The American Ceramic Society

41st International Conference & Exposition on Advanced Ceramics and Composites

ABSTRACT BOOK

January 22–27, 2017
Daytona Beach, Florida

The American Ceramic Society
www.ceramics.org

Introduction

This volume contains abstracts for more than 970 presentations during the 41st International Conference & Exposition on Advanced Ceramics & Composites in Daytona Beach, Florida. The abstracts are reproduced as submitted by authors, a format that provides for longer, more detailed descriptions of papers. The American Ceramic Society accepts no responsibility for the content or quality of the abstract content. Abstracts are arranged by day, then by symposium and session title. An Author Index appears at the back of this book. The Meeting Guide contains locations of sessions with times, titles and authors of papers, but not presentation abstracts.

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.

Copyright © 2017 The American Ceramic Society (www.ceramics.org). All rights reserved.

MEETING REGULATIONS

The American Ceramic Society is a nonprofit scientific organization that facilitates the exchange of knowledge meetings and publication of papers for future reference. The Society owns and retains full right to control its publications and its meetings. The Society has an obligation to protect its members and meetings from intrusion by others who may wish to use the meetings for their own private promotion purpose. Literature found not to be in agreement with the Society's goals, in competition with Society services or of an offensive nature will not be displayed anywhere in the vicinity of the meeting. Promotional literature of any kind may not be displayed without the Society's permission and unless the Society provides tables for this purpose. Literature not conforming to this policy or displayed in other than designated areas will be disposed. The Society will not permit unauthorized scheduling of activities during its meeting by any person or group when those activities are conducted at its meeting place in interference with its programs and scheduled activities. The Society does not object to appropriate activities by others during its meetings if it is consulted with regard to time, place, and suitability. Any person or group wishing to conduct any activity at the time and location of the Society meeting must obtain permission from the Executive Director or Director of Meetings, giving full details regarding desired time, place and nature of activity.

Diversity Statement: The American Ceramic Society values diverse and inclusive participation within the field of ceramic science and engineering. ACerS strives to promote involvement and access to leadership opportunity regardless of race, ethnicity, gender, religion, age, sexual orientation, nationality, disability, appearance, geographic location, career path or academic level.

The American Ceramic Society plans to take photographs and video at the conference and reproduce them in educational, news or promotional materials, whether in print, electronic or other media, including The American Ceramic Society's website. By participating in the conference, you grant The American Ceramic Society the right to use your name and photograph for such purposes. All postings become the property of The American Ceramic Society.

During oral sessions conducted during Society meetings, **unauthorized photography, videotaping and audio recording is prohibited.** Failure to comply may result in the removal of the offender from the session or from the remainder of the meeting.

Registration Requirements: Attendance at any meeting of the Society shall be limited to duly registered persons.

Disclaimer: Statements of fact and opinion are the responsibility of the authors alone and do not imply an opinion on the part of the officers, staff or members of The American Ceramic Society. The American Ceramic Society assumes no responsibility for the statements and opinions advanced by the contributors to its publications or by the speakers at its programs; nor does The American Ceramic Society assume any liability for losses or injuries suffered by attendees at its meetings. Registered names and trademarks, etc. used in its publications, even without specific indications thereof, are not to be considered unprotected by the law. Mention of trade names of commercial products does not constitute endorsement or recommendations for use by the publishers, editors or authors.

Final determination of the suitability of any information, procedure or products for use contemplated by any user, and the manner of that use, is the sole responsibility of the user. Expert advice should be obtained at all times when implementation is being considered, particularly where hazardous materials or processes are encountered.

Copyright © 2017. The American Ceramic Society (www.ceramics.org). All rights reserved.

Table of Contents

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

S9: Porous Ceramics: Novel Developments and Applications

S10: Virtual Materials (Computational) Design and Ceramic Genome

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

S12: Materials for Extreme Environments: Ultrahigh Temperature Ceramics (UHTCs) and Nano-laminated Ternary Carbides and Nitrides (MAX Phases)

S14: Crystalline Materials for Electrical, Optical and Medical Applications

S15: Additive Manufacturing and 3-D Printing Technologies

3rd Pacific Rim Engineering Ceramics Summit

6th Global Young Investigator Forum

FS2: Advanced Ceramic Materials and Processing for Photonics and Energy

S1: Mechanical Behavior and Performance of Ceramics & Composites

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

S3: 14th International Symposium on Solid Oxide Fuel Cells (SOFC): Materials, Science and Technology

S4: Armor Ceramics

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

S7: 11th International Symposium on Nanostructured Materials: Functional Nanomaterials and Thin Films for Sustainable Energy Harvesting, Environmental and Health Applications

S9: Porous Ceramics: Novel Developments and Applications

S10: Virtual Materials (Computational) Design and Ceramic Genome

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

S12: Materials for Extreme Environments: Ultrahigh Temperature Ceramics (UHTCs) and Nano-laminated Ternary Carbides and Nitrides (MAX Phases)

S14: Crystalline Materials for Electrical, Optical and Medical Applications

S15: Additive Manufacturing and 3-D Printing Technologies

3rd Pacific Rim Engineering Ceramics Summit

6th Global Young Investigator Forum

FS2: Advanced Ceramic Materials and Processing for Photonics and Energy

S1: Mechanical Behavior and Performance of Ceramics & Composites

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

S3: 14th International Symposium on Solid Oxide Fuel Cells (SOFC): Materials, Science and Technology

S4: Armor Ceramics

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

S7: 11th International Symposium on Nanostructured Materials: Functional Nanomaterials and Thin Films for Sustainable Energy Harvesting, Environmental and Health Applications

S8: 11th International Symposium on Advanced Processing and Manufacturing Technologies for Structural and Multifunctional Materials and Systems (APMT11)

S9: Porous Ceramics: Novel Developments and Applications

S10: Virtual Materials (Computational) Design and Ceramic Genome

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

S12: Materials for Extreme Environments: Ultrahigh Temperature Ceramics (UHTCs) and Nano-laminated Ternary Carbides and Nitrides (MAX Phases)

S14: Crystalline Materials for Electrical, Optical and Medical Applications

3rd Pacific Rim Engineering Ceramics Summit

6th Global Young Investigator Forum

FS2: Advanced Ceramic Materials and Processing for Photonics and Energy

S1: Mechanical Behavior and Performance of Ceramics & Composites

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

S3: 14th International Symposium on Solid Oxide Fuel Cells (SOFC): Materials, Science and Technology

S4: Armor Ceramics

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

S7: 11th International Symposium on Nanostructured Materials: Functional Nanomaterials and Thin Films for Sustainable Energy Harvesting, Environmental and Health Applications

S8: 11th International Symposium on Advanced Processing and Manufacturing Technologies for Structural and Multifunctional Materials and Systems (APMT11)

S9: Porous Ceramics: Novel Developments and Applications

S10: Virtual Materials (Computational) Design and Ceramic Genome

S12: Materials for Extreme Environments: Ultrahigh Temperature Ceramics (UHTCs) and Nano-laminated Ternary Carbides and Nitrides (MAX Phases)

S7: 11th International Symposium on Nanostructured Materials: Functional Nanomaterials and Thin Films for Sustainable Energy Harvesting, Environmental and Health Applications

S8: 11th International Symposium on Advanced Processing and Manufacturing Technologies for Structural and Multifunctional Materials and Systems (APMT11)

S10: Virtual Materials (Computational) Design and Ceramic Genome

S12: Materials for Extreme Environments: Ultrahigh Temperature Ceramics (UHTCs) and Nano-laminated Ternary Carbides and Nitrides (MAX Phases)

S13: Advanced Materials for Sustainable Nuclear Fission and Fusion Energy

S14: Crystalline Materials for Electrical, Optical and Medical Applications

3rd Pacific Rim Engineering Ceramics Summit

FS1: Geopolymers, Chemically Bonded Ceramics, Eco-friendly and Sustainable Materials

FS2: Advanced Ceramic Materials and Processing for Photonics and Energy

S1: Mechanical Behavior and Performance of Ceramics & Composites

S3: 14th International Symposium on Solid Oxide Fuel Cells (SOFC): Materials, Science and Technology

S5: Next Generation Bioceramics and Biocomposites

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

S7: 11th International Symposium on Nanostructured Materials: Functional Nanomaterials and Thin Films for Sustainable Energy Harvesting, Environmental and Health Applications

S8: 11th International Symposium on Advanced Processing and Manufacturing Technologies for Structural and Multifunctional Materials and Systems (APMT11)

S10: Virtual Materials (Computational) Design and Ceramic Genome

S13: Advanced Materials for Sustainable Nuclear Fission and Fusion Energy

3rd Pacific Rim Engineering Ceramics Summit

FS1: Geopolymers, Chemically Bonded Ceramics, Eco-friendly and Sustainable Materials

S1: Mechanical Behavior and Performance of Ceramics & Composites

S3: 14th International Symposium on Solid Oxide Fuel Cells (SOFC): Materials, Science and Technology

S5: Next Generation Bioceramics and Biocomposites

S7: 11th International Symposium on Nanostructured Materials: Functional Nanomaterials and Thin Films for Sustainable Energy Harvesting, Environmental and Health Applications

S8: 11th International Symposium on Advanced Processing and Manufacturing Technologies for Structural and Multifunctional Materials and Systems (APMT11)

S13: Advanced Materials for Sustainable Nuclear Fission and Fusion Energy

FS1: Geopolymers, Chemically Bonded Ceramics, Eco-friendly and Sustainable Materials

S1: Mechanical Behavior and Performance of Ceramics & Composites

S3: 14th International Symposium on Solid Oxide Fuel Cells (SOFC): Materials, Science and Technology

S5: Next Generation Bioceramics and Biocomposites

S8: 11th International Symposium on Advanced Processing and Manufacturing Technologies for Structural and Multifunctional Materials and Systems (APMT11)

S13: Advanced Materials for Sustainable Nuclear Fission and Fusion Energy

Monday, January 23, 2017

Plenary Session

Room: Coquina Salon D
Session Chairs: Andrew Gyekenyesi, Ohio Aerospace Institute; Jingyang Wang, Shenyang National Laboratory for Materials Science, Institute of Metal Research

9:00 AM

(ICACC-PLEN-001-2017) Geopolymers: Structural Inorganic Polymers

W. M. Kriven*[1]; 1. University of Illinois at Urbana-Champaign, USA

Geopolymers are polysialates which are charge-balanced, aluminosilicate, ceramic-like materials made from an aluminosilicate source such as metakaolin, dehydrated clays, fly ash, slag, and alkali metasilicate “waterglass” solution. The stoichiometric chemical composition is $M_2O\bullet Al_2O_3\bullet 4SiO_2\bullet 11H_2O$ where M may be Group I elements of Li, Na, K, Rb or Cs, and the water content depends on particle size, specific surface area and aluminosilicate source. The inorganic polymer is made by high shear mixing with the liquid suspension undergoing dissolution, polycondensation or precipitation under ambient conditions. The resulting inorganic polymer has a microstructure which is impervious, nanoporous (diameter ~6.8 nm), of ~40 vol % nanoporosity, and is nanoparticulate (10-40 nm). The ceramic-like, cross-linked product shares the brittle nature of ceramics, but can be reinforced with particulates, platelets, chopped fibers, uniaxial fibers, or fiber weaves yielding a strong and tough composite, which is fire and acid corrosion resistant. Geopolymers also have refractory adhesive properties up to 1,000°C whereupon they crystallize into ceramics of tailorable, crystallographic thermal expansion. Under inert nitrogen or argon gases at 1400°C to 1600°C, geopolymers undergo carbothermal reduction or carbothermal nitridization forming soft agglomerates of SiC, Si_3N_4 or SiAlON nanoparticles. Geopolymers are a potential partial solution to global warming.

9:40 AM

(ICACC-PLEN-002-2017) Additive-free hot-pressed silicon carbide ceramics: A material with exceptional properties

P. Sajgalik*[1]; 1. Institute of Inorganic Chemistry, Slovak Academy of Sciences, Slovakia

Freeze-granulated and under infrared lamp annealed silicon carbide powder was densified to the full density without any sintering aids by hot-pressing/ultra-rapid hot-pressing at 1850°C. Presented silicon carbide hot-pressed ceramics has excellent properties. Vickers hardness is 29 GPa and indentation fracture toughness is 5.25 MPa.m$^{1/2}$. Samples densified by ultra-rapid hot-pressing have also full density and hardness of 27.4 GPa and fracture toughness of 5.3 MPa.m$^{1/2}$. Creep rate of ultra-rapid hot-pressed samples at 1450°C and 100 MPa load is 3.8 x 10^{-9} s^{-1} and at 1400°C and the same load conditions is 9.9 x 10^{-10} s^{-1}. Partial phase transformation β/α-SiC was observed in granulated and hot-pressed/ultra-rapid hot-pressed samples. Possible explanation of high density silicon carbide ceramics is based on formation of transient liquid phase. Transient liquid is possibly formed by aluminum, carbon and silica. Aluminum is present as impurity in starting powder. Increased concentration of aluminum can be explained by its segregation to triple-points at hot-pressing temperature. Carbon is present as residuum of organic agents used for granulation silicon carbide powder. Silica is present as main impurity of starting silicon carbide powder. Oxidation behaviour at 1350-1450°C/0-204h was investigated. This SiC ceramics has excellent oxidation resistance (4.91x10^{-5} mg^2/cm^{4}h at 1450°C).

10:40 AM

(ICACC-PLEN-003-2017) MLCC/Inductor trends and technological evolution

H. Kishi*[1]; 1. Taiyo Yuden Co., Ltd., Japan

In this talk, trends of devices, such as smartphone, will be shown in comparison with those in automotive industry. Through understanding such trends, the technological evolution of capacitor/inductor will be explained. For instance, energy density increase for further miniaturization in capacitor was realized by the material and process evolutions. Higher current capability in inductor was realized by the material change from ferrite to metal. The technological breakthroughs of material and process in capacitor and inductor were necessary to meet the change in device trends and requirements.

11:20 AM

(ICACC-PLEN-004-2017) Fifteen Years of Commercializing Ceramic Medical Devices Using Nanotechnology

T. J. Webster*[1]; 1. Northeastern University, USA

There is an acute shortage of organs due to disease, trauma, congenital defects, and most importantly, age related maladies. The synthetic materials used in tissue engineering applications today are typically composed of millimeter or micron sized particles and/or fiber dimensions. Although human cells are on the micron scale, their individual components, e.g. proteins, are composed of nanometer features. By modifying only the nanofeatures on material surfaces without changing surface chemistry, it is possible to increase tissue growth of any human tissue by controlling the endogenous adsorption of adhesive proteins onto the material surface. In addition, our group has shown that these same nanofeatures and nano-modifications can reduce bacterial growth without using antibiotics, which may further accelerate the growth of antibiotic resistant microbes. Inflammation can also be decreased through the use of nanomaterials. Finally, nanomedicine has been shown to stimulate the growth and differentiation of stem cells, which may someday be used to treat incurable disorders, such as neural damage. This strategy also accelerates FDA approval and commercialization efforts since new chemistries are not proposed, rather chemistries already approved by the FDA with altered nanoscale features.

3rd Pacific Rim Engineering Ceramics Summit

Current Trends and Future Directions I

Room: Coquina Salon C
Session Chairs: Young-Wook Kim, University of Seoul; Takashi Goto, IMR Tohoku University

1:30 PM

(ICACC-PACRIM-001-2017) Additive Manufacturing of Light Weight Composite Materials and Structures: Technical Challenges and Opportunities (Invited)

M. Singh*[1]; M. C. Halbig[2]; 1. Ohio Aerospace Institute, USA; 2. NASA Glenn Research Center, USA

Recently there has been tremendous interest in the additive manufacturing of light-weight materials and structures utilizing a wide variety of polymer and ceramic systems. The availability of affordable 3-D printers as well as open source printers and platforms have facilitated the large scale growth of distributed digital manufacturing of polymer composites utilizing commercially available filaments (ABS, PLA) and their variants. However, the effect of filament composition, reinforcements (chopped fibers and nanotubes), and 3-D printing variables on the microstructure and thermomechanical behavior is not well understood. On the other hand, there has been limited activity on additive manufacturing of ceramic matrix composites (CMCs). In this presentation, laminated object manufacturing (LOM), binder jet process, and 3-D printing approaches for developing ceramic composite materials are presented. The effect of different materials, reinforcements, and printing parameters on the microstructure and mechanical properties has been discussed in detail. Various technical challenges and opportunities for additive manufacturing of polymer and ceramic composites will be presented.

2:00 PM

(ICACC-PACRIM-002-2017) The Ways Travelled and the Ways to go in Sintering, the Key Technique of Ceramic Fabrication (Invited)

S. L. Kang*[1]; R. K. Bordia[2]; E. Olevsky[3]; 1. Korea Institute of Ceramic Engineering and Technology (KICET), Republic of Korea; 2. Clemson University, USA; 3. San Diego State University, USA

The majority of modern ceramic products are fabricated by sintering, probably the oldest human technology with the firing of pottery. One of the papers published in the first volume of the Journal of the American Ceramic Society in 1918 was on the sintering of MgO. This talk will be presented in commemoration of the centennial anniversary of the first non-clay sintering paper in the Journal. We will first briefly review the current understanding of sintering phenomena, densification, grain growth and microstructural evolution. An emphasis will be laid on microstructural evolution with respect to grain boundary structure. We will discuss the fundamentals of various sintering techniques with the application of pressure, microwave, electric and magnetic fields. Sintering-related modern techniques, such as 3-D printing and solid-state conversion of single crystals, will also be explained. We will also comment on the sintering of multi-phase and complex systems. Future directions of scientific and technical researches, including the developments of new sintering theories and techniques, will be suggested.

2:30 PM

(ICACC-PACRIM-003-2017) Laser oscillation of Lu_2O_3 transparent ceramics by spark plasma sintering (Invited)

T. Goto*[1]; L. An[1]; A. Ito[1]; 1. IMR Tohoku University, Japan

Transparent single crystals have wide-ranged applications such as scintillators and lasers. Polycrystalline ceramics can be more advantageous than single crystals because the sintering process is short-time and low-temperature with high mechanical strength and less segregation of dopants. Although conventional sintering process still needs a long time and a high temperature, spark plasma sintering (SPS) can consolidate many ceramics at a low temperature in a short time. In this study, SPS has been employed to prepare Lu-based oxide transparent ceramics and laser oscillation of the SPSed ceramics has been first demonstrated. Transparent Lu-based transparent ceramics were synthesized by using commercially available powders. In order to obtain transparent ceramics, voids should be eliminated (porosity < 0.05%) by optimizing a sintering profile (temperature, pressure and time). A two-step sintering profile and post-annealing were employed to prepare transparent Lu_2O_3 (1 mm in thickness, in-line transmittance 71%) with a grain size of 0.91 mm at 1723K, 100MPa for 2.7ks. Nd^{3+} 1 at% doped Lu_2O_3 ceramics by SPS has first exhibited laser oscillation. Transparent $Lu_3Al_5O_{12}$ ceramics (1 mm in thickness, in-line transmittance 78%) with a grain size of 0.33-0.52 mm were first prepared by SPS, and new transparent $Lu_2Ti_2O_7$, Lu_3NbO_7 and $Lu_2Hf_2O_7$ ceramics were first synthesized.

