Italy

The fabrication of solid functional ceramics demands the application of sintering processes, incompatible with the maintenance of metastable crystalline states or non-stoichiometric compositions. A relevant example is given by nanocrystalline, ion-doped apatite, widely recognized as elective material to produce scaffolds for bone regeneration. The prompt conversion of biomimetic apatites into poorly bioactive phases under sintering substantially limits the development of highly bioactive and mechanically-competent 3-D bone substitutes. Here we present a novel chemical approach to develop bone scaffold characterized by biomimetic composition and structure. Natural wood with bone-mimicking hierarchical architecture is exposed to designed conditions of temperature, atmosphere and gas pressures to activate heterogeneous chemical reactions and induce controlled compositional changes whereas maintaining the original structure at the multi-scale. The final product is a large (3 cm) nanocrystalline multi-doped apatitic scaffold inducing drastic overexpression of several genes active in bone regeneration, in bioreactor, and with hierarchical pore architecture at the base of high strength and bone-mimicking mechanical performance. Such results, so far prevented in large 3-D ceramic scaffolds, open to frontier applications in the regeneration of large load-bearing bones.

11:00 AM

(ICACC-FS3-025-2018) SiAlON coating by laser chemical vapor deposition using polymeric and metal-organic precursors

H. Katsui*[1]; T. Nakano[1]; T. Goto[1]

1. Institute for Materials Research, Tohoku University, Japan

SiAlON is well known as a host material for phosphors and has been extensively studied as high-temperature engineering ceramics. Because of its excellent chemical stability and corrosion resistance at high temperatures, SiAlON can be a bond coat of environmental barrier coatings for ceramic matrix composites. To date, few studies on the growth of crystalline SiAlON by vapor phase deposition techniques were reported in the literature. Chemical vapor deposition (CVD) is a versatile technique to synthesize films and coatings of high-temperature well-crystalized ceramic materials such as silicon carbide and alumina, although industrially common precursors are halides corrosive and difficult to be handled. In this study, we demonstrated the growth of crystalline SiAlON by a laser CVD technique using polymeric and metal-organic precursors, and effects of deposition conditions on the phase compositions, microstructure and deposition rates were investigated. Crystalline phases of SiAlON formed in the deposition temperature range of 1100–1400 K at growth rates greater than 1 mm/h. The α′-SiAlON film prepared at 1195 K exhibited dense microstructure with polygonally faceted grains in which nano-size precipitates with high Al compositions were presented.

11:20 AM

(ICACC-FS3-026-2018) Flexible organic-inorganic hybrid aerogels and xerogels from organoalkoxysilane precursors (Invited)

K. Kanamori*[1]

1. Kyoto University, Department of Chemistry, Graduate School of Science, Japan

Polyorganosiloxanes ($R_nSiO_{(4-n)/2}$), or silicones, derived from organo-substituted alkoxysilanes are an important class of organic-inorganic hybrid materials. A careful design of hydrolysis and polycondensation chemistry of these alkoxysilanes, which include organotrialkoxysilanes ($R_nSi(OR')_{4-n}$), organo-bridged alkoxysilanes (such as $(R'O)_3Si–R–Si(OR')_3$ and $(R'O)_2(CH_3)Si–R–Si(CH_3)(OR')_2$) or mixtures of these, leads to a homogeneous network that can be extended to materials formations in various microstructures and shape formats. In recent years, we have developed the chemical design of these systems, and found that resultant wet gels can be transformed into aerogels via supercritical drying, and moreover, into aerogel-like xerogels via ambient pressure drying due to improvements of mechanical strength and flexibility. In our primal case, a polymethylsilsesquioxane (PMSQ, $CH_3SiO_{3/2}$) gel has been developed through a well-controlled sol-gel process to yield a highly porous, transparent xerogel, which would offer a cost-effective process for massive industrialization of aerogel(xerogel)-based thermal superinsulators. Several other polyorganosiloxane aerogels and xerogels have been developed to further improve the mechanical properties, which also will be discussed in the talk.

11:50 AM

(ICACC-FS3-027-2018) One-pot synthesis of a C/SiFeN(O)-based ceramic paper with in-situ generated hierarchical micro/nano-morphology (Invited)

E. Ionescu*[1]; H. Kleebe[1]; R. Riedel[1]

1. Technical University Darmstadt, Materials and Earth Sciences, Germany

In the present study, a C/SiFeN(O)-based ceramic paper with in situ generated hierarchical micro/nano-morphology was prepared upon thermal treatment of a cellulose-based paper surface-modified with a polymeric single-source precursor prepared from perhydropolysilazane (PHPS) and iron(II) acetylacetonate ($Fe(acac)_2$). The ammonolysis at 1000 °C of the paper/precursor hybrid materials

leads to a C/SiFeN(O)-based ceramic paper which exhibits the same morphology as that of the cellulose paper. Subsequent annealing of the ceramic paper in nitrogen atmosphere at temperatures from 1200 to 1400 °C results in the in-situ generation of ultra-long silicon nitride nanowires with aspect ratios in the range of 10^3 on the surface and in the macropores of the ceramic paper. The nanowires exhibit round Fe_3Si tips at the end, indicating that the growth occurred via iron-catalyzed VLS (vapor-liquid-solid) mechanism. The combination of single-source precursor procedure, porous template and in situ VLS growth of 1D nanostructures provides a convenient one-pot synthesis approach to produce ceramic nanocomposites with hierarchical morphologies.

12:10 PM

(ICACC-FS3-028-2018) Synthesis, chemical and structural characterization of polymer drived SiAlCN ceramics

C. Salameh*[1]; C. Gervais[2]; F. Babonneau[2]; P. Miele[1]

1. Ecole Nationale Supérieure de Chimie de Montpellier, France
2. LCMCP, Université Pierre et Marie Curie, France

The Polymer-Derived Ceramics route represents a chemical route in which the chemistry, the processing properties and the reactivity of related polymers can efficiently be controlled and tailored to supply, after pyrolysis, non-oxide ceramics with the desired structure, phase distribution and homogeneity. The single-source precursor approach in PDCs is very interesting due to the ability to control the composition of polymers and derived ceramics at the atomic scale, introduce functional groups that are beneficial for the shaping ansd pyrolysis of the polymer and determine the functionality of the derived ceramics. SiCHCH2, SiH and NH groups offer the opportunity to attach supplementary elements to the polymer structure. Aluminum can be introduced by hydroalumination of olefinic groups, using alane adducts. Reactions may involve dehydrocoupling with formation of Si-N units. The SiAlCN systems represent an amorphous structure made of covalent bonds stable at high temperatures. Individual steps of the procedure to prepare polymer-derived SiAlCN ceramics were investigated. In particular, we describe the synthesis of a series of poly(carbo)silazanes modified by Al. The as-obtained polymers were chemically and structurally characterized then pyrolyzed to deliver amorphous materials. A systematic mechanistic study of the polymer-to-ceramic conversion is provided using solid-state NMR spectroscopy

12:30 PM

(ICACC-FS3-029-2018) Synthesis and comparison of electrochemical properties of lithium organic thin films

J. Heiska*[1]; M. Nisula[1]; M. Karppinen[1]

1. Aalto University, Chemistry and Materials Science, Finland

Organic electrode materials are an interesting alternative for the traditional inorganic Li-ion battery electrode materials. They often possess a high gravimetric capacity and fast reaction kinetics. The structural diversity of the organic materials makes it possible to tune the electrochemical properties by adding an electron donating or withdrawing functional group or a heteroatom to the organic skeleton. In the actual battery applications, the organic materials suffer from a low intrinsic conductivity; however, when the active material is deposited as a thin film the electrode does not require any additives to be functional. In this work, terephthalic acid and its four derivatives are evaluated as organic components for the Li-organic thin-film electrode materials. For the deposition of these ultrathin hybrid films the state-of-the-art gas-phase atomic/molecular layer deposition technique is employed. We will show that the chemical, structural and electrochemical characteristics of the films are largely controlled by the choice of the organic component. In particular, the electrochemical properties and most importantly the lithiation voltage depend on the functional group of the organic molecule, i.e. amino or bromo group decrease or increase the redox voltage, respectively. In addition, the lithiation mechanism is drastically affected by the asymmetry caused by the functional groups.

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