3:20 PM

(ICACC-PACRIM-004-2017) Thermal Shock Resistance, Wear Behavior and Oxidation Resistance of Silicon Nitride Based Nano-composites (Invited)

P. Sajgalik*[1]; M. Hnatko[1]; Z. Lences[1]; J. Dusza[2]; P. Tatarko[2]; A. Kovalcikova[2]; M. Kasiarova[2]; 1. Institute of Inorganic Chemistry, Slovak Academy of Sciences, Slovakia; 2. Institute of Materials Science, Slovak Academy of Sciences, Slovakia

Six different sintering aids (Lu_2O_3, Yb_2O_3, Y_2O_3, Sm_2O_3, Nd_2O_3 and La_2O_3) were used for the processing of dense Si_3N_4/SiC micro/nano composites. Thermal shock resistance: A critical temperature difference increased with an increasing ionic radius of RE^{3+} for both the composites and the monoliths. Wear behavior: The friction coefficient as well as the specific wear rate decreased with a decreasing ionic radius of rare-earth elements in both the monoliths and the composites. High bonding strength and the high fracture toughness are the reasons why the ceramics doped by Lu exhibited the best wear resistance. Oxidation resistance: Composites exhibited predominately parabolic oxidation behavior indicated diffusion as the rate limiting mechanism. Exception was only the Si_3N_4-SiC composite sintered with Lu_2O_3. In this case diffusion of cation has been strongly suppressed because of the beneficial effect of stable grain boundary phase and the presence of the SiC particles predominately located at the grain boundaries of Si_3N_4.

3:50 PM

(ICACC-PACRIM-005-2017) Advanced protective ceramic coating for accident tolerant zircaloy fuel cladding (Invited)

J. Wang*[1]; 1. Shenyang National Laboratory for Materials Science, Institute of Metal Research, China

Exploring advanced ceramic coating on zircaloy clad has been considered as a critical challenge for the development of accident-tolerant fuel/clad system. The key roles of protective coating are to impede the severe oxidation rate, degradation of the performances of zircaloy cladding, and hydrogen generation rate during loss-of-coolant accident (LOCA) scenario. Crucial requirements for the ceramic coatings include but not limited to: excellent irradiation damage resistance; good oxidation resistance in steam; matched mechanical and thermal properties, and chemical compatibility with zircaloy cladding; and retention of the stability of zircaloy cladding during the processing of dense coating. Transition metal carbides are promising protective coating for zircaloy cladding. I will firstly present the concept of optimal irradiation damage tolerance of ternary and binary transitional metal carbides (MAX phase and sub-stoichiometric ZrC_{1-x}) through theoretical modeling of intrinsic point defect sinks, namely high concentration of nano-twinned crystal structure and self-assembly of carbon vacancies, inside their crystal structures. Breakthrough is made on low temperature PVD processing of integrated M-Al-C (M is a transition metal element) coating with tunable composition. The mechanical integrity, thermochemical compatibility, and steam oxidation of the coating system are comprehensively reviewed.

4:20 PM

(ICACC-PACRIM-006-2017) Anisotropic properties of c-axis oriented SiC prepared by using strong magnetic field (Invited)

T. S. Suzuki*[1]; 1. National Institute for Materials Science, Japan

The physical and mechanical properties of ceramics can be tailored by controlling their microstructure. The controlled development of crystallographic orientation is one effective way to improve the properties of ceramics. We already reported that development of texture could be controlled by consolidation in a strong magnetic field followed by heating even for diamagnetic ceramics. The crystalline orientation in alpha-SiC could also be controlled by a strong magnetic field and the c-axis was aligned parallel to the direction of the magnetic field. Aqueous suspensions of pH 10 were prepared that contained 30 vol% solids. The suspensions were then consolidated in a strong magnetic field of 12 T at room temperature. The green SiC compacts were densified by hot pressing at 2273K for 2 h at a pressure of 40 MPa or spark plasma sintering at 2173K for 10min at a pressure of 85 MPa. The preparation of the dense and textured SiC polycrystal was achieved. The 3-point bending strengths of the samples sintered by hot pressing were 907 and 799 MPa for the crack-growth directions parallel and perpendicular to the c-axis in the c-axis oriented SiC, respectively, and the strength of the random SiC was 724 MPa. The thermal conductivity and the electrical conductivity perpendicular to the c-axis was higher than that parallel to the c-axis in the textured SiC.

4:50 PM

(ICACC-PACRIM-007-2017) Heat Resistant Liquid-Phase Sintered Silicon Carbide Ceramics with a Small Amount of Additives (Invited)

Y. Kim*[1]; Y. Seo[1]; 1. University of Seoul, Republic of Korea

Three different liquid-phase sintered SiC ceramics (LPS-SiC) with a small amount of additives were prepared by conventional hot-pressing: 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 0.5 wt% Y_2O_3 (SCY). Observation of the ceramics using high resolution transmission electron microscopy (HRTEM) showed the followings: the SCALu and SCY exhibited a lack of amorphous films in both SiC-SiC boundaries and junction areas; and the SCYSc exhibited both clean and crystallized SiC/SiC boundaries, as well as clean SiC/junction phase boundaries with a fully crystallized junction phase. The SCALu maintained 100% of its room temperature (RT) strength (630 MPa) up to 1600°C. The SCYSc maintained 93% of its RT strength (536 MPa) up to 1600°C, and showed rapid degradation at 1700°C and 1800°C. Degradation at temperatures above 1600°C was due to softening of the grain boundary phase, as evidenced by the nonlinear behavior of load-displacement curves. The SCY maintained 100% of its RT strength (550 MPa) up to 1600°C. The present results suggest that LPS-SiC can be more heat resistant than solid state sintered SiC ceramics by judicious selection of sintering additive composition and content.

6th Global Young Investigator Forum

Ceramics for Magnetic and Electric, Energy Conversion and Energy Storage

Room: Coquina Salon F
Session Chair: Eva Hemmer, University of Ottawa

1:30 PM

(ICACC-GYIF-001-2017) Multiscale Designing of Solar-Driven Photocatalysts (Invited)

G. Liu*[1]; 1. Institute of Metal Research, CAS, China

Solar-driven water splitting or reduction of CO_2 on semiconductor based photocatalysts represents a promising technique to convert solar energy into storable chemical energy. The central task in realizing the practical application of photocatalysis is to develop highly efficient solar-driven photocatalysts. The efficiency of photocatalysis is determined by the synergistic effects of solar light absorption, charge separation and catalytic process, which are three basic processes of photocatalysis. Concerning these three basic processes, we focus on three aspects to tailor the properties of photocatalysts. They are band engineering together with exploring unknown photocatalysts, heterostructuring, and facet controlling, which support the rational construction of high-efficiency photocatalysts in our studies. In this talk, the related processes will be introduced.

2:00 PM

(ICACC-GYIF-002-2017) Redox site visualization in plasmonic photocatalyst composed of TiO_2 and Au nanoparticles (Invited)

G. Kawamura*[1]; H. Muto[1]; A. Matsuda[1]; 1. Toyohashi University of Technology, Japan

Plasmonic photocatalyst, which was named in 2008, has recently been attracting much attention because of its high quantum efficiency of the photocatalyses and partially veiled reaction mechanisms. The probable mechanisms for plasmonic photocatalyses such as hot electron transfer, plasmonic energy transfer, and local field enhancement have been suggested by several researchers, whereas experimental proofs especially for hot electron transfer are still insufficient. In this work, one of the typical composites of plasmonic metal nanoparticles (NPs) and semiconductors to form plasmonic photocatalysts, Au NPs and TiO_2, was employed to study the carrier behavior during plasmonic photocatalyses. With this plasmonic photocatalyst, the carrier behavior becomes quite simple because plasmonic energy transfer and local field enhancement mechanisms can be excluded from consideration. This is because the energies of Au NP surface plasmon resonance and TiO_2 bandgap are not overlapped. A scanning transmission electron microscope (STEM) equipped with an energy-dispersive X-ray spectrometer (EDX) was mainly used to visualize the redox sites on plasmonic photocatalyst under independent irradiations of ultra-violet and visible light.

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

Room: Coquina Salon F
Session Chair: Derek King, UES, Inc.

3:30 PM

(ICACC-GYIF-004-2017) The versatile use of iron oxide nanoparticles in nanomedicine (Invited)

I. Gessner*[1]; S. Ilyas[1]; S. Siribbal[1]; E. Krakor[1]; M. Schütz[1]; T. Fischer[1]; S. Mathur[1]; 1. University of Cologne, Germany

Out of a vast variety of metal and metal oxide nanoparticles that have been developed for medicinal purposes, iron oxides are one of a few materials that made it through clinical trials. Due to their high biocompatibility, stability and the abundance of iron in our environment, which results in low costs of iron based materials, diverse iron oxide nanoparticles (IONPs) have been prepared. Especially the magnetic properties of maghemite and magnetite nanoparticles have aroused great interest as imaging agents for MRI and for magnetic targeting and separation processes. In our workgroup, IONPs including γ-Fe_2O_3, α-Fe_2O_3 and Fe_3O_4 have been synthesized using a broad range of well-established synthetic procedures. By changing the reaction conditions and applying suitable surface ligands, the morphology, surface charge and dispersibility of IONPs can be tuned according to the desired application. Besides the synthesis of novel MRI contrast agents using natural products, dual MRI/PET imaging agents have been prepared by attaching a radionuclide to the particle surface. Furthermore, by employing the highly selective click chemistry, magnetic separation of proteins was possible out of a proteome mixture. With regard to therapeutic purposes, IONPS have been used in our workgroup as drug delivery vehicles by either attaching a drug to the particle surface or loading hollow magnetic particles.

4:00 PM

(ICACC-GYIF-005-2017) Synthesis and Application of Nanoscale Graphene Oxide-Doped Gelatin Hydrogels as Scaffolds for Tissue Engineering and Drug Delivery

A. P. Sridhar*[1]; K. Sadhu[1]; C. Marmorat[1]; M. Simon[2]; M. H. Rafailovich[1];
1. Stony Brook University, USA; 2. Stony Brook University, USA

As the fields of tissue engineering and drug delivery move closer to clinical applications, challenges of engineering functional, biomimetic, and inexpensive scaffolds persist. The discovery of versatile nanoscale graphene oxide (nGO) has opened up avenues for engineering hydrogels with enhanced structural stability and physiological viability. This study engineered and characterized cross-linked, nGO-doped gelatin hydrogels through Rheology, FTIR Spectroscopy, Contact Angle, and TGA. The nGO-doped hydrogels exhibited a stiff, tunable structure with increased biocompatibility and water retention capacity. Then, nGO was observed to selectively suppress the growth of cancerous keratinocytes, and the nGO-doped hydrogels showed potential as drug delivery vehicles for selective, localized cancer therapy. Additionally, the gels supported adhesion and proliferation of normal keratinocytes and induced pluripotent stem cells, while preventing dermal fibroblasts from adhering. These findings indicated the gels' strong anti-fibrotic tissue engineering potential. Finally, the gels were applied as drug delivery vehicles and exhibited enhanced loading and sustained release of curcumin, a potent therapeutic. Ultimately, this study proposed using nGO-doped gelatin hydrogels as easy-to-synthesize, cost-effective vectors for novel scaffold-based disease therapy.

4:20 PM

(ICACC-GYIF-006-2017) Effect of strain rate on plastic deformation of hydroxyapatite

H. Hino*[1]; S. Kobayashi[1]; T. Furushima[1]; 1. Tokyo Metropolitan University, Japan

In the case of taking a medicine, an oral administration is a common way. A medicine which reaches affected area is limited and a side effect may be developed. In order to improve these defects, a medical device is set at affected area. In addition, the medical device is required to be bioabsorbable for reduction of the burden for the patient. In this study, plasticity and phase transformation of hydroxyapatite (HA) was examined to fabricate a bioabsorbable device. The HA compact was fired in air at temperature between 1200 and 1300°C for 2h and 5h at a uniaxial compressive pressure of 150 MPa and a cold isostatic pressure of 200 MPa. The relative density of HA compacts increased with increasing firing temperature and compressive pressure. The substantially dense HA specimen which has 96.6 % to 98.1 relative density was tested under compression at a temperature between 1100 and 1200°C using strain rate in range 1.67×10^{-3} to 1.67×10^{-5} s^{-1}. The compressive stress and strain behavior for HA was attributed to the fine grain size and test temperature. In addition, the HA specimen fabricated same firing route heat-treated at 1400°C in various time. The X-ray diffraction pattern was measured to evaluate phase transition to bioabsorbable tricalcium phosphate.

4:40 PM

(ICACC-GYIF-007-2017) Multifunctional HA-TNT-PDA-Ag nanocomposite scaffold for bone tissue engineering

S. S. Bhosle[1]; M. Taheri*[1]; T. Shokuhfar[1]; 1. University of Illinois at Chicago, USA

Poly methyl methacrylate (PMMA) based scaffold with enhanced mechanical properties, capable of silver release has been fabricated. Such scaffold can be used to promote antibacterial activity and stimulate new tissue formation. The scaffold is designed to incorporate nanomaterials such as Titanium Nanotubes(TNT) and hydroxyapatite(HA) nanoparticles which are able to enhance the mechanical properties and bioactivity of composite, respectively. Initially, a thin film of polydopamine (PDA) was coated on TNT which were synthesized by rapid breakdown anodization. Silver ions were then reduced on the TNT-PDA powder to provide antibacterial properties. Secondly, HA nanoparticles were prepared using sol-gel and freeze drying, mimicking the composition of native bone extracellular matrix. Finally, composite was produced by mixing HA-TNT-PDA-Ag with PMMA monomers. Sodium bicarbonate was then added to generate pores upon thermal treatment. The XRD, FESEM, and TEM were used to characterize the nanocomposite in terms of composition, morphology, and structures, respectively. The results demonstrate developed nanocomposite poses antibacterial properties along with 10-20% increase in hardness and flexural strength compared to the control. This study ascertains that proposed scaffold could be a promising candidate for enhanced osteointegration thus possessing the potential applications for bone tissue regeneration.

5:00 PM

(ICACC-GYIF-008-2017) Electrolytic Deposition of Calcium Phosphate on Metallic Substrates

M. K. Mahapatra[1]; N. Jindal*[1]; 1. University of Alabama at Birmingham, USA

Hydroxyapatite-coated implants have been shown to improve the long-term wear of orthopedic implants and minimize the need for revision surgeries for hip and knee replacement while reducing post-surgical pain. Currently, only 7% of surgeries use hydroxyapatite-coated implants, in large part due to high cost. A simple and low-cost electrochemical deposition of hydroxyapatite has the potential to decrease cost while allowing for greater uniformity and morphological control of the coating. SS 316L and titanium alloys are considered biocompatible for orthopedic implants. Functional ceramic coatings such as hydroxyapatite on the metallic implants introduce a bioactive surface and improve the wear resistance of the metallic implants. In this work, a calcium phosphate coating has been electrolytically deposited on SS 316 alloy and titanium substrates. The role of the electrolytic bath composition and applied current has been investigated on the coating chemistry, uniformity, morphology, and bonding with the substrate. The coatings have been analyzed using XRD, SEM, and SEM-EDS techniques. It has been observed that the evolution of calcium phosphate compounds can be tuned in-situ during electrodeposition as well as with a post-treatment of the coating in a NaOH solution. Post-treatment NaOH submersion has been shown to improve the bonding and promote phase change.

5:20 PM

(ICACC-GYIF-009-2017) Development of a new biocompatible bismuth-free Aurivillius-like layered ferroelectric material $BaIn_2Ta_2O_9$

Y. Fujimoto*[1]; E. Nakamachi[1]; Y. Morita[1]; 1. Doshisha University, Japan

A new biocompatible Aurivillius-like layered ferroelectric was designed by first-principles analysis and its thin film was fabricated by using RF magnetron sputtering, which is utilized for sensors of Bio-MEMS devices. Commonly used Aurivillius layered ferroelectrics contain the bio-toxic elements, bismuth. Therefore, we designed and fabricated a new biocompatible Aurivillius-like ferroelectrics as follows: 1) selection of candidate biocompatible elements, 2) molecular structure design AM2B2O9 by using the first- principles calculation, and 3) thin film generation of the predicted material by using RF magnetron sputtering. We carried out the first-principles calculations of 37 candidates; structure optimization, band gap for insulation, soft mode for ferroelectric phase transition, and estimation of piezoelectric properties. Finally, BaIn2Ta2O9 was selected, which has the spontaneous polarization, 7.95 nC/cm2. Through the experimental design procedure using L27 orthogonal array, we determined the sputtering conditions. After post- annealing, polarization-electric field hysteresis loop was observed. This results show the possibility of the Aurivillius-like ferroelectric material, BaIn2Ta2O9.

FS2: Advanced Ceramic Materials and Processing for Photonics and Energy

Photovoltaics and Solar Fuels

Room: Halifax A/B
Session Chairs: Francesco Enrichi, Centro Studi e Ricerche E. Fermi (Italy) and Luleå University of Technology (Sweden); Xuhui Sun, Soochow University

1:30 PM

(ICACC-FS2- 001- 2017) Efficient and Stable Organic-inorganic Hybrid Solar Cells Using Anodic Nanostructures (Invited)

O. K. Varghese*[1]; M. Paulose[1]; A. Torabi[1]; P. Kaur[1]; 1. University of Houston, USA

Although solar cells based on organic materials and organic-inorganic heterojunctions have been well-demonstrated, these technologies have not yet reached the market place primarily because of the issues associated with efficiency or stability or both arising from light induced or environment dependent physical/chemical properties of the organic or organometallic light absorber materials. Recent studies indicated that the efficiency as well as stability in these hybrid solar cells could be enhanced dramatically by using inorganic nanostructures. For example, titania nanotube array electron transport layers fabricated using anodic oxidation have recently shown remarkable potential in improving the stability of organometal halide perovskite solar cells. The nanotube architecture could also enhance the efficiency of low cost organic materials with poor carrier diffusion length. The presentation will provide an overview of oxide nanostructures grown by anodic oxidation. The focus of the presentation will be on the roles of these structures in enhancing the performance and stability of hybrid solar cells consisting of organic as well as organometal halide perovskite absorbers.

2:00 PM

(ICACC-FS2- 002- 2017) Transition Metal Based Nanostructures for Efficient Solar Water Splitting (Invited)

J. Deng[1]; H. Zhang[1]; K. Nie[1]; J. Zhong[1]; X. J. Sun*[1]; 1. Soochow University, China

The solar-driven water splitting process is highly attractive for alternative energy utilization, while developing efficient, earth-abundant, bifunctional catalysts for both oxygen evolution reaction (OER) and hydrogen evolution reaction (HER) has remained as a major challenge. Pt and noble metal oxides (e.g. IrO_2, RuO_2) are regarded as pioneering HER and OER catalysts, respectively, and can be processed in a large window of solution pHs with high efficiencies, while the capability of large-scale application is prohibited by the lack of supply and high cost of these materials. Alternatively, the transition metal oxides, sulfides and nitrides are currently intensively investigated to improve the intrinsic activity as potential earth-abundant material candidates for water splitting catalysts. In our recent work, enormous efforts have been focused on improving the performance of transition metal based nanostructure photoelectrode such as hematite, Co based nanostructures, etc. Different methods such as morphology and composition control, elemental doping, and improvement of the charge transport of these photoelectrodes have been developed to improve their performance in solar water splitting.

2:30 PM

(ICACC-FS2- 003- 2017) Advanced materials for energy application through solution synthesis (Invited)

G. Westin*[1]; 1. Uppsala University, Sweden

The development and utilisation of catalysts for solar fuels, solar-cells and photo-assisted water and air cleaning, as well as high performance batteries and capacitors require robust synthesis routes capable of reproducibly producing advanced complex materials at low cost and high rate. These multi-functional devices should be effective in absorbing photons for generation of holes and electrons, charge transport and catalysis of surface reactions and be stable. Here is discussed solution based processing routes using either metal-organic reactive alkoxide precursors or complexed inorganic salts to achieve complex thin- and ultra-thin films on various flat and complex nano-structured substrates. Efficient processing of multi-metallic metal particles in multi-metallic oxide matrixes will be described. The materials prepared were characterized with a large range of analytical techniques including; TEM, SEM, XRD, XPS, IR- and Raman spectroscopy, TG and DSC, and their structures will be related to their properties.

3:20 PM

(ICACC-FS2- 004- 2017) Assessing morphology and interfacial diffusion of MoO_3 in organic solar cells using x-ray and neutron scattering (Invited)

J. F. Martinez Hardigree*[1]; M. Riede[1]; I. Ramirez[1]; G. Mazzotta[1]; A. Morel[1]; P. Gutfreund[2]; D. Wermeille[3]; 1. University of Oxford, United Kingdom; 2. Institut Laue-Langevin, France; 3. ESRF, France

Molybdenum trioxide (MoO_3) has found increased use as an inexpensive, thermally-evaporated hole transporting (HTL) layer in vacuum processed small molecule organic photovoltaics (OPV). Despite the relatively thin (~2 nm) layers required for efficient device performance, recent studies have identified MoO_3 diffusion and concomitant electronic doping of the overlying organic layers as a key contributor to performance degradation in thin film OPV. In this study we examine MoO_3/organic bilayers with polycrystalline α-sexithiophene and amorphous TAPC organic donor layers, using in-situ time-of-flight neutron reflectometry to monitor the evolution of the inorganic/organic interface as the films are heated from room temperature past their glass-transition (T_g) and melting (T_m) temperatures, respectively. We compare these to ex-situ measurements of similar films obtained with grazing-incidence wide-angle x-ray scattering (GIWAXS), and assess the relative performance and fabrication tradeoffs of MoO_3 relative to solution-processed organic HTLs.

3:50 PM

(ICACC-FS2- 005- 2017) Rare earth ions and Ag nanoaggregates in silica-hafnia sol gel films as efficient down-converters for silicon solar cells (Invited)

F. Enrichi*[1]; C. Armellini[2]; S. Belmokhtar[3]; A. Bouajaj[3]; E. Cattaruzza[4]; M. Ferrari[2]; F. Gonella[4]; M. Mardegan[4]; G. Righini[5]; L. Zur[5]; 1. Centro Studi e Ricerche E. Fermi (Italy) and Luleå University of Technology (Sweden), Italy; 2. CNR-IFN, Istituto di Fotonica e Nanotecnologie, CSMFO Lab. & FBK-CMM, Italy; 3. Laboratoire des Technologies Innovantes, LTI, Département de Génie industriel ENSA. Université Abdelmalek Essaâdi, Morocco; 4. Dipartimento di Scienze Molecolari e Nanosistemi, Università Ca' Foscari Venezia, Italy; 5. Centro Studi e Ricerche Enrico Fermi, Italy

Energy harvesting in solar cells may be improved by spectral conversion layers, to convert poorly absorbed frequencies of the solar spectrum into the region of maximum absorption of the cell. The frequency conversion properties of rare earth ions may be enhanced by the presence of metal nanoparticles. As an example, here we report the study of efficient down-converting silica-hafnia waveguides doped with 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 nanoparticles. The preparation of 70 SiO_2 – 30 HfO_2 layers was carried out by sol-gel route, followed by Ag ion-exchange in a molten salt bath. The films 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.

FS3: Carbon Nanostructures and 2-D Materials and Composites

Focued Session 3: Carbon Nanostructures and 2-D Materials and Composites

Room: Coquina Salon A
Session Chair: Gustavo Costa, NASA Glenn Research Center

1:30 PM

(ICACC-FS3- 001- 2017) High Performance Energy Storage Carbons from Agricultural Byproducts (Invited)

D. Mitlin*[1]; 1. Clarkson U, USA

In agriculture and forestry very little is wasted, but many byproduct streams wind up having low economic value. While it is relatively straightforward to pyrolyze these organic materials, the obtained biochar or activated carbon possesses lowly value as well. In an ideal scenario such “waste” streams may be rather transformed into sought after products with a range of end uses, such as energy, water treatment, gas separation, etc. Here I will provide several examples on tailoring the synthesis methodology to take advantage of the bio precursors’ inherent properties to obtain advanced carbons with structure and chemistry optimized for electrochemical supercapacitor, lithium ion battery (LIB), sodium ion battery (NIB) and hybrid battery – capacitor applications. In all cases the performance of these inexpensive waste-derived materials is on par or even better than the much more costly “designer” systems such as graphene or carbon nanotubes.

2:00 PM

(ICACC-FS3- 002- 2017) Graphene/Sulfur for advance Li-S battery (Invited)

F. Li*[1]; R. Fang[1]; H. Cheng[1]; 1. Institute of Metal Research, Chinese Academy of Science, China

Lithium-sulfur (Li-S) batteries are currently being explored intensely due to their high theoretical specific energy density and low cost. However, the areal sulfur loading of electrodes is usually less than 2.0 mg cm^{-2}. Therefore, considering the practical reliability of Li-S batteries, it is of great importance to develop new materials and structures for the cathode that not only allows a high areal sulfur loading, but also enables high sulfur utilization and good cycling stability. Here, we report the facile synthesis of a highly porous graphene as the sulfur host, allowing a high sulfur content of 80 wt%, which further enables a high sulfur loading of 5 mg cm^{-2}. From the perspective of integrated structural design of the cathode, we further proposed an all-graphene configuration, in which highly conductive graphene with an almost perfect structure was employed as the current collector, and partially oxygenated graphene with a moderate number of oxygen-containing functional groups was used as a polysulfide-adsorption layer. This unique cathode structural design enables both high initial gravimetric specific capacity (1500 mAh g^{-1}) and areal specific capacity (7.5 mAh cm^{-2}), together with excellent cycling stability for 400 cycles, indicating great promise for more reliable lithium-sulfur batteries.

2:30 PM

(ICACC-FS3- 003- 2017) Solid-state growth of carbon-supported ceramic nanostructures for electrocatalysis and lithium batteries (Invited)

K. Xiao[1]; R. Amal[1]; D. Wang*[1]; 1. University of New South Wales, Australia

A simple and cost-effective route for the synthesis of metal nitride and carbide nanowires is reported. A metal-doped polymer was used as the sole precursor. The polymer supplies either nitrogen or carbon sources to forming nitride and carbide via a solid-state thermal carburization/nitriding treatment. This method is more convenient for the sake of the easy-to-prepare precursor and the one-step thermal annealing. The structures of the annealed products are temperature dependent. Combining XRD and XPS analysis, we found that the metal oxide precipitates at low temperature and converts to nitride at or carbides when temperature is raised up. This observation shows the temperature-modulated solid state conversion process. Both SEM and TEM revealed the nanostructures. The products showed improved activity for hydrogen evolution reaction with smaller overpotential comparing to the case without the nanostructured ceramic phases. It is also noticed that the ceramic components played a significant role in stabilizing the charge-discharge stability of lithium battery electrodes. The bifunctional capacity of this new carbon/ceramic material is promising for future new energy technologies.

3:20 PM

(ICACC-FS3- 004- 2017) Two-dimensional Structures of Tungsten Diselenide (WSe_2) by Liquid Exfoliation

D. Gerchman[1]; F. A. Berutti[1]; A. K. Alves*[1]; 1. UFRGS, Brazil

Previously the existence of two-dimensional crystalline films was considered impossible due to their instability caused by temperature and the decrease of the material’s melting point with the reduction of thickness. However, in 2004, Geim, Novoselov et al. produced graphene for the first time, a single layer of carbon atoms packed in a honeycomb lattice, introducing a new field of research of two-dimensional materials. Researches involving these materials have recently brought to attention transition metal dichalcogenides (TMDs), MX_2, where M is a metal (Ti, Nb, Ta, Mo, W) and X is a chalcogen (Se, S, Te). This family of materials has a layered structure that can be exfoliated and produce semiconductors in a nanometric scale. In this study the liquid exfoliation of WSe_2 was performed. This method was based on mixing WSe_2 with a solvent, ultrasonication to break the weak bonds between layers, centrifugation in order to separate the precipitate from the supernatant poorly exfoliated. Its was determined the most suitable solvent for exfoliation of this material (water, NMP or isopropanol) and the effect of an anionic surfactant. The time and the kind of the ultrasound applied were also studied. The exfoliated materials were characterized using UV-Vis spectroscopy, SEM, TEM, EDS, AFM in order to verify the effective presence of WSe_2 single-layers and the most suitable method to produce them.

3:40 PM

(ICACC-FS3- 005- 2017) Controlled vacancy formation in Mo_2C MXene

Q. Tao*[1]; M. Dahlqvist[1]; J. Lu[1]; S. Kota[2]; R. Meshkian[1]; J. Halim[1]; J. Palisaitis[1]; L. Hultman[1]; M. W. Barsoum[2]; P. Persson[1]; J. Rosen[1]; 1. Linköping University, Sweden; 2. Drexel University, USA

MXene (where M is a transition metal, X is C and/or N) is a comparatively young class of 2D materials, which stem from selective etching of the “A” element, typically Al, from the atomically laminated parent 3D MAX phase. The rich chemistries of MXenes (~20 to date), elevates their applicability. Mo_2C is a recent MXene for which the transport and energy storage properties have been investigated. It performs well when tested as an electrode material for Li-ions, especially at high rates. To date the primary property tuning potential is based on altering the M element or the MXene functionalization. Here we propose a new method for introducing vacancies in a Mo_2C, based on selective etching of the parent MAX phase with hydrofluoric acid (HF). The vacancy formation is imaged by atomically resolved scanning transmission electron microscopy (STEM). Comparing Mo_2C with, and without, vacancies, we find that vacancy formation leads to superior capacitances, as well as a decrease in the room temperature resistivity by four orders of magnitude - from 0.6 to $3.2x10^{-5}\Omega$.m. We further suggest that our method of introducing vacancies can be applied to other MXenes and represents an important tool to engineer and tailor both the MAX phases and their corresponding MXenes for numerous applications.

S1: Mechanical Behavior and Performance of Ceramics & Composites

Mechanics, Characterization and Joining

Room: Coquina Salon D
Session Chairs: Rabih Mansour, The University of Akron;
Marion Bartsch, DLR - German Aerospace Center

1:30 PM

(ICACC-S1-001-2017) Interlaminar Fracture Properties of 2D Woven CMC at Room and High Temperature (Invited)

R. Mansour*[1]; Y. P. Singh[1]; G. N. Morscher[1]; 1. The University of Akron, 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 develop an experimental setup that can be used at both room and high temperature. Hence, a non-visual crack monitoring technique has been successfully developed to estimate crack length in woven SiC/SiC composite using electrical resistance. This method was utilized to compare interlaminar fracture properties at room and high temperature for different CMC systems. Acoustic emission was used as well to determine the onset of crack initiation and damage accumulation during the test.

2:00 PM

(ICACC-S1-002-2017) Mechanical behavior of a wound all-oxide ceramic matrix composite

S. Hackemann*[1]; 1. DLR - German Aerospace Center, Germany

The winding technique of the investigated all-oxide CMC enables variable fiber architecture, but makes the experimental investigations of the mechanical behavior and modeling of the material rather complex. The transformation of elastic constants from unidirectional material to wound material with deviating fiber orientations cannot be performed straightforward, as the influences of shrinking cracks and the cross-over lines from the winding process have to be taken into account. Unidirectional material was tested to determine the failure envelope of the lamina and to discuss appropriate failure criteria with focus on failure mode based criteria. The choice of these criteria enables the determination of fiber dominated failure and matrix dominated failure but causes comparably high numerical effort. The experimental results of material with a 0|90° fiber orientation are the basis for the verification, but also revealed the necessity to modify the model derived from unidirectional data. A second material law has to be introduced for the cross-over lines. The cross-over lines and their vicinity show exceptionally high strains at failure in comparison to the layer-like material sections. Furthermore these lines are a main source of failure when fiber bundle dominated failure occurs. Experimental and numerical results will be presented.

2:20 PM

(ICACC-S1-003-2017) Generating 3-dimensional microstructure models of porous ceramic matrix composites by means of X-ray tomography and FIB-slicing

M. Bartsch*[1]; K. Artzt[1]; J. Wischek[1]; M. Eggeler[1]; P. Watermeyer[1]; K. Kelm[1]; A. Manero[2]; S. Raghavan[2]; P. Kenesei[3]; J. Okasinski[3]; J. Almer[3]; 1. DLR - German Aerospace Center, Germany; 2. University of Central Florida, USA; 3. Argonne National Laboratory, USA

Ceramic matrix composites (CMC) are developed for providing a high damage tolerance compared to monolithic ceramics. The mechanical behaviour of CMCs is defined by the properties of the constituents and the complex microstructure, which is controlled by the processing parameters. In this study the 3-dimensional microstructure of an all oxide porous CMC has been characterized on different length scales, utilizing non-destructive X-ray tomography in the range from mm to µm and combined sequential focused ion beam (FIB) ablation and scanning electron microscopy in the submicron range. The derived virtual microstructures were analysed, and data were used for generating numerical models for further mechanical or thermal analyses by finite element method. The CMC material was processed at DLR by winding a continuous alumina fibre bundle, which is infiltrated by aqueous alumina slurry, onto a mandrel and further sintering of the generated ceramic 'green body'. The result is a CMC with complex hierarchical microstructure. Fibre arrangement, fibre volume fraction, and matrix porosity can be varied in a wide range.

2:40 PM

(ICACC-S1-004-2017) In-situ analyses of thermal barrier coating's elastic and inelastic deformation via synchrotron X-rays

A. Manero*[1]; B. Sarley[1]; K. Knipe[1]; J. Wischek[2]; C. Meid[2]; J. Almer[3]; J. Okasinski[3]; A. M. Karlsson[4]; M. Bartsch[2]; S. Raghavan[1]; 1. University of Central Florida, USA; 2. DLR - German Aerospace Center, Germany; 3. Argonne National Lab, USA; 4. Cleveland State University, USA

In-situ synchrotron measurements were conducted on a thermal barrier coating system on a superalloy substrate. The use of high energy X-rays affords real-time monitoring of complex mechanical behavior; when coupled with an integrated high temperature and mechanical loading testing apparatus, variations in realistic loading representative for service conditions can be investigated for their influence on constituents of the multi-layer system. This provides a method for identifying how changes in loading conditions across a turbine blade may play a role in the initiation of failure mechanisms and lifetime expectancy of the coating. Collecting diffraction measurements on a 2D area detector, elastic strain analysis is conducted to elucidate the influence of external thermal loading, applied mechanical loading, and internal cooling on the thermally grown oxide and ceramic zirconia topcoat. Analysis has identified the influence of these loadings on the system. Investigating the texturing and preferred orientation of the diffraction rings, further insight on the influence of plastic strain is discussed including how grains' lattice plane rotation and subdivision during high temperature loading affect the residual strain state. Together the non-destructive methods of evaluating the multi-layer system shed light on the performance of thermal barrier coatings in operational conditions.

3:20 PM

(ICACC-S1-005-2017) Electrical Resistance as a NDE tool for damage detection and health monitoring in Ceramic Matrix Composites

Y. P. Singh*[1]; M. J. Presby[1]; G. N. Morscher[1]; 1. The University of Akron, USA

Electrical resistance (ER) is a useful non-destructive evaluation (NDE) technique for in-situ system health monitoring and quality control purposes. We utilize multidirectional measurement of ER using several contacts placed on the surface of ceramic matrix composite (CMC) materials. We perform measurements on materials with different relative electrical conductivity of fibers and the matrix. Also different fiber architectures e.g., woven fibers or laminate composites were used. We discuss the optimization of lead locations for better resolution in damage detection. Our results show that utilizing the ER method we can track the initiation and growth of transverse cracks developing in these materials under conditions of loading. We also show that utilizing a suitable lead placement on different positions at the surface of a specimen, the resistance can be measured along surface or through thickness in many directions. This gives the potential to determine cracks which exist internally or on the surface.

3:40 PM

(ICACC-S1-006-2017) Apparent Fracture Toughness in Brittle Materials under Static and Fatigue Loading

O. Kravchenko*[1]; S. Kravchenko[2]; C. Sun[2]; 1. Case Western Reserve University, USA; 2. Purdue University, USA

The apparent fracture toughness and fatigue crack propagation rate (FCPR) in brittle materials were found to depend on the local state of stress at the crack tip. Nonstandard fracture samples were used to conduct static fracture toughness and fatigue experiments on brittle materials. Unidirectional carbon fiber epoxy composite and polymethylmethacrylate (PMMA) were evaluated. The sample configuration was found to significantly affect the experimental measurements of the apparent fracture toughness and FCPR. Consequently, experimental results indicated that fracture behavior and FCPR could not be characterized by the critical value or by the amplitude of the stress intensity factor alone. A second parameter, the nonsingular opening stress component, was required. The magnitude and the sign of the nonsingular stress component were calculated from finite element analysis. The nonsingular stress influenced the size of K-dominance zone and depended on sample size and loading conditions. The new phenomenological two-parameter fracture mechanics model for apparent fracture toughness and FCPR prediction was developed. The proposed model incorporates the effect of size and geometry on the fracture behavior of brittle materials in static and fatigue conditions allowing accurate transferability of laboratory data to life prediction in engineering components.

4:00 PM

(ICACC-S1-057-2017) Potentials of Niobium Carbide (NbC) as cutting tools and for wear protection

M. Woydt*[1]; H. Mohrbacher[3]; S. Huang[2]; J. Vleugels[2]; 1. BAM Federal Institute for Materials Research and Testing, Germany; 2. Katholieke Universiteit Leuven, Belgium; 3. Niobelcon bvba, Belgium

The present paper illuminates the metallurgical progress on niobium carbide based hard metals since 2013, 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 developments 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 1000°C, dry sliding wear (T= 22/400°C; 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).

4:20 PM

(ICACC-S1-008-2017) Torsion shear strength of ceramics joined by brittle or ductile materials

L. Goglio*[1]; 1. Politecnico di Torino, Italy

Assessing the shear strength of a joined ceramic or CMC is an essential task in components design. Indeed, what is needed for design purposes, is the true shear strength, while several standard tests only give conventional values, disregarding multiaxiality and non-uniformity of the actual stress distribution. If used under proper conditions, torsion test has the advantage of producing a state of pure shear stress with a known distribution in the joint section. A distinction must be done whether the joining material is brittle or ductile. In case of brittle behavior, the stress concentration factor due to the specimen geometry must be taken into account; moreover, since the stress state is intrinsically biaxial, failure may occur out of the joint section. In case of ductile behavior, the non-linear effect of plasticity must be included. The talk discusses these aspects and proposes a procedure suitable to assess the pure shear strength of joined ceramics.

4:40 PM

(ICACC-S1-009-2017) Shear tests on joined materials

M. Ferraris*[1]; L. Goglio[2]; M. Salvo[1]; F. Smeacetto[1]; S. De La Pierre Des Ambrois[1]; V. Casalegno[1]; 1. Politecnico di Torino, Italy; 2. Politecnico di Torino, 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 glass-ceramics or by adhesives. The hourglass shape must be designed to minimize stress concentration and have shear stress only in the joined area. When the joining material is purely brittle (glass-ceramics), the shear strength is evaluated from the maximum of the torque/rotation curve. In the case of non purely brittle joining materials (adhesives) 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 the failure mode and therefore the meaning of the measured strength.

5:00 PM

(ICACC-S1-010-2017) Mechanical properties of ternary eutectic ceramic from room to very high temperature

S. Gourdin*[1]; L. Marcin[1]; M. Podgorski[1]; L. Carroz[2]; 1. Safran Tech - Safran Group, France; 2. RSA le Rubis SA, France

Current challenges in CO_2 and NO_x emissions reduction push aircraft engine manufacturers to innovate technologically while increasing the efficiency of their engines (stress and temperature). Therefore, current materials such nickel based superalloys can not be used anymore and new materials are thus considered. For the hottest parts of jet engines, eutectic ceramics have potentially interesting features. In order to determine the mechanical properties of ternary eutectic ceramic, tensile and 4-point bending tests were carried out from room temperature up to 1500°C. To perform experiments at such high temperatures, a specific device has been developed. The first results show a slight reduction of the material strength when the temperature rises and an expected difference, about a factor two, between the two types of tests. This clearly shed light on the importance of taking into account the effective volume via probabilistic approach in order to design part. In addition, numerical multi-scale analyses have also been performed to assess the thermo-mechanical properties of the material.

5:20 PM

(ICACC-S1-011-2017) Evaluation of Flash Sinter-Bonding method for producing ceramic-metal composites

C. Madec*[1]; B. Frédéric[2]; S. Le Gallet[2]; E. Petitpas[1]; B. Bettencourt[1]; B. Salesse[1]; F. Barthelemy[3]; 1. NEXTER Systems, France; 2. University de Bourgogne Franche-Comté, France; 3. DGA, France

Spark Plasma Sintering is an attractive method which was developed recently, to sinter various types of materials with a shorter sintering time compared to other processes. This technique enables the association of materials with different properties (electrical, thermal…), to get for example composites. In this study, the goal is to associate a ceramic and a metal, the physical and mechanical properties of which are completely different. The starting powders are alumina for the ceramic and titanium for the metal. The powder of alumina was densified by SPS. The resulting disks display a density upper than 99% and a grain size less than 1 μm. With this technique, high heating speed can be achieved, limiting grain growth and resulting in a fine microstructure. When the metal powder is co-sintered with the ceramic to obtain a 1,5 mm total thickness of densified metal, the sample is cracked in the ceramic. Consequently, a compromise has to be found in order to get a good cohesion between the metal and the ceramic while avoiding the ceramic cracking. The proposed

solution consisted in adding an interlayer, with one or more layers between Al_2O_3 and Ti. Interlayers are composed by mixes of Al_2O_3 and Ti. The sintering behavior of these mixtures has been studied. Several thickness and compositions have been tested to highlight the influence of adhesion and damage of the different parts.

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

Thermal Barrier Coatings : Processing and Characterization

Room: St. John
Session Chairs: Douglas Wolfe, Pennsylvania State University; Bryan Harder, NASA Glenn Research Center

1:30 PM

(ICACC-S2-001-2017) Orientation dependent mechanical and thermal properties of spray deposited ceramics

G. Smith*[1]; G. Dwivedi[1]; S. Sampath[1]; 1. Stony Brook University, USA

Thermal spray deposited materials are generally anisotropic, associated with the droplet based assemblage of thin and thick coatings. Although most coatings are considered in-plane orthotropic, significant differences in properties are possible between in-plane and through thickness directions Due to thickness limitations and constraints associated with their bonding to the substrates, it is difficult to rigorously characterize the mechanical and thermal properties of these coatings even in one orientation, let alone two or three. In this paper, we report mechanical (modulus and toughness) and thermal properties of free standing plasma sprayed yttria-stabilized zirconia deposits produced to thicknesses of 25 mm which allows testing of these complex materials in two orientations in near bulk form. Two variants of microstructures were examined, notionally high and low porosity, which can impact the nature of the anisotropy. Fracture toughness and energy release rates were obtained through single-edge notched beam three-point bend technique, while thermal properties were obtained from laser flash techniques. Samples were examined both in as-deposited and sintered forms. The results point to some 15-20% differences in orientation dependent properties. The implications of these findings on both processing and performance will be discussed.

1:50 PM

(ICACC-S2-002-2017) Microstructural Effects on Erosion Durability in Thermal Barrier Coatings

M. P. Schmitt*[1]; A. K. Rai[2]; J. M. Schreiber[1]; T. J. Eden[3]; D. E. Wolfe[1]; 1. The Pennsylvania State University, USA; 2. UES, Inc., USA; 3. The Applied Research Laboratory at The Pennsylvania State University, USA

The rare earth zirconate pyrochlores ($RE_2Zr_2O_7$) present an alternative to standard 7YSZ due to their higher phase stability limits (GZO: >1500°C, YSZ: ~1200°C) and lower thermal conductivities. However, pyrochlore zirconates exhibit relatively low toughness values compared to 7YSZ, which results in poor erosion durability and therefore reduces their performance benefits. Previous work has revealed the ability of air plasma spray (APS) composites to enhance the erosion performance of pyrochlore analyte containing coatings. This work investigates tailored design architectures to maximize the erosion durability by modifying the microstructural alignment and aspect ratio/splat morphology. The coatings were tested in terms of their erosion thermal conductivity and compared to standard 7YSZ and $Gd_2Zr_2O_7$ coatings. Modeling of the room temperature impact behavior was performed on the APS coatings to further understand and validate the hypotheses. This modeling framework is then extended to electron beam – physical vapor deposition (EB-PVD) and plasma spray – physical vapor deposition (PS-PVD) coatings in an effort to explore impact behavior in columnar coatings and explain the enhanced erosion durability of PS-PVD structures.

2:10 PM

(ICACC-S2-003-2017) Characterization of the Deposition Formation Dynamics of Suspension Plasma Spray Coatings using In-Situ and Ex-Situ Curvature Measurements

R. Chidambaram Seshadri*[1]; G. Smith[1]; V. Viswanathan[1]; G. Dwivedi[1]; S. Sampath[1]; 1. Stony Brook University, USA

Suspension plasma spraying (SPS) enables a variety of microstructures with unique mechanical and thermal properties. Considering complex deposition dynamics, there is a need to better understand the relationship between spray conditions, particle behaviour, stress evolution and resultant properties. In this investigation nano yttria-stabilized zirconia (YSZ) particles suspended in ethanol were deposited using a single electrode cascaded-arc plasma torch. The stresses generated during the deposition of the layers were monitored via in-situ curvature measurements. Depending on the deposition conditions, coating microstructures ranged from feathery porous to dense and cracked deposits. Post-deposition bi-layer curvature measurement was carried out using exsitu curvature measurement to quantify the thermo-elastic response of the coatings during low temperature thermal cycling. This study involving insitu diagnostics and exsitu characterization along with process mapping concepts provides a framework for coating formation mechanisms, process parametrics and microstructure description for this emerging technology.

2:30 PM

(ICACC-S2-004-2017) Novel GAP/GZO Composites With High Temperature Phase Stability and Improved Durability

D. E. Wolfe*[1]; 1. Pennsylvania State University, USA

In order to increase efficiency of gas turbine engines, new materials or systems of materials must be developed which can operate at higher temperatures than current state-of-the-art 7YSZ thermal barrier coatings. GZO was initially identified as a replacement candidate material due to its high temperature phase stability (>1500°C for pyrochlore phase), low thermal conductivity, and low sintering rates. However, the poor erosion performance of GZO severely inhibits its durability and overall reliability. Gadolinium aluminum perovskite (GAP) was identified as a new candidate material for use as a toughening phase in GZO composite coatings. GAP exhibits a relatively high toughness, is phase stable up to its melting temperature (2030°C), and is phase stable with GZO. Pellets were fabricated of monolithic GZO and GAP as well as GZO composites containing 10%, 30%, and 50% (wt.%) GAP. Heat treatments at 1400°C and characterization via XRD, SEM, and EDS were performed to confirm phase stability of the high temperature capable composites. Indentation and erosion were used to confirm the enhanced mechanical properties and erosion response with results showing significant improvement, offering the first system with high temperature stability and a robust mechanical response.

3:10 PM

(ICACC-S2-005-2017) Thermal and Mechanical Properties of ZrO_2-Y_2O_3 Thermal Barrier Coatings by Thermal Exposure (Invited)

B. Jang*[1]; K. Yasuda[2]; S. Kim[3]; Y. Oh[3]; H. Kim[3]; 1. National Institute for Materials Science (NIMS), Japan; 2. Tokyo Institute of Technology, Japan; 3. Korea Institute of Ceramic Engineering and Technology, Republic of Korea

Thermal barrier coatings (TBCs) have received a large attention because they increase the thermal efficiency of gas turbine engines by increasing the gas turbine inlet temperature and reducing the amount of cooling air required for the hot section components. To optimize TBCs for integration into gas turbines, characterization of the relationship between microstructure and thermal-mechanical

properties of the coatings is necessary. The purpose of this work is to investigate the influence of the microstructure as well as porosity on mechanical properties of ZrO_2-4mol%Y_2O_3 (YSZ) coatings deposited by air plasma spray (APS) or EB-PVD (electron beam-physical vapor deposition). The mechanical properties of plasma sprayed YSZ coatings were evaluated by three-point bending method. The bending strength, Young's modulus and residual stress depend on microstructure as well as coating distance. TGO (thermally grown oxide) at interface of top and bond coat was generated after thermally cyclic test at 1100°C. The hot corrosion between YSZ coatings and volcanic ash was examined by isothermal heating at 1200°C in air between 10 min and 100hrs. The thickness of corrosive region at top surface of TBCs by the reaction between YSZ coating and volcanic ash was increased with increasing the oxidation time.

3:40 PM

(ICACC-S2-006-2017) Thick, Durable, Low Thermal Conductivity Thermal Barrier YAG Coatings Using The Solution Precursor Plasma Spray Process

M. Gell*[1]; E. H. Jordan[2]; B. Nair[3]; R. Kumar[1]; C. Jiang[3]; 1. University of Connecticut, USA; 2. Uniiversity of Connecticut, USA; 3. HiFundaLLC, USA

Low thermal conductivity is one of the most critical properties for a thermal barrier coating (TBC) used in gas turbine engines. There are a number of approaches for gaining low thermal conductivity, including (a) selecting a composition that inherently has strong phonon scattering, (b) engineering the microstructure to introduce defects that contribute to phonon scattering, and (c) using thicker TBCs. In this work, we employ all three strategies. We have selected (a) the yttrium aluminum garnet (YAG) composition, which has lower elevated temperature thermal conductivity than YSZ, (b) introduced planar arrays of porosity, called inter-pass boundaries (IPBs), into the microstructure, and (c) we have evaluated SPPS YAG TBC coating thicknesses of 250, 500 and 750 microns. This presentation will present the microstructure and thermal cycle data for these SPPS YAG TBCs.

4:00 PM

(ICACC-S2-007-2017) Characteristics of Oxides in ZrO_2-La_2O_3-Gd_2O_3 Systems for TBC Applications (Invited)

S. Kim*[1]; S. Lee[1]; Y. Oh[1]; S. Lee[1]; H. Kim[1]; B. Jang[2]; 1. Korea Institute of Ceramic Engineering and Technology (KICET), Republic of Korea; 2. National Institute for Materials Science (NIMS), Japan

Among candidate materials for replacing YSZ in TBC applications, rare-earth zirconate oxides with fluorite and/or pyrochlore structure have been prevailing ones. The low thermal conductivities of rare-earth zirconate oxides with these structures are attributed to the phonon scattering by point defects in the crystallographic structures. In addition, these oxides exhibit little phase transformation upon heating up to melting temperature, which is one of indispensable characteristics for future TBC materials. In this study, characteristics of selected oxide compositions in ZrO_2-La_2O_3-Gd_2O_3 systems, are investigated. Phase formation, microstructures, and thermal conductivities are examined with the sintered samples as well as coatings fabricated by suspension plasma spray. The possibilities of these oxides for TBC application are also discussed.

4:30 PM

(ICACC-S2-008-2017) Measurement and Understanding of the Distribution of Residual Stress in APS TBC as a Function of Depth

C. Li*[1]; R. Cernik[1]; P. Xiao[1]; Y. Chen[1]; X. Zhang[1]; S. Jaques[1]; M. di Michiel[2]; J. Behnsen[1]; 1. University of Manchester, United Kingdom; 2. ESRF, France

The residual stress distribution in Air Plasma Sprayed (APS) Thermal Barrier Coatings (TBC) is considered to be the driving force of the failure of the coating. We have measured the residual stress in APS TBC as a function of depth by using high energy synchrotron XRD in transmission geometry. We corroborate the observed stress distribution in the 3D microstructure and 2D microstructure of the TBC by X-Ray Computed Tomography (CT) and SEM. We have also used image based modelling to explain the trend measured by XRD. We found that the stress distribution in the TBC was compressive varying from the surface (~ 20MPa) to the interface (~ 500MPa) in a non-linear trend. The distribution was observed to be different from the one predicted by the analytical and numerical model where an almost a linear trend was predicted. We found that the complex microstrcuture and the cracks generated from heat treatment of the coating were the reasons for the non-linear trend of the residual stress distributed in TBC.

4:50 PM

(ICACC-S2-009-2017) Thermal Property Measurement for Thermal Barrier Coatings by Pulsed Thermal Imaging

J. Sun*[1]; 1. Argonne National Lab, USA

Thermal barrier coatings (TBCs) are extensively used on hot gas-path components in gas turbines to improve engine performance and extend component life. For these applications, the most important TBC parameters include thermal conductivity and thickness. Nondestructive evaluation (NDE) methods have been developed to measure some TBC parameters; they however generally have limitations and impractical to examine TBCs on engine components. In this presentation, we will describe a recently developed NDE method, the pulsed thermal imaging – multilayer analysis (PTI-MLA), which can evaluate essentially any TBC samples with one or more coating layers and determine TBC property distributions over the entire TBC surface. The measurement uncertainty for typical TBCs was found to be down to 2-3 percent when using a state-of-the-art infrared camera. However, the high cost and large size of such cameras may limit the wider utilization of this technology. Recently a commercial low-cost, small-size, room-temperature infrared camera was evaluated and found to be suitable for such application. This development may therefore significantly reduce the cost of the thermal imaging NDE technology and expand its use in industrial applications. This presentation reports these recent developments and experimental results.

S3: 14th International Symposium on Solid Oxide Fuel Cells (SOFC): Materials, Science and Technology

SOFC/SOEC: Overview of Activities

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

1:30 PM

(ICACC-S3-001-2017) Overview of NETL's SOFC Program and Accomplishments (Invited)

S. Markovich*[1]; 1. DOE - National Energy Technology Laboratory, USA

The U.S. Department of Energy's (DOE) Office of Fossil Energy (FE), through the National Energy Technology Laboratory (NETL) continues to lead the research and development of solid oxide fuel cell (SOFC) technology. The SOFC program has been working under the Solid State Energy Conversion Alliance (SECA), a unique alliance between industry, government, and academia, since 1999 to develop commercially viable SOFC systems. A milestone was reached this past year with the award of a 400 kW SOFC system to be field tested. In addition to this accomplishment, the FE Fuel Cells program continues to fund research in improving the reliability and robustness of SOFC systems. Coal continues to be the focus of these systems, but the program remains cognizant that these systems can be adapted for advanced power generation systems using natural gas as a fuel. It is thought that be using natural gas and advanced manufacturing techniques, that these systems could be first to the marketplace within the next decade. Recent developments in the SECA program will be presented.

2:00 PM

(ICACC-S3-002-2017) Achievements of NEDO projects on SOFC durability cooperated among industrial stack developers, research institutes and universities in Japan (Invited)

H. Yokokawa*[1]; 1. The University of Tokyo, Japan

SOFC stack durability/reliability has been investigated cooperatively. CRIEPI measured the stack performance by adopting current interruption technique and changing gas compositions in anode and cathode atmospheres to separate the voltage loss into several contributions like overpotentials of electrodes and ohmic losses. This enables to extract the degradation rate for cathode/anode overpotentials and ohmic losses. Post-test examinations have been made on respective parts of stacks; AIST made SIMS analyses on material behaviors at interfaces and Raman analyses on YSZ electrolytes in addition to normal microstructure observation, Kyoto University mad FIB-SEM analyses to make 3D reconstruction of microstructures of electrode/interlayer/electrolyte multilayers, Kyushu University made STEM analyses on chemical states at interfaces, and Tohoku University made analyses on mechanical instabilities of cell assemblies by accumulating data for respective cell components and appropriate simulation techniques. The major degradation issues extracted from stack behaviors are investigated withe simulation techniques developed by Kyoto Univ., the Univ. of Tokyo, and Tohoku Univ. Among 9 stacks tested so far, several common interesting points have been extracted in degradation related phenomena and will be discussed.

2:30 PM

(ICACC-S3-003-2017) Overview on SOFC activities in Germany and Austria (Invited)

M. Kusnezoff*[1]; S. Megel[1]; A. Michaelis[1]; 1. Fraunhofer IKTS, Germany

Beyond all different types of fuel cells SOFC is recognized to be the most promising fuel cell technology to realize high electrical efficiencies up to 60 % based on natural gas. The SOFC technology has been developed and tested in the last decades towards various applications, power ranges and fuels. The industry today sees a transition of R&D activities from purely functional development to durability and cost driven system optimization to achieve market entry requirements. SOFC systems in the power range of 0.1-2.5 kW_{el} are currently the most advanced and closest to the market. The typical target applications are electricity and heat (hot water) supply for single households and power supply for remote consumers. The small SOFC systems are mostly based on CPOX fuel reforming (except of Bluegen system from SOLIDpower (former CFCL)). Currently these systems are widely tested in German and European field test programmes such as "callux" (Germany, 2008-2015) and its successor "Ene.field" (EU, 2012-2017). The field test activities are supported by R&D directed to improvement of durability of stacks and other balance of plant components. Furthermore the R&D work on metal supported cells, stacks, stack modules as well as on systems in power range >5 kW_{el} is in progress. A large number of companies in Germany and Austria is involved in these activities building supply and value chain for future SOFC business.

3:20 PM

(ICACC-S3-004-2017) Leveraging Decades of SOFC Development Investments to Create a Renewable Energy Powered Synfuel Economy (Invited)

L. Frost[1]; J. Elwell[1]; S. Elangovan[1]; J. Hartvigsen*[1]; 1. Ceramatec, Inc., USA

Over the past three decades, tens of millions (10^7 USD) have been invested in SOFC development activities at Ceramatec, with multiple billions (10^9 USD) invested worldwide. There have been great technical advances in performance, system design, and manufacturing learning curves, yet commercial acceptance has been limited to narrow high value markets. In addition to the existing challenges for a business model based on generating commodity electric power from natural gas using SOFC systems, two new barriers are emerging. First is the inevitable march toward CO_2 emissions regulations designed to slow climate-change. Secondly the cost of wind and PV power is falling and the challenges of stabilizing a grid becoming dominated by non-dispatchable generation are increasing. SOFC technology applied as solid oxide electrolysis of CO_2 and steam (SOEC) can be used to produce synthetic liquid transportation fuels to enable storage of renewable energy. Dedicated dispatchable loads employing co-electrolysis to fuels will enable utilization of vastly greater renewable generation capacity and divert fossil fuels from the transportation sector. Technology advances will be discussed including addressing SOEC specific degradation modes, techno-economic analysis, and the back end conversion of co-electrolysis produced synthesis gas to liquid fuels.

Solid Oxide Fuel Cells

Room: Crystal
Session Chair: Mihails Kusnezoff, Fraunhofer IKTS

3:50 PM

(ICACC-S3-005-2017) Saint-Gobain's All-Ceramic SOFC Technology: Progress in Design 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

Energy generation using solid oxide fuel cells (SOFCs) is a strategic project for Saint-Gobain, a global leader in ceramic materials and components. The novel all-ceramic SOFC technology is developed and manufactured to exceed the reliability and durability targets for residential and commercial distributed power generation applications. Ultra-thin ceramic interconnects, simplified stack-supported design and multi-cell co-firing 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 manifolds are presented along with other aspects for future performance improvements. Updates on the Saint-Gobain's SOFC technology, including the improved performance, stable operation for extended hours and the hot-box development are reported in this paper. 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 reported.

4:20 PM

(ICACC-S3-006-2017) Solid Oxide Fuel Cell Technology Developement at FuelCell Energy, Inc.

J. M. Barton*[1]; A. Torabi[1]; C. Willman[1]; H. Ghezel-Ayagh[1]; E. Tang[2];
1. FuelCell Energy, Inc., USA; 2. Versa Power Systems, Ltd, Canada

Fuel Cell Energy, inc (FCE) is advancing the current state of Solid Oxide Fuel Cell (SOFC) technology towards commercial deployment for efficient and nonpolluting generation of electric power from natural gas and other fuels such as coal. SOFC technology represents an important opportunity to utilize fossil fuels in an efficient and environmentally-friendly manner. SOFCs are scalable, efficient (not subject to Carnot cycle limitations), and produce low emissions (e.g. nitrogen oxides) compared to combustion-based electrical power generation technologies (due to lower operating temperatures). SOFC power systems have the potential to achieve greater than 60 percent efficiency (based on higher heating value of fuel) and more than 97 percent carbon capture at a cost-of-electricity that is projected to be approximately 40 percent below that presently available through integrated gasification fuel cell (IGFC) systems with carbon capture. An attractive pathway to deployment of either IGFC or natural gas-fueled fuel cell (NGFC) systems is through near-term market opportunities in natural gas fueled distributed generation (DG) applications. FCE is working on a 400 kWe thermally self-sustaining atmospheric-pressure Solid Oxide Fuel Cell (SOFC) prototype system, with an average stack operating temperature greater than 700°C, to be placed at a prominent site.

4:40 PM

(ICACC-S3-007-2017) Industrial-size demonstration of biogas-fed SOFC: Insight on fuel contaminants

M. Santarelli*[1]; A. Lanzini[1]; M. Gandiglio[1]; D. Papurello[1]; 1. Politecnico di Torino, Italy

The DEMOSOFC project (www.demosofc.eu) aims to demonstrate the technical and economic feasibility of operating an industrial size (174 kWe) SOFC in a wastewater treatment plant (WWTP): the largest plant in EU so far. The scope of the project is demonstrate the high-efficiency conversion of renewable fuel (biogas) into electricity and heat. The three SOFC modules (3x58 kWe) are expected to operate with a net electric efficiency in the range 52-55%. A heat-recovery loop allows recovering useful thermal energy from the hot SOFC exhaust (90-100 kWth). Dealing with fuel contaminants (mainly sulfur and siloxanes) is one of the key issue in the biogas-fed SOFC. There is a two-fold interest, (1) to deepen the understanding of the tolerance limits of the Ni-anode towards contaminants; (2) to elucidate the degradation mechanism related to these contaminants, aimed to the optimized design and operation of the clean-up section. In this work, we analyse: (1) the impact of contaminants on the SOFC stack in order to identify the threshold limits of the fuel cell system towards specific contaminants; (2) the technological solutions and related adsorbent materials to remove contaminants in a dedicated clean-up unit upstream of the fuel cell plant.

5:00 PM

(ICACC-S3-008-2017) Optimal Control of a Hybrid Solid Oxide Fuel Cell – Gas Turbine System based on Stall/Surge Risk

M. Azizi*[1]; J. Brouwer[1]; 1. National Fuel Cell Research Center, University of California, Irvine, USA

One of the main purposes of an SOFC-GT hybrid system is for distributed power generation applications. This study investigates the possible use of an SOFC-GT hybrid system to power multi-MW dynamic loads. Based upon the integration of commercially available gas turbine technology, control strategies for the SOFC-GT hybrid system are investigated for different stationary power applications. Risk analysis of surge in the hybrid SOFC-GT power system as it is dynamically dispatched to meet demand is assessed in transient pre-load and post-load modes. Cycles that compare different types of reformers (SMR and CPOX) and different cycle configurations (anode recirculation included and without anode recirculation) are compared. Optimal PID control algorithms are developed and applied to predict the system response to sudden load demand changes. Possible integration of the hybrid SOFC-GT system with other components of a distributed generation system (Chiller, thermal storage, batteries, solar PVs and wind turbines) is discussed, specifically as it may affect stall/surge phenomena. Several different cases such as base load, diurnal peaking, energy storage shift and load following operation are studied. High surge risk was found to be primarily associated in with pre-load to post-load transitional operating conditions.

S4: Armor Ceramics

Ceramic Response to Ballistic Impact I

Room: Coquina Salon E
Session Chair: Jerry LaSalvia, U.S. Army Research Laboratory

1:30 PM

(ICACC-S4-001-2017) The Effect of Impact Conditions on Fracture and Damage in Brittle Materials (Invited)

B. B. Aydelotte*[1]; B. Schuster[1]; P. Jannotti[1]; 1. US Army Research Laboratory, USA

Brittle materials such as ceramics and glasses are widely employed because they possess a variety of useful properties including hardness, strength, wear resistance and/or transparency. A variety of static indentation and impact experiments are used to explore their potential performance or characterize their behavior. Normal and oblique impacts are an important class of impact scenarios with respect to the types of damage and fracture they induce. Classic work on normal and sliding sphere indentation is reviewed and compared with the results of normal and oblique sphere and $L/D \leq 5$ projectile impacts on brittle materials. Continuum scale simulations are used to gain insight into the mechanics of these results and examine the evolving state of the target and projectiles during impact of these idealized projectiles.

2:00 PM

(ICACC-S4-002-2017) Analysis of the interaction of projectiles with ceramic targets by means of Flash X-ray cinematography and optical methods (Invited)

E. Strassburger*[1]; S. Bauer[1]; 1. Fraunhofer EMI, Germany

In order to improve the performance of ceramic composite armor in an efficient way, a profound knowledge of the mechanisms during each phase of the projectile/target interaction and their influence on the penetration resistance is essential. Visualization techniques enable insight into the dynamics of projectile penetration. While optical techniques are suitable to examine the impact, shock wave and fracture propagation phase, flash X-ray imaging is needed to study the penetration phase. A flash X-ray imaging method has been developed at EMI, which provides up to eight flash radiographs in one experiment. A multi-anode flash X-ray tube is utilized with this method, and the fluorescent image of the transmitted radiation is photographed by means of a high-speed digital camera. This technique has been applied to visualize and analyse the penetration of AP projectiles into different opaque (SiC, B_4C, Al_2O_3) and transparent (MgO-Al_2O_3-Spinel, AlON) ceramics. The influences, not only of the ceramic but also the backing material, on dwell time and projectile erosion have been studied. The combined application of flash x-ray and optical visualization techniques on ceramic composite targets allowed a correlation of instantaneous depth of penetration, state of fragmentation of the ceramic and deformation of the ceramic and backing.

Ceramic Response to Ballistic Impact II

Room: Coquina Salon E
Session Chair: Lionel Vargas-Gonzalez, U.S. Army Research Laboratory

3:20 PM

(ICACC-S4-003-2017) The Effect of Projectile Orientation, Shape, and Surface Conditions on Ballistic Impacts (Invited)

B. B. Aydelotte*[1]; B. Schuster[1]; P. Jannotti[1]; 1. US Army Research Laboratory, USA

Brittle materials such as ceramics and oxide glasses are widely employed because they possess a variety of useful properties including hardness, strength, wear resistance and/or transparency. A variety of static indentation and impact experiments are used to explore their performance or characterize their behavior. Normal and oblique impacts of different projectile configurations are an important class of impact scenarios with respect to the types of surface and interior conditions which they induce. Classic work on normal and sliding sphere indentation is reviewed and compared with the results of normal and oblique sphere and L/D ≤ 5 projectile impacts on brittle materials. Continuum scale simulations are used to explore these results and examine the evolving state of the target and projectiles during impact as a function of projectile orientation, shape, and surface conditions on the target.

3:50 PM

(ICACC-S4-004-2017) Tribochemical Reactions in Boron Carbide Impacted at High-Velocity

J. LaSalvia*[1]; V. Domnich[2]; B. Schuster[1]; B. B. Aydelotte[1]; E. R. Shanholtz[4]; A. Giri[1]; S. D. Walck[1]; K. D. Behler[1]; C. J. Marvel[3]; M. P. Harmer[3]; 1. Army Research Laboratory, USA; 2. Rutgers University, USA; 3. Lehigh University, USA; 4. Independent, USA

For boron carbide impacted with a cemented carbide sphere at high-velocity, millimeter long, micrometer wide features consisting of micron and nano-sized particles were observed several millimeters from the site of impact. Morphologies of alumina inclusions found within these features suggest the presence of a high temperature melt. Furthermore, oxidation of boron carbide during the ballistic event appears plausible because Raman spectra from these features were similar to those from boron carbide oxidized unintentionally at high temperatures. Energy-dispersive spectroscopy confirmed the presence of oxygen in these features. These results, along with results from thermal analytical experiments, provides evidence for melt formation via tribochemical reactions. During impact, cemented carbide fragments slide across the boron carbide surface resulting in frictional heating. Oxidation reactions are initiated which lead to melt formation. The resulting melt penetrates into cracks, resulting in the observed features. This is supported by an analytical model and TEM analyses. Experimental procedures, results, and analyses are presented.

4:10 PM

(ICACC-S4-005-2017) Role of inertia in armor ceramics

E. Carton*[1]; G. Roebroeks[1]; J. Weerheijm[1]; A. Diederen[1]; 1. TNO, Netherlands

Ever since research on armor ceramics started one has, with limited success, searched for relations between mechanical properties and the ballistic efficiency of this class of materials. Also at TNO research on this subject has been performed using a range of experimental test set-ups (mainly using small and mid-caliber rounds) and diagnostic tools on a range of ceramic materials and tile thicknesses. This research has highlighted the large role that inertia (mass) plays during the projectile-target interaction process. In fact, all our experimental results can be well understood from an inertia point of view. This includes the widely accepted (increasing) order in ballistic efficiency of the most used armor ceramics when tested at equal areal density: Alumina, SiC, B_4C. The role of inertia can also be obtained from the early work on armor ceramics by M. Wilkins (end sixties, early 70's). Based on the gained knowledge, TNO has generated an engineering model for ceramic based armor (ceramic strike face with/without a backing material) that can quantify the projectile-target interaction and quite accurately calculates the mass-loss and deceleration of the projectile in a time-resolved manner. Understanding the large role of inertia could change the direction of the development of advanced armor ceramics.

4:30 PM

(ICACC-S4-006-2017) Studying Surface Waves and Failure Fronts in Glass Using a Combined Experimental and Computational Approach

J. McDonald*[1]; S. Satapathy[1]; M. Pena[2]; M. Tabia[3]; B. Otoole[3]; 1. US Army Research Laboratory, USA; 2. National Security Technologies, LLC, USA; 3. University of Nevada Las Vegas, USA

DOC/NV/25946-2988 The failure front phenomena in glass has previously been well documented under conditions of 1-d strain and 1-d stress. Under the complex stress states induced by projectile impact the existence and possible role of failure fronts in glasses and ceramics is not currently understood. In the present study the multi-probe Photon Doppler Velocimetry (PDV) technique is utilized to measure surface velocities at the impact face of a glass block following impact by a Polycarbonate cylinder. It was discovered that, by utilizing a separate probe to track the projectile velocity through the back surface, it is possible to detect the velocity of a surface moving through the glass which attains a maximum velocity much higher than the impact velocity. We believe that the origin of this signal is the direct reflection of the PDV laser by a failure front. To our knowledge this is the first reported observation of such a phenomena. Comparison of the measured data to computational results employing different damage models will be presented and a discussion given of the apparent importance of various failure criteria in these highly-inhomogeneous stress states. This work was done by National Security Technologies, LLC, under Contract No. DE-AC52-06NA25946 with the U.S. Department of Energy and supported by the Site-Directed Research and Development Program

4:50 PM

(ICACC-S4-007-2017) Influence of encased aluminum alloy on the ballistic performance of alumina armor plate

J. Lo*[1]; R. Santos[1]; R. Zhang[1]; D. Walsh[1]; G. Birsan[1]; F. Benkel[1]; R. Bowes[2]; L. Goheen[2]; 1. CanmetMATERIALS, Canada; 2. CanmetCERL, Canada

Ceramic armour plates with various backing materials have been developed for the purpose of improving the ballistic performance of the ceramics. The ceramic plate serves the purpose of deforming and fracturing incoming projectiles, while a backing layer is meant to reduce and absorb shock waves, as well as to confine fracture debris. It has been found that the ballistic performance of a ceramic plate can be substantially improved when a backing material is present. In this work, the effects of ballistic performance of an alumina with encased aluminum alloys is investigated. A technique of providing a backing material to a ceramic armor plate as well as to encase the plate in an aluminum alloy has been developed at CanmetMATERIALS. The encasement of the ceramic plate also offers the benefit multi-hit capability. In this work, the mechanical properties and microstructural information of the aluminum alloys used were measured and acquired respectively. This is followed with a Depth of Penetration (DOP) test which was used to determine the extent of improvement on the ballistic performance of alumina armour with a backing and encased material. Finally, failure analysis was conducted on the tested plates to elucidate the penetration mechanisms as well as how the ballistic energy was absorbed by the backing and encased plates.

5:10 PM

(ICACC-S4-008-2017) Hybrid Ceramic Composite Armor

J. Stiglich*[1]; A. Fortini[1]; 1. Ultramet, USA

Ultramet has begun design of a low visibility armored vest concept for undercover personnel in situations where identification of the vest by an adversary would have immediate negative consequences. A key component of the vest is Ultramet's high-toughness ceramic matrix composite backing structure, which is bonded to a monolithic ceramic strike face. This presentation will describe prototype/DoP ballistic testing of ceramic matrix composites (CMC's) as an important component of a low visibility ballistic protection structure. The carbon fiber reinforcements are available in 2D and 3D

structures developed for high temperature aerospace applications. Matrix examples include SiC, ZrC, TiB2, and alloys or mixtures of these compounds. Both flat and curved shapes have been developed. Prototype Si-SiC matrix CMC's have been tested with SiC strike plates and demonstrated defeat of 0.30 cal/7.62 mm APM2 projectiles at 2800 to 2900 fps in DoP ballistic experiments. By replacing the SiC with other materials, armor thicknesses as low as 0.31" are anticipated for protection against AP M2 threats. Ballistic tests and data will be presented.

5:30 PM

(ICACC-S4-009-2017) Evaluation of temperature jump at the front of comminution and compaction of the ceramic target material at high-velocity impact

B. A. Galanov[1]; V. V. Kartuzov*[1]; S. M. Ivanov[1]; A. A. Pryadko[1]; 1. IPMS, Ukraine

From experimental observations it is known that there is a temperature jump at the boundary between the cracked and crushed material during penetration into ceramic. High-energy collision is sometimes accompanied by the light emission, one of the causes of which may be an increase in temperature. The report presents a model predicting the temperature jump at the comminution and compaction front during penetration of the projectile into ceramic materials. The model is based on the analysis of the stress-strain state of the material during the expansion of a spherical cavity in brittle materials. The main source of the temperature jump - a jump of pressure and shear stresses at the boundary between the region of comminution and cracking region of the material. The model allows to estimate the energy loss on heating depending on the parameters of the material within the framework of analytical model.

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

Thermal Energy Conversion and Energy Storage I

Room: Tomoka A

Session Chair: Palani Balaya, National University of Singapore

1:30 PM

(ICACC-S6-001-2017) Electrochemistry as a tool to explore the Na_xMoO_2 phase diagram (Invited)

C. H. Delmas*[1]; l. Vitoux[1]; M. Suchomel[2]; J. Christian[3]; N. Sharma[3]; M. Guignard[1]; 1. ICMCB, France; 2. APS -, USA; 3. School of Chemistry, Australia

Sodium layered oxides have been intensively studied recently as positive electrode for sodium-ion batteries, which present a great interest for stationary energy storage. Research also focuses on sodium layered oxides to understand the strong correlation they exhibit between their chemical composition, structure and original physical properties. This work focus on Na_xMoO_2 layered oxides, In this presentation, we will present our results on the investigation of the Na_xMoO_2 phase diagram by electrochemical (de)intercalation and in-situ and ex-situ X-ray diffraction. The $Na_{2/3}MoO_2$ phase has been synthesized by solid state reaction and used as positive electrode in a sodium cell. The very undulating aspect of the cycling curves reveals numerous reversible structural transitions in the $0.2 < x < 0.85$ range. In addition, GITT shows high intercalation kinetics and very small polarization. In-situ X-ray Synchrotron diffraction experiments enabled to draw out a first phase diagram. Triclinic distortions as well as several superstructures and modulations were also evidenced for $0.58 < x < 0.75$. Interestingly, a change of 0.02 in the sodium content in this domain of composition, have direct effect on the structure. All structural transitions are fully reversible in the $0.5 < x < 1.0$ range. For higher amount of Na deintercalation, the reversibility is slightly reduced due to reaction with the electrolyte.

2:10 PM

(ICACC-S6-002-2017) Development of Na-Ion Batteries for Energy Storage (Invited)

Y. Hu*[1]; 1. Institute of Physics, Chinese Academy of Sciences, China

Among the various energy storage technologies, electrochemical approach represents one of the most promising means to store the electricity in large-scale because of the flexibility, high energy conversion efficiency and simple maintenance. Li-ion batteries have been explored as power sources for various types of important applications, such as portable electric devices and electrical vehicles. However, the rarity (0.0065%) and non-uniform distribution of lithium in the Earth's crust may not simultaneously support these two important application areas: electric vehicles and smart grid. Alternatively, Na-ion batteries have been reconsidered particularly for grid application, where cost and cycle life are more critical factors than energy density due to the abundant sodium resources (2.75%) and low cost as well as similar "rocking-chair" Na storage mechanism as Li. Furthermore, we can use Na^+ ions as the charge carrier to explore new chemistry and new materials to further decrease the cost. In this talk, I will present our recent research progress from IoP-CAS. In particular, I will focus on a series of Na-Cu-Fe-Mn-O cathode and a superior low cost amorphous carbon anode made from renewable biomass and anthracite. On the basis of above results, prototype Na-ion pouch cells were fabricated. I will also show the electrochemical and safety performance of these pouch cells.

2:40 PM

(ICACC-S6-003-2017) Design of new materials as electrode materials for Na ion batteries

W. Deriouche[2]; M. Freire[1]; N. Amdouni[2]; A. Maignan[1]; V. Pralong*[1]; 1. CNRS, France; 2. Unité de Recherche Physico-chimique des Matériaux Condensés, Tunisia

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. We will discuss on our strategies to generate such original frameworks. The first approach is based on topotactic reactions starting from existing phases with a compact anionic framework. 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. We will discuss on our strategies to generate such original frameworks, new materials for Na ion battteries based on vanadium and manganese oxides.

Thermal Energy Conversion and Energy Storage II

Room: Tomoka A

Session Chair: Shyue Ping Ong, University of California, San Diego

3:20 PM

(ICACC-S6-004-2017) Oxide-Based Solid State Electrolytes for All-Solid-State Li-Ion Batteries (Invited)

M. J. Hoffmann*[1]; T. Hupfer[2]; F. Lemke[1]; E. Bucharsky[1]; G. Schell[1]; A. Hintennach[3]; 1. Karlsruhe Institute for Technology (KIT), Germany; 2. Robert Bosch GmbH, Germany; 3. Daimler AG, Germany

Lithium lanthan titanate (LLTO) and lithium titanium aluminum phosphate (LATP) are potential candidates as oxide solid state electrolytes with a high Li-ion conductivity. LATP with the composition $Li_{1,3}Al_{0,3}Ti_{1,7}(PO_4)_3$ was prepared by a modified sol gel method. Densification was obtained by field assisted sintering (FAST) as well asconventional pressureless sintering. LLTO was prepared by a standard mixed oxide route and pressureless densified. The resulting phase composition, crystal structure and theoretical density of both phases were obtained from XRD analysis. Electrical properties, such as ionic conductivity, were determined by impedance spectroscopy in the frequency range from 1 Hz to 1 MHz. A maximum conductivity in the order of 10^{-3} S/cm at room temperature is obtained for phase-pure LATP compositions combined with a high relative density. However, strong anisotropy in thermal expansion causes microcracking in coarse-grained materials which finally leads to a reduction in ionic conductivity. The LLTO contains small quantities of Li-titanate as second phase and reveals an ionic conductivity of 10^{-3} for the grains and up to 10^{-4} for the grain boundaries. No significant influence of the second phase has been detected, but grain growth enhances grain boundary conductivity by approximately one order of magnitude.

3:50 PM

(ICACC-S6-005-2017) Synthesis and Characterization of Nanostructured $Li_7La_3Zr_2O_{12}$ Solid Electrolytes using Electrospinning and Nanocellulose Templating (Invited)

C. K. Chan*[1]; 1. Arizona State University, USA

Garnet-type lithium lanthanum zirconate ($Li_7La_3Zr_2O_{12}$, LLZO) is a promising ceramic solid electrolyte for all-solid-state lithium batteries. However, the different phases of LLZO (tetragonal and cubic), differ in lithium ionic conductivity by several orders of magnitude, with extrinsic dopants often required to stabilize the high conductivity cubic phase. Here we show that cubic LLZO can be stabilized at room temperature in nanostructured particles without the use of extrinsic dopants. LLZO nanowires were synthesized using electrospinning and formed cubic phase materials after only 3 h calcination at 700°C. Nanostructured LLZO was also formed via templating on various cellulosic fibers. Templating was determined to be an effective method for controlling the LLZO particle size and morphology, with most experiments resulting in fibrous LLZO of the same magnitude and morphology as the starting template. Bulk LLZO with tetragonal structure was transformed to the cubic phase using particle size reduction via ball milling. Heating conditions that promoted particle coalescence and grain growth induced a transformation from the cubic to tetragonal phase in both types of nanostructured LLZO. Using nanostructured LLZO over bulk LLZO can be beneficial in terms of ionic conductivity, cycle life, and mechanical strength.

4:20 PM

(ICACC-S6-006-2017) $Li_7La_3Zr_2O_{12}$ Interface Modification for Li Dendrite Prevention (Invited)

C. Tsai*[1]; V. Roddatis[2]; C. Chandran[3]; Q. Ma[1]; S. Uhlenbruck[1]; P. Heitjans[3]; O. Guillon[1]; 1. Institute of Energy and Climate Research, Germany; 2. Institute of Materials Physics, Germany; 3. Institute of Physical Chemistry and Electrochemistry, Germany

The development of electrochemical energy storage devices is toward to higher energy density and safety. The use of metallic Li as anode for rechargeable batteries could increase tenfold in the anode storage capacity when compared to carbon anode. However, it is not successful until now due to the difficulty of suppressing the growth of Li dendrite. Theoretical calculations suggest that a shear modulus of used electrolyte is more than twice that of metallic Li or a Li-ion transfer number t_{Li+} approaching 1 could supress dendrite growth. Therefore, the garnet structured $Li_7La_3Zr_2O_{12}$ (LLZ) is an ideal material for using as electrolyte because its $t_{Li+} \sim 1$, high mechanical strength and chemical stability to metallic Li. However, Li dendrite formation was reported by Ishiguro et al [1,2] for their Nb- and Ta-substituted LLZ. In this research, Al-free Ta-substituted LLZ were fabricated by hot pressing with a relative densities >99%. During the dendrite studies, impedance measurements show rapid decrease in total resistances indicates the dendrite can be formed in such a highly dense ceramic in a short time. Solid-State NMR demonstrates the presence of metallic Li inside the dense pellet which is also supported by TEM-EELS result [3]. The dendrite test results, reasons for the formation of the Li-dendrite and pathways to prevent the formation of Li-dendrite by interface modification will be presented.

4:50 PM

(ICACC-S6-007-2017) A new solid solution with NASICON structure and high ionic conductivity: $Na_{3+x}Sc_2Si_xP_{3-x}O_{12}$

O. Guillon*[1]; M. Guin[1]; F. Tietz[1]; 1. Forschungszentrum Juelich, Germany

Although many NASICON compositions were investigated so far, the solid solution $Na_{3+x}Sc_2(SiO_4)_x(PO_4)_{3-x}$ with $0.05 \le x \le 0.8$ has been investigated for the first time. The various compositions were synthesized by solid state reaction and their crystallographic and electrical properties were measured. The increase in charge carriers led to a maximum conductivity at x = 0.4 with $\sigma_{Na,total} = 6.9 \times 10^{-4}$ S cm^{-1} at 25°C. Reasonable correlations with specific crystallographic parameters were found determining the transport properties. The change in lattice size directly influenced the size of the pathway for the Na^+ conduction and the hopping distance of the Na^+ ions to the next vacancy. A hopping distance that is too long requires a high activation energy for the conduction, which was observed for the compounds $Na_{3.6}Sc_2(SiO_4)_{0.6}(PO_4)_{2.4}$ and $Na_{3.8}Sc_2(SiO_4)_{0.8}(PO_4)_{2.2}$. The distance between the charge carriers to the nearest oxygen atoms seems to be very important for high ionic conduction.

5:10 PM

(ICACC-S6-008-2017) Towards All Solid State Batteries using perovskite solid electrolytes

M. Lachal*[1]; T. Bibienne[1]; L. Groleau[1]; M. Marmiesse[1]; F. Bardé[2]; L. Castro[2]; M. Dolle[1]; 1. University of Montréal, Canada; 2. Toyota Motor Europe, Belgium

Safety and environmental concerns are some of the main issues when considering lithium batteries technology. As an alternative, All Solid State Batteries (ASSB) may give a fundamental solution to solve those concerns. Indeed, the solvent-free ASSB show major advantages over current lithium batteries: thermal stability, possibility of multicell assembly, absence of leakage and pollution (safety and environmental impact on disposal), possibility to use high potential electrode materials (no electrolyte degradation). We recently reported the approach to assemble ASSB in one step by Spark Plasma Sintering (SPS) using phosphate materials [1,2]. Investigations on oxide electrolytes were considered to extend our approach. Amongst the solid oxide electrolytes, perovskite materials cover most of these requirements. Several perovskite electrolytes were synthesized and characterized by XRD and impedance spectroscopy after sintering by SPS. Their thermal stability towards well-known electrode materials was also investigated to ascertain the best materials combination in order to develop ceramic composite electrodes. Finally, electrodes and electrolyte interfaces were described using SEM and EDS observations. This presentation will focus on the required steps to develop ASSB using perovskite solid electrolytes.

S9: Porous Ceramics: Novel Developments and Applications

Innovations in Processing Methods & Synthesis of Porous Ceramics I

Room: Coquina Salon G
Session Chair: Paolo Colombo, University of Padova

1:30 PM

(ICACC-S9-001-2017) Porous ceramics development by additive manufacturing: Design, manufacturing and testing (Invited)

A. Ortona*[1]; 1. SUPSI, Switzerland

Additive manufacturing has the potential to revolutionize engineering because of its obvious advantages in the new product development phase. This is especially true for plastic and metallic components where small series are already produced at competitive costs. The technical ceramic industry is now starting to seriously consider this technique which, thanks to the recent developments in ceramic additive manufacturing, is now mature for industrial applications. Often though the "additive manufacturing" part of new products R&D is considered alone. We show in this work that, especially for porous ceramic architectures, design, simulation and testing are extremely important in R&D of new ceramic components. This work is a critical review of several components we developed and produced by additive manufacturing in the automotive field, in aerospace, but mostly in energy production, transfer and storage.

2:00 PM

(ICACC-S9-002-2017) Highly porous B-doped hardystonite bioceramics from preceramic polymers and engineered fillers: From foams to 3D-printed scaffolds

H. Elsayed[1]; P. Colombo[1]; E. Bernardo*[1]; 1. University of Padova, Italy

Highly porous hardystonite-based bioceramics, in the form of foams and 3D scaffolds, have been obtained by the thermal treatment of silicone resins and engineered micro-sized oxide fillers, in air. Besides CaO and ZnO precursors ($CaCO_3$ and ZnO powders), commercial silicone resins embedded calcium borate, in both hydrated and anhydrous form ($Ca_2B_6O_{11}.5H_2O$ and $Ca_2B_6O_{11}$, respectively), having a significant impact on the microstructural evolution. In hydrated form, calcium borate led to a substantial foaming of silicone-based mixtures, at low temperature (420°C); after dehydration, upon firing, the salt provided a liquid phase, favouring ionic interdiffusion, with the development of novel B-contaning hardystonite-based solid solutions ($Ca_2Zn_{1-x}B_{2x}Si_{2-x}O_7$). In anhydrous form, the specific filler did not lead to any foaming but, used in formulations for direct ink writing, in turn leading to reticulated scaffolds, kept its role in phase evolution upon firing. Although fired at lower temperature than previously developed silicone-derived hardystonite cellular ceramics (950°C, instead of 1200°C), the newly obtained foams and scaffolds exhibit remarkable mechanical properties (compressive strength exceeding 4 MPa, with a total porosity above 67%).

2:20 PM

(ICACC-S9-003-2017) Microstructural, mechanical and thermal characterization of alumina gel-cast foams manufactured with the use of agarose as gelling agent

T. Fey*[1]; Z. Bodo[1]; G. Peter[1]; P. Marek[2]; 1. Friedrich-Alexander-Universityet Erlangen-Neurnberg, Germany; 2. Rzeszow University of Technology, Poland

Alumina gel-cast foams manufactured using agarose as gelling agent were examined in terms of microstructural, mechanical and thermal properties. The microstructural measurements of alumina foams made using SEM were compared with those using X-ray micro tomography. Young's Modulus of alumina foams was determined using impulse excitation and ultrasonic sound velocity measurements. A good accordance was found between two independent techniques. The experimental data were also compared with the behavior that is expected for completely open-cell and closed-cell foams according to Gibson and Ashby model. The thermal conductivity measurements were carried out by Laser-Flash Analysis correlating to the pore network in the alumina foam structure.

2:40 PM

(ICACC-S9-004-2017) Preparation of porous polycrystalline ceramic solid electrolytes via thermal removal of sacrificial alkali halides

R. Muccillo*[1]; T. Porfirio[1]; 1. IPEN, Brazil

One of the requirements for electrodes in solid oxide fuel cells and in ceramic membranes for selective permeation of gaseous species is their controlled porosity. Samarium-doped ceria (SDC), an oxide ion conductor was prepared with variable degree of porosity by adding and thermally removing either NaCl, KCl or LiF alkali halides during sintering. The porosity was determined by analysis of surfaces of the specimens by scanning electron and scanning probe microscopies. The degree of porosity was analyzed by electrochemical impedance spectroscopy at temperatures where oxide ion conductivity is predominant (400-500°C). The residues of alkali halides were evaluated by X-ray fluorescence (XRF). Porous ceramic samples with high skeletal density were obtained. Impregnation of $K,Li(CO_3)$ under vacuum at its eutectic temperature was carried out to prepare SDC-molten carbonate composite solid oxide fuel cells. XRF analysis showed the face-to-face. Impedance spectroscopy results at the eutectic temperature showed the enhancement of the ionic conductivity via percolation through the molten carbonate in the SDC matrix.

Innovations in Processing Methods & Synthesis of Porous Ceramics II

Room: Coquina Salon G
Session Chair: Tobias Fey, Friedrich-Alexander-University at Erlangen-Neurnberg

3:20 PM

(ICACC-S9-005-2017) Porous alumina ceramics fabricated by novel powder processing (Invited)

J. Tatami*[1]; M. Iijima[1]; 1. Yokohama National University, Japan

Most of porous ceramics were fabricated by powder processing. Improvement of powder processing should result in better porous ceramics. In this study, novel powder processing to fabricate porous alumina ceramics will be discussed. Organic binder is usually added to improve the strength of the green body of porous ceramics. However, it is sometimes difficult to maintain the shape of the green body after burning out. We fabricated porous alumina ceramics using basic aluminum lactate as an inorganic binder. The density of the porous alumina was independent of the quantity of basic aluminum lactate. The strength of green body not only before but also after dewaxing was improved by adding basic aluminum lactate. Addition of basic aluminum lactate as an inorganic binder was also effective in the improvement of the strength of sintered porous alumina, which resulted from neck growth by alumina formed from the basic aluminum lactate. Porous honeycomb alumina was also fabricated using a small resin mold prepared by stereolithography. A slurry of alumina powder and chopped carbon fiber was molded into the small mold in a magnetic field. After firing the green body, honeycomb alumina with unidirectional pore was obtained. As a result, combination of a magnetic molding technique and use of a small resin mold prepared by stereolithography was found to be effective in control of pore structure in porous ceramics.

3:50 PM

(ICACC-S9-006-2017) Properties of Anisotropic Porous Alumina Fabricated by Alumina Platelets (Invited)

S. Honda*[1]; K. Matsubara[1]; Y. Daiko[1]; S. Hashimoto[1]; Y. Iwamoto[1]; 1. Nagoya Institute of Technology, Japan

Porous alumina has been investigated as support material for the ceramic permselective membranes. There is a fundamental trade-off between the mechanical properties and the permeation property, and the fluid permeability through the porous support decreases consistently with the decrease in the porosity or pore size. In this research, the porous alumina with anisotropic microstructure focused on the porous support substrates for permselective microporous ceramic membrane was fabricated by alumina platelets. The permeability, thermo-mechanical properties and thermal shock strength of porous alumina were evaluated. To study the influence of anisotropic microstructure and porosity for the properties, porous alumina with different porosities were fabricated by various sintering conditions. The anisotropy of thermal conductivity and permeability increased with porosity. This research clarified quantitatively to the relation between properties of porous alumina and anisotropic porous microstructure.

4:20 PM

(ICACC-S9-007-2017) Open Cell Geopolymer foams

P. Colombo*[1]; C. Bai[1]; 1. University of Padova, Italy

Open cell alkali or acid-based geopolymer foams were produced by direct foaming using different fabrication approaches. Potassium-based foams with a porosity up to 85 vol% were obtained from metakaolin, potassium silicate and potassium hydroxide, while metakaolin and phosphoric acid were used to fabricate foams containing an aluminum phosphate crystal phase already after synthesis at room temperature, and a total porosity of ~80 vol%. The strength of the foams depended on the porosity of the components as well as the heat treatment temperature.

4:40 PM

(ICACC-S9-008-2017) Development of high porosity-high strength Si_3N_4 bodies prepared via different routes

F. Golestanifard*[1]; M. Sadeghpour[1]; P. Tabrizian[1]; A. Parsi[1]; 1. University of Science and Technology of Iran, Islamic Republic of Iran

Porous silicon nitride bodies are interesting for a wide range of application such as filters and biocompatible implants. The main challenge is to retain the strength in a level of above 150 MPa for bodies with 40 to 50 percent porosity. In this paper, processing and characterization of porous Si_3N_4 bodies prepared from primary Si will be compared with those obtained from Si_3N_4 powder. Gel casting and sol-gel methods were the main routes in both cases. Sintering aids were different amounts of Al_2O_3 and Y_2O_3. Products were characterized in terms of density, porosity, MOR, phase evolutions and microstructural developments. It was found that the optimized processing would lead to 150-250 MPa flexural strength in bodies with high level of porosity. The amount of β phase was found to play the main role and its development depends on many processing conditions and soaking time. Properties and formulation of monomers, sintering temperature and time are other important factors. The result of present research will be explained with the emphasis on application and comparison with previous reports.

5:00 PM

(ICACC-S9-009-2017) Fabrication and properties of porous alumina prepared by alumina slurry including aluminum and polysiloxane

K. Kita*[1]; M. Fukushima[1]; H. Hyuga[1]; N. Kondo[1]; 1. National Institute of Advanced Industrial Science and Technology (AIST), Japan

Porous ceramics are one of promising materials in engineering fields such as vehicles, environmental purification, water conservation and petro chemistry, etc. As a novel method of making porous alumina, sintering of the mixed powder containing aluminum powder, alumina powder and polysiloxane was employed. Volume expansion of porous alumina as interesting remarks was found, because of oxidation of aluminum during sintering, in which the rate of volume change and the bending strength were measured and compared with those of the porous alumina derived from compacted alumina powder.

S10: Virtual Materials (Computational) Design and Ceramic Genome

Modelling - Electrical Properties

Room: Ponce DeLeon
Session Chair: Michael Tonks, Pennsylvania State University

1:30 PM

(ICACC-S10-001-2017) Strategies for enhancing the thermoelectric performance of oxides (Invited)

U. Schwingenschlogl*[1]; 1. KAUST, Saudi Arabia

We employ first-principles calculations to develop strategies for improving the thermoelectric response of selected oxides. The talk first focusses on substitutional doping in prototypical $SrTiO_3$, in particular on the consequences of induced spin polarization. We also take into consideration strain effects. The second part of the talk deals with the thermoelectric properties of the layered rhodates K_xRhO_2 and Na_xRhO_2, for which the roles played by the cation concentration and lattice parameters are investigated. Comparison of 2H and 3R phases (modified stacking of atomic layers) provides insight into the effect of the interlayer coupling.

2:00 PM

(ICACC-S10-002-2017) Thermoelectric thin film chromium- and scandium-based nitrides studied by an integrated theoretical-experimental approach (Invited)

P. Eklund*[1]; 1. Linkoping University, Sweden

Thermoelectric devices directly convert heat into electricity or vice versa. However, the conversion efficiency of thermoelectric devices of today is limited. The critical material-dependent parameter is the figure of merit ($ZT = S^2T/\rho\kappa$, where ρ is the electrical resistivity, S is the Seebeck coefficient and κ is the total thermal conductivity). Here, we present results from our experimental and theoretical investigations of CrN- and ScN-based thin film systems. ScN thin films exhibit an anomalously high power factor (S^2/ρ), but has high thermal conductivity. To reduce thermal conductivity, potential strategies are nanostructuring, superlattices, alloying or nanoinclusions. Pure CrN exhibits a high power factor enabled by a high electron concentration thermally activated from N vacancies. Further, the trends in mixing thermodynamics and densities-of-states of rocksalt-$Cr_{1-x}Sc_xN$ solid solutions ($0\leq x\leq 1$) were investigated by first-principles calculations, and $Cr_{1-x}Sc_xN$ thin films synthesized by magnetron sputtering. To understand which alloying elements that could be of interest at elevated temperatures where diffusion can be activated, we have investigated the trends in mixing thermodynamics of ScN-based solid solutions, correlated with experimental studies and developed models for grain-size and microstructural effects on thermal conductivity.

2:30 PM

(ICACC-S10-003-2017) The Universal Conductive Network in Compounds with Chalcogenide Sublattices (Invited)

J. Yang*[1]; 1. Shanghai University, China

The concept of 'Phonon glass-electron crystal' (PGEC), which allows us to tune the electrical and thermal transport separately, offers new directions to seek novel high performance thermoelectric (TE) materials. Recently, we recognized that the PGEC paradigm can be annotated in the compounds with conductive networks. The compounds with conductive networks have only part of the structures that contribute to the electrical transport, while the tuning of the carrier concentrations and lattice thermal conductivities can be achieved in the rest part. Our previous work on filled skutterudites and Cu_2SnSe_3 have shown that the TE performance can be improved by using the conductive networks. In this work, we systematically study the general conductive network in chalcogen-based compounds by first principles. Our results demonstrate that compounds with similar chalcogen sublattice almost follow the similar Seebeck coefficients. This implies that the electrical transport properties of these compounds are mainly dependent on the sublattice of chalcogen, which is the key feature of "electron crystal". The variations on electrical transport properties of different chemical characteristic from S, Se, to Te are investigated. New TE compounds can be inspired by the concept of conductive network in chalcogenides.

3:20 PM

(ICACC-S10-004-2017) Correlation between Grain Boundary Segregation and Resultant Ionic Conduction in Grain Boundaries (Invited)

M. Yoshiya*[1]; T. Yokoi[1]; 1. Osaka University, Japan

Segregation of matrix element species, doped impurities, and associated point defects at grain boundaries or hetero-phase interfaces is always present in polycrystalline materials, since change in local atomic coordinations brings about change in chemical potential near the boundaries or interfaces. It has been well known that the segregation not only modifies microscopic local properties in the vicinity of grain boundaries or interfaces but also often governs overall macroscopic properties of polycrystalline materials, despite the fact that volume fraction of the grain boundaries and interface is negligibly small. Recent progress in Cs corrected scanning transmission electron microscopy enables us to directly compare spatial distribution of the segregants with computed ones near the boundaries and further atomistic simulations enable us to reveal physics or microscopic thermodynamics behind the phenomena observed by experiments. In this talk, focusing aliovalent oxide-doped zirconia-based materials which are well known for solid state electrolytes in solid oxide fuel cell as well as high temperature refractory materials, we will discuss how segregants are driven to segregate at grain boundaries and how the segregation modifies the ionic transport in the vicinity of grain boundary.

3:50 PM

(ICACC-S10-005-2017) Prediction of cubic-YSZ conductivity by applying the CALPHAD approach

M. Asadikiya*[1]; Y. Zhong[1]; 1. Florida International University, USA

The cubic polymorph of yttria-stabilized zirconia (YSZ) is applied in the solid oxide fuel cells (SOFCs) due to its high ionic conductivity. It is important to know the conductivity changes of cubic-YSZ vs. temperature and oxygen partial pressure, to control the function of the cells in all conditions. In this study, the CALPHAD (calculation of phase diagrams) approach is applied to calculate the oxygen vacancy concentration in different temperatures and oxygen partial pressures. By extracting the oxygen ion mobility from the literature, the ionic conductivity of cubic-YSZ will be calculated in a range of temperature and oxygen partial pressure. The experimental results from previous related researches will be used to validate the calculated results. Therefore, a map of ionic conductivity changes of cubic-YSZ will be prepared by which designing electrolytes for SOFCs will be more accurate.

4:10 PM

(ICACC-S10-006-2017) Genome-like Ion Parameters, Radii and Polarizability, For Predicting Temperature Dependent Material Properties

S. C. Tidrow*[1]; 1. Alfred University, USA

The "new" "simple" material model (NSMM), an extensible model, is reviewed and utilized to demonstrate superior performance over Goldschmidt's formalism for prediction of a wide range of Perovskite material properties. Through correlated temperature dependent genome-like ion parameters, radii and polarizability, the fidelity of NSMM is demonstrated through comparison with temperature dependent material properties of a significant number of "simple" "ideal" Perovskites and their solid solutions. Material properties and parameters including but not limited to lattice structure, lattice parameter, phase transition temperature, permittivity, Curie constant and Curie temperature, are discussed. The relationship between genome-like parameters, radii and polarizability, are used to demonstrate that the Curie and Curie – Weiss Laws, correlation relations, are incorporated within the fundamental Clausius – Mossotti permittivity or related Lorentz – Lorenz index of refraction equations. Further, it is shown that these genome-like ion parameters are directly related to Maxwell's conduction equation through the complex frequency dependent permittivity. Importantly, NSMM provides a space-time-energy mapping that can be utilized within more sophisticated algorithms to provide an even greater range of temperature dependent material properties.

4:30 PM

(ICACC-S10-007-2017) Computational Design: From a Simple Chemical Concept to 3D Topological Materials (Invited)

X. Chen*[1]; 1. Shenyang National Laboratory for Materials Science, Institute of Metal Research, Chinese Academy of Sciences, China

Within recent years, topological materials, including topological insulators, topological crystalline materials, topological (Dirac) semimetals, topological Wely semimetals, topological Wely line-node (or node-line) semimetal as well as topological metals, become extensively interesting not only for condensed matter physics and materials science but also for fundamental Dirac fermions. We have proposed a simple chemical consideration to design three-dimensional topological materials according to the electronegativity concept in combination with first-principles calculations. In this presentation, I will summarize what we have obtained for computational design of 3D materials from topological insulators to 3D Dirac/Weyl semimetal to 3D Dirac node-line in some ceramics.

5:00 PM

(ICACC-S10-008-2017) Accelerating Materials Design and Discovery using Computational Approaches: A Case Study in All-Solid-State Li-ion Battery (Invited)

Y. Mo*[1]; 1. University of Maryland, College Park, USA

First principles computation is a powerful technique in the design and discovery of new materials with little experimental input. In this presentation, I will share our success stories of leveraging first principles computation techniques in resolving a number of key material challenges in solid-state Li-ion batteries, a new battery technology with potentially intrinsic safety, high energy density, and enhanced cyclability. I will first present how we use first principles computation to design new solid electrolyte materials with enhanced properties, which are verified by multiple experimental studies. In addition, I will present our recently developed computational techniques for the design of heterogeneous interfaces in solid-state devices. These techniques are applied to resolve the problems, such as interface degradation and interphase formation, at the electrolyte-electrode interfaces in the solid-state batteries. In addition, the computation has been demonstrated to predict and suggest interfacial engineering strategies to resolve multiple interfacial issues in the example of solid-state batteries. Our computation methods for designing bulk-phase materials and solid interfaces are highly transferable to any materials system for a wide range of applications, paving the way for accelerated design of advanced materials.

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

Production Root Technology I

Room: Coquina Salon H
Session Chair: Jacob Jones, North Carolina State University

1:30 PM

(ICACC-S11-001-2017) Pulverization of nanoparticles using nanocomposite particles prepared by mechanical treatment (Invited)

J. Tatami*[1]; K. Jeong[1]; M. Iijima[1]; T. Takuma[2]; 1. Yokohama National University, Japan; 2. Kanagawa Academy of Science and Technology, Japan

Nanoparticles are being extensively investigated in industries and academia due to their widespread applications in various areas of science and manufacturing. Unfortunately, nanoparticles easily aggregate. This problem of nanoparticle aggregation creates challenges for various applications. We focus on using nanocomposite particles prepared via mechanical treatment to pulverize the aggregated nanoparticles. Y_2O_3 nanoparticles were used in this study. $CaCl_2$ and aggregated Y_2O_3 nanoparticles were mechanically treated using a process in which high shear and compressive stress are applied to them in order to generate nanocomposite particles. In order to rinse out $CaCl_2$, the nanocomposite particles were then added to distilled water to dissolve the $CaCl_2$ cores, and they centrifuged before supernatant solution was decanted. The recovered Y_2O_3 nanoparticles had a significantly smaller particle size and larger specific surface area than the aggregates. Thus, we confirmed that the aggregated Y_2O_3 nanoparticles were effectively pulverized in a short time by using nanocomposites prepared by mechanical treatment. Further, the relative density of the sintered body prepared from the pulverized Y_2O_3 nanoparticles was higher than that of the as-received and ball-milled Y_2O_3 nanoparticles.

2:00 PM

(ICACC-S11-002-2017) Advances in Nanocomposite Design: Towards Electronic and Biomedical Applications (Invited)

J. Andrew*[1]; 1. University of Florida, USA

In many single-phase materials certain properties are mutually exclusive. Examples of this property dichotomy include strength and toughness, high electric permittivity and high magnetic permeability, and soft and hard magnetic properties. Nanostructured composite materials have the potential to overcome some of these limitations of single-phase materials. For example, magnetic and ferroelectric materials can be combined on a single particle or fiber, yielding new nanostructured multiferroic composites with enhanced properties, opening up new applications including tunable microelectronics, sensors and dual-state memory. Here, we present the first example of composite nanostructured building block with a Janus-type morphology for multiferroic applications. By fabricating composites on a single particle or fiber in an anisotropic manner (e.g. Janus-type) the surface and bulk properties of each phase remain accessible, providing additional degrees of freedom in composite design. The results from magnetic, dielectric, and magnetoelectric measurements will be presented along with the effects of processing on the size, crystallinity and morphology of these novel composites.

2:30 PM

(ICACC-S11-003-2017) Processing and Integration Science of Capacitors Based on $Bi(M)O_3$-$BaTiO_3$ Dielectrics for High Field and/or High Temperature Operation (Invited)

G. L. Brennecka*[1]; M. Beuerlein[1]; H. Brown-Shaklee[2]; N. Raengthon[3]; N. Triamnak[4]; N. Kumar[4]; D. Cann[4]; 1. Colorado School of Mines, USA; 2. Sandia National Laboratories, USA; 3. Chulalongkorn University, Thailand; 4. Oregon State University, USA

A number of applications demand capacitors that can work at temperatures above 150C with higher capacitance, energy density, resistivity, and reliability at lower costs than currently-available options. Dielectrics based on $xBi(Zn_{0.5}Ti_{0.5})O_3 - (1-x)BaTiO_3$ and other members of the $xBi(M)O_3 - (1-x)BaTiO_3$ family (where M refers to a trivalent ion or trivalent-equivalent combination of ions) show very attractive properties for such operating conditions. Permittivity values stay high (e.g., >1000) over broad temperature ranges (e.g., >200C) and under extremely large operating fields (e.g., >300 kV/cm). Resistivity values are commonly orders of magnitude greater than other high-permittivity materials, and measurements of activation energies for charge transport are often half of the optical band gap, indicative of intrinsic conduction mechanisms. From an electrical standpoint, these materials appear to be quite homogenous with remarkably-low defect concentrations, but the microstructures are complex, with significant compositional heterogeneity within and among grains. Understanding the processing science of these materials, particularly the complex interplay among defect chemistry, diffusion kinetics, and properties, is crucial for fabrication of the desired low cost, high reliability capacitors.

Production Root Technology II

Room: Coquina Salon H
Session Chairs: Jennifer Andrew, University of Florida; Tadej Rojac, Jozef Stefan Institute

3:20 PM

(ICACC-S11-004-2017) Combination Effects of high-throughput bulk preparation system and high-pressure process (Invited)

K. Fujimoto*[1]; M. Gibu[1]; Y. Yamaguchi[2]; A. Aimi[1]; 1. Tokyo University of Science, Japan; 2. National Institute of Advanced Industrial Science and Technology (AIST), Japan

Combinatorial technology, which means high-throughput materials preparation and evaluation, is notably developing and promising the effective fast screening tool of novel functional materials by combination of computational chemistry and data analysis technique. We have hitherto established the combinatorial system for exploring functional oxides films or fine particles based on the solution process. Further, we have recently developed the tool for library preparation under high pressure (~200 MPa) and temperature (~500°C). We could have discussed the reactivity of $Mg(OH)_2$, $Al(OH)_3$ and AlOOH under high-pressure by using the above tool. Until now, we have some problem about the evaluation of physical property for obtained powder library. It was difficult to evaluate the electrical conductivity because the form of powder library was normally not sintered body. So, we tried to make the dense sintered powder library by combination of the combinatorial powder preparation system and the high-pressure combinatorial tool. For example, in case of 5% Bi substituted $CaMnO_{3-\delta}$, the electrical conductivity of the densified powder showed better value ($63\ S\cdot cm^{-1}$) than as prepared powder ($13\ S\cdot cm^{-1}$). Combination of combinatorial system and high-pressure process was effective method not only the reactivity of solid study, the exploration of high-pressure phase but also the densification of powder library.

3:50 PM

(ICACC-S11-005-2017) Processing issues in BiFeO3 ceramics and thick films (Invited)

T. Rojac*[1]; E. Khomyakova[1]; M. Makarovic[1]; J. Walker[2]; A. Bencan[1]; B. Malic[1]; 1. Jozef Stefan Institute, Slovenia; 2. Pennsylvania State University, USA

With hundreds of papers published annually, bismuth ferrite ($BiFeO_3$) has recently been the most investigated perovskite material, marking unprecedentedly the field of multiferroics. Despite the great interest in this material, it has been clear since the early studies in 1960's that $BiFeO_3$ presents a number of processing challenges, making it difficult to process the ceramics to high phase purity and controlled microstructure. In this contribution, we will present and explain in detail the major thermodynamic and kinetic issues responsible for the persistent appearance of non-perovskite, secondary phases both in chemically unmodified $BiFeO_3$ and complex $BiFeO_3$-based solid solutions. It will be shown that these key issues are associated with the presence of impurities and the likely formation of low-temperature melting Bi-rich phases, both of which should be carefully controlled to tailor the processing of $BiFeO_3$-based ceramics. In the last part of the contribution, we will give emphasis on the integration of $BiFeO_3$ thick films by the screen-printing deposition and identify processes, such as the interdiffusion and reactions between the thick-film-structure layers, which must be particularly considered in order to obtain high-quality $BiFeO_3$ thick films. We will present evidences of a self-poling effect in the films related to strain gradients, which develop during cooling from the Curie transition.

4:20 PM

(ICACC-S11-006-2017) Cold Sintering Process: A New Technique for Low-Temperature Processing of Ceramics and Ceramic-Based Composites (Invited)

H. Guo*[1]; A. Baker[1]; T. Bayer[1]; J. Guo[1]; S. Berbano[1]; S. Funahashi[2]; C. A. Randall[1]; 1. Pennsylvania State University, USA; 2. Murata Mfg. Co., Ltd, Japan

Research on sintering of dense ceramic materials has been very active in the past decades and still keeps gaining in popularity. Although a number of new techniques have been developed, the sintering process is still performed at high temperatures. Very recently we established a novel protocol, the "Cold Sintering Process (CSP)", to achieve dense ceramic solids at extraordinarily low temperatures of < 300°C. A wide variety of chemistries and composites were successfully densified using this technique. In this talk, an introduction to "Cold Sintering Process" will be delivered by employing ferroelectrics and ZrO_2-based materials as examples. Other materials we succeeded so far are also briefly outlined. Together with detailed experimental demonstrations, fundamental mechanisms, as well as the underlying physics from a thermodynamics perspective, are collaboratively comprehended. Such an impactful technique opens up a new way for cost-effective and energy-saving ceramic processing. We hope the CSP will provide a clear roadmap to guide future studies on ultralow temperature ceramic sintering or ceramic materials related integration.

4:40 PM

(ICACC-S11-007-2017) Temperature Sensor for Battery Management System in Electrical Vehicle

S. Mhin*[1]; 1. Korea Institute of Industrial Technology, Republic of Korea

New automotive technology, electrical vehicle (EV), expands its area in the automotive industry due to advantages such as effective cost, no gas and emission. With Li ion battery as a power source of the EV, battery management system (BMS) is an important component to manage voltage, temperature and current of the rechargeable battery. In order to protect the battery from safety issue relating with over and under-temperature, accurate and fast detection of temperature inside battery pack is key technology. One of the candidates for the temperature detection in the BMS is Negative Temperature Coefficient (NTC) thermistor. The NTC thermistor is semiconducting materials, which shows decrease of resistance with increasing temperature. For development of the NTC thermistor with high precision and good thermal response, influence of various processing route and chemical composition on electrical properties of $(Ni_x,Co_y,Mn_{3-x-y})O_4$ are investigated. Also, hopping mechanism of the $(Ni_x,Co_y,Mn_{3-x-y})O_4$ depending on the chemical composition and processing route is discussed.

5:00 PM

(ICACC-S11-008-2017) Design of Flexoelectric Piezoelectric Metamaterials of High Electromechanical Response (Invited)

B. Chu*[1]; W. Zhou[1]; P. Chen[1]; Q. Pan[1]; X. Zhang[1]; 1. University of Science and Technology of China, China

Flexoelectricity is the ability of a dielectric material to generate the electric polarization by a strain gradient (direct effect) or stress by an electric field gradient (converse effect). By exploiting this effect, flexoelectric piezoelectric composite (or piezoelectric metamaterial) can be designed. The composite metamaterial can exhibit an apparent piezoelectric response originating from the flexoelectric effect of specially designed gradient-generating structure or geometry although none of the constituents in the composite is required to be piezoelectric. In this presentation, several designs of piezoelectric metamaterials are demonstrated and the mechanisms of the apparent piezoelectric response are discussed. We show that by inducing a domed structure in a ferroelectric ceramic wafer through chemical reduction or diffusion, a strong apparent piezoelectric response comparable to conventional lead oxide-based piezoelectric materials can be achieved. The response can be sustained well above the Curie temperature of the ferroelectric ceramic. We provide strong evidence that the apparent piezoelectric response mainly originates from the flexoelectric effect of the ferroelectric ceramic. Our work provides a unique approach to designing high-performance electromechanical materials.

5:30 PM

(ICACC-S11-009-2017) Ceramic processing of tubular, multilayered oxygen transport membranes (Invited)

A. B. Haugen*[1]; S. Ovtar[1]; J. Gurauskis[2]; A. Kaiser[1]; R. Kiebach[1]; P. Hendriksen[1]; 1. Technical University of Denmark, Denmark; 2. Æneam IVS, Denmark

Pure oxygen gas supplied by ceramic oxygen transport membranes (OTMs) can facilitate reduced CO_2 emissions through more efficient gasification or combustion processes and easier CO_2 capture and storage. For maximum oxygen flux and 100% selectivity, the active OTM layer should be thin and dense, and have a large and catalytically activated surface area. These requirements call for an asymmetric OTM design with a thin, dense OTM layer (~15 μm) sandwiched between two porous catalyst layers (~15 μm) and mechanically supported on a porous ceramic substrate (~1 mm). This talk pertains to our work at the Technical University of Denmark (DTU) related to processing such multilayered ceramic components with a tubular geometry, focusing on scalable process technologies. This includes thermoplastic extrusion to shape the porous, tubular support, deposition of thin dense and porous layers by dip coating, co-sintering of these layers, and infiltration of electrocatalysts. Material and processing considerations for two different combinations of materials in the multilayered components will be discussed, and performance of these components under laboratory testing and integrated in pilot-scale biomass gasifiers will be presented.

S12: Materials for Extreme Environments: Ultrahigh Temperature Ceramics (UHTCs) and Nano-laminated Ternary Carbides and Nitrides (MAX Phases)

Materials Design, New Composition and Composites I

Room: Tomoka B
Session Chair: Michel Barsoum, Drexel University

1:30 PM

(ICACC-S12-001-2017) Zr-Based MAX Phases for Gen-IV Lead Fast Reactors (Invited)

K. Lambrinou*[1]; T. Lapauw[2]; B. Tunca[2]; R. Delville[1]; A. Weisenburger[3]; A. Jianu[3]; J. Vleugels[2]; 1. SCK-CEN, Belgium; 2. KU Leuven, Belgium; 3. Karlsruhe Institute of Technology, Germany

MAX phases are promising materials for Gen-IV lead-cooled fast reactors (Gen-IV LFRs), due to their stability in oxygen-poor lead alloys (e.g. lead-bismuth eutectic, LBE). Zr-rich MAX phases are interesting due to the small fast neutron cross-section of Zr. In the Zr-Al-C system, two MAX phases were synthesised, i.e. Zr_2AlC and Zr_3AlC_2, but producing phase-pure materials was challenging. The desired for fuel cladding applications phase purity needed to limit differential swelling by neutrons was achieved by adding select dopants to the starting powders. Adding elements to Zr-Al-C MAX phases to form solid solutions on both M and A sites affected their performance (e.g. oxidation resistance). XRD was employed to assess the effect of alloying on the MAX phase stability. The corrosion/erosion resistance of Zr_2AlC, Zr_3AlC_2 and $(Nb,Zr)_4AlC_3$ was assessed in oxygen-poor ($[O] < 10^{-8}$ mass%) LBE, at 500°C for 1000 hours, under both static and fast-flowing (> 10 m/s) conditions. The test results were compared to results of comparable tests on 316L stainless steel, a Gen-IV LFR candidate structural steel. The mechanical properties (four point-bending strength, fracture toughness) of $(Nb,Zr)_4AlC_3$, a promising material for pump impellers, were evaluated in inert atmosphere and LBE at 500°C to assess its susceptibility to liquid metal embrittlement. The interaction of Zr-based MAX phases with liquid LBE was studied by EPMA, EBSD, FIB and TEM.

2:00 PM

(ICACC-S12-002-2017) Novel Cr_2AlC MAX-phase/SiC fiber composites: Processing and properties

J. Gonzalez-Julian*[1]; J. Llorente[2]; M. Bram[1]; M. Belmonte[2]; O. Guillon[1]; 1. Forschungszentrum Juelich, Germany; 2. Institute of Ceramics and Glass, CSIC, Spain

Cr_2AlC is one of the most promising MAX phases for high temperature application (> 1000°C) due to its excellent oxidation and corrosion resistances, as well as other features such as high thermal and electrical conductivities. Incorporation of fibers to improve properties – mainly the mechanical ones – has been extensively explored in advanced ceramic systems, leading to ceramic matrix composites (CMCs). However, investigations of CMCs based on MAX phases are very limited despite their large potential for high temperature applications. In this work, processing of the Cr_2AlC composites containing SiC fibers and their mechanical and tribological response are presented. Cr_2AlC phase was synthesized from its elemental constituents and mixed with different contents (5 and 10 wt.%) of SiC fibers. Afterwards, the composites are fully densified using Field Assisted Sintering Technique/Spark Plasma Sintering (FAST/SPS). Regarding the properties of the developed composites, mechanical properties were characterized by Vickers indentation and the tribological response by reciprocating ball-on-plate configuration under dry conditions.

2:20 PM

(ICACC-S12-003-2017) First-principles-based evaluation of Ti2AlC-Cr2AlC phase diagram

T. Duong*[1]; A. Talapatra[1]; W. Son[1]; M. Radovic[1]; R. Arroyave[1]; 1. Texas A&M University, USA

In this work, we report the first attempt to evaluate the finite-temperature pseudo-binary phase diagram of Ti2AlC-Cr2AlC through ab initio methods. In particular, first-principles calculations were conducted to estimate finite-temperature free energies of MAX and competing phases, taking into account both vibrational and electronic contributions. The calculated energies were in turn attributed to a linear minimization process to derive the most competitive phases at different composition and temperature conditions. The phase diagram was then constructed based on the results of the analysis of phase competition. It has been shown that the evaluated phase diagram is in reasonable agreement with previous experiments, albeit there still exists room for further refinements.

2:40 PM

(ICACC-S12-004-2017) Preparation and Properties of Ag/Ti_3AlC_2 composites as electrical contact materials

J. Ding*[1]; Z. Sun[1]; P. Zhang[1]; W. Tian[1]; M. Zhang[1]; Y. Zhang[1]; 1. Southeast University, China

In order to develop environmentally friendly substitutes for Ag/CdO electrical contact materials, Ag matrix composites with 10 mass% and 20 mass% Ti_3AlC_2 (Ag/TAC), were prepared by using pressureless sintering process. Ag/TAC composites with a relative density above 95% showed low electrical resistivity in the range of $5\sim18\times10^{-2}\mu\Omega m$. The Ti_3AlC_2 reinforcing phase was found, by SEM and OM, to disperse in Ag matrix uniformly. The arc erosion resistances of Ag/20TAC and Ag/10TAC contacts were tested under the condition of 100 A/400 V for 3000 times in commercial contactors, and compared with those of Ag/CdO commercial contact. Ag/20TAC contacts were found to suffer from serious arc erosion, by which both Ag matrix and TAC particles were severely eroded. In comparison, Ag/10TAC contacts exhibited a good resistance to arc erosion, where Ag matrix was protected well by TAC particles from arc erosion. The Ag/10TAC displayed contact properties superior to those of Ag/CdO commercial contact, suggesting this composite, with 21% Ti_3AlC_2 in volume fraction, is a promising substitute to Ag/CdO contact. The mechanisms for the better performance of the Ag/10TAC contact materials is discussed in terms of the wetting behavior and microstructural factors.

Materials Design, New Composition and Composites II

Room: Tomoka B
Session Chairs: Miladin Radovic, Texas A&M University;
Thierry Cabioch, University of Poitiers

3:20 PM

(ICACC-S12-005-2017) Out-of-plane Ordering in Mo-Containing Quaternary MAX Phases (Invited)

J. Halim*[1]; B. Anasori[2]; R. Meshkian[1]; M. Dahlqvist[1]; E. Moon[2]; J. Lu[1]; S. May[2]; E. Caspi[3]; L. Hultman[1]; P. Eklund[1]; J. Rosen[1]; M. W. Barsoum[2]; 1. Linkoping University, Sweden; 2. Drexel University, USA; 3. Nuclear Research Centre, Israel

A recent development in the MAX phase field is the existence of out-of-plane chemically ordered $(M',M'')_{n+1}AlC_n$ phases. In this study, we report on two such phases: Mo_2TiAlC_2 and $Mo_2Ti_2AlC_3$ where two Mo layers sandwich one or two Ti layers, respectively. The phases were synthesized by reacting elemental mixtures at elevated temperatures in argon flow. High resolution scanning electron microscopy (HR-STEM) accompanied with Energy-dispersive X-ray spectroscopy mapping was used to show that the ordered stacking sequence is Mo-Ti-Mo-Al-Mo-Ti-Mo for Mo_2TiAlC_2 and Mo-Ti-Ti-Mo-Al-Mo for $Mo_2Ti_2AlC_3$. Rietveld refinements of the

X-ray diffraction (XRD) patterns from Mo_2TiAlC_2 show that the outer M layer has a moderate intermixing of 75 and 25% of Mo and Ti atoms, respectively, while the inner layer is fully occupied by Ti atoms. In $Mo_2Ti_2AlC_3$ the same moderate intermixing in the outer layer (77 and 23% of Mo to Ti, respectively) is observed; in the inner layers the intermixing is less with 14% and 86 % of Mo to Ti, respectively. Thus in both cases, the major M element in the outer layers is Mo and the major M element in the inner layers is Ti. X-ray photoemission spectroscopy (XPS) analysis provides an insight on the chemical nature of the various elements and confirms the ordering in the M species. We also discuss replacing Ti with another M elements, to realize other novel MAX phases.

3:50 PM

(ICACC-S12-006-2017) Computational and experimental approach to structural, mechanical, and themodynamic properties of Ti3(SixAl1-x)C2 (Invited)

W. Son*[1]; H. Gao[1]; A. Talapatra[1]; T. Duong[1]; M. Radovic[1]; R. Arroyave[1]; 1. Texas A&M University, USA

We report on the structural, mechanical, and thermodynamic properties of Ti3SixAl1-xC2. The calculations were based on the Density Functional Theory (DFT), which is implemented in the Vienna Ab initio Simulation Package (VASP), and the solid solutions were modeled using special quasirandom structures (SQS), which is generated by the Alloy Theoretic Automated Toolkit (ATAT), and the experimental samples were synthesized from high purity powder precursors through spark plasma sintering (SPS). In particularly, we calculated lattice parameters, elastic modulus (Young's, bulk, and shear modulus), coefficient of thermal expansion (CTE), heat capacity, hardness, and stacking fault energy, and they show good agreement with experimental values. The lattice parameters, and elastic modulus follow monotonic behavior, which is calculated using the stress-strain approach, and experimentally determined using X-ray diffraction (XRD) and Resonant Ultrasound Spectroscopy (RUS). The hardness shows solid solution hardening effect, which is calculated under loading mode 1, and experimentally determined using micro-hardness tester. Lastly, the SFE shows monotonic or slight solid solution softening effec, which is calculated by applying the pure alais shear deformation.

4:20 PM

(ICACC-S12-007-2017) A Review of Different Types of MAX-based Composite Systems for Multifuncational Applications

J. Nelson*[1]; M. Olson[1]; F. Al-Anazi[1]; S. Ghosh[1]; S. Gupta[1]; 1. University of North Dakota, USA

It is well known that $M_{n+1}AX_n$ (MAX) phases (over 60+ phases) are thermodynamically stable nanolaminates displaying unusual, and sometimes unique, properties. These phases possess a $M_{n+1}AX_n$ chemistry, where n is 1, 2, or 3, M is an early transition metal element, A is an A-group element, and X is C or N. The MAX phases are highly damage tolerant, thermal shock resistant, readily machinable, and with Vickers hardness values of 2–8 GPa, are anomalously soft for transition metal carbides and nitrides. Recently, different studies are being performed to design MAX Phase based composites for different applications. In this presentation, we will present a comprehensive review of some of the recent studies on designing MAX phase reinforced composites.

4:40 PM

(ICACC-S12-008-2017) Synthesis and Characterization of a Nanolaminated Alumina-forming Boride: MoAlB

S. Kota*[1]; E. Zapata-Solvas[2]; A. Ly[3]; J. Lu[4]; O. Elkassabany[1]; A. Huon[1]; W. Lee[2]; L. Hultman[4]; S. May[1]; M. W. Barsoum[1]; 1. Drexel University, USA; 2. Imperial College London, United Kingdom; 3. Drexel University, USA; 4. Linkoping University, Sweden

Atomic-scale chemical layering in materials can induce exotic properties or enhance other performances. This type of nanolaminated structure is represented by the MAX phases, a family of ternary carbides and/or nitrides consisting transition metal carbide/nitride sublattice interleaved by monolayers of Group IIIA-VA elements, and leads to both ceramic-like and metallic properties. Yet, the properties of analogously layered transition metal ternary borides, TMBs, have not been studied extensively. Herein, we synthesize dense, predominantly single-phase samples of one such layered boride, MoAlB, for the first time. Atomically-resolved scanning transmission electron microscopy confirmed the atomic-scale layering consisting of a MoB-sublattice interleaved by bilayers of Al. Unique among TMBs, MoAlB forms a dense alumina scale when heated in air to temperatures up to 1400°C and the oxidation kinetics follow a cubic time dependence, like the MAX phase Ti_2AlC. The good match between the thermal expansion coefficients of MoAlB and alumina led to the oxide scale adhering well. Like other transition metal borides, MoAlB is an excellent electronic conductor (0.36-0.49 mW-m at 300 K) and thermal conductor (35 $Wm^{-1}K^{-1}$ at 300 K). Interestingly, MoAlB is mechanically soft (10.6 GPa) relative to other TMBs. These results warrant further study on the potential use of MoAlB as a high-temperature structural ceramic.

4:40 PM

(ICACC-S12-009-2017) Intrinsic Alloying Behavior in M and A Sublattices in 211 and 312 MAX Phases: Insights from Ab Initio Calculations (Invited)

R. Arroyave*[1]; A. Talapatra[1]; T. Duong[1]; W. Son[1]; H. Gao[1]; M. Radovic[1]; 1. Texas A&M University, USA

In this talk, I will present recent results in which the intrinsic alloying behavior of the M and A sublattices in some prototypical 211 and 312 MAX Phases has been investigated from first-principles. Specifically the energetics of the configurational space in these systems is explored through cluster expansions in which the energy of arbitrary configurations is parameterized in terms of their constituent pairs, triples, tetrahedra, etc. The study of close to 100 Al-containing pseudobinary systems required the DFT calculations of 5,000 structures. This extensive study has already yielded significant insights into the intrinsic alloying trends of several systems: while in some cases there are clear tendencies towards strong ordering, in other cases there are equally strong tendencies towards phase separation, with a sizable fraction of the systems studied exhibiting almost ideal mixing. Observed trends are rationalized in terms of the underlying changes in the electronic structure of the compounds. The observed mixing trends are also discussed in terms of their relevance for yet undiscovered MAX solid solutions. Finally, I will discuss (and will provide some examples) the types of analyzes––at the DFT level--that are necessary in order to provide synthesis pathways for promising MAX solid solutions.

S14: Crystalline Materials for Electrical, Optical and Medical Applications

Optical Material 1

Room: Tomoka C
Session Chair: Mariya Zhuravleva, University of Tennessee

1:30 PM

(ICACC-S14-001-2017) Chalcogenide glass and glass ceramics for integrated photonics (Invited)

J. Li[1]; G. Yin[1]; J. Michon[1]; Y. Huang[1]; Q. Du[1]; L. Li[1]; H. Lin[1]; A. Agarwal[1]; A. Yadav[2]; K. Richardson[2]; J. Hu*[1]; 1. Massachusetts Institute of Technology, USA; 2. University of Central Florida, USA

Chalcogenide glasses, namely the amorphous compounds containing S, Se and/or Te, represent an emerging class of optical materials suitable for on-chip photonic integration. Our prior work has demonstrated a series of photonic devices including spectroscopic chemical sensors and flexible optical systems capitalizing on the unique optical and processing characteristics of chalcogenide materials. These materials also exhibit controllable crystallization behavior, and the large optical property change accompanying the amorphous-crystalline phase transition can be utilized to enable "re-programming" of photonic circuits. In this talk, we will discuss our recent development of novel chalcogenide glass and glass ceramic materials as building blocks for tunable and reconfigurable photonic components.

2:00 PM

(ICACC-S14-002-2017) Single crystal architecture in glass (SCAG): New active metamaterials for photonics (Invited)

D. Savytskii[1]; K. Veenhuizen*[1]; S. McAnany[1]; B. Aitken[2]; D. Nolan[2]; V. Dierolf[1]; H. Jain[1]; 1. Lehigh University, USA; 2. Corning Incorporated, USA

Transforming glass into a single crystal by spatially selective heating with a laser offers a unique capability of fabricating complex SCAG in an otherwise inexpensive glass matrix, thereby facilitating new devices for integrated optics, optical computing, optical communication, high density optical memory, etc. Here we focus on the fabrication of SCAG, and its advantages over conventional crystal growth methods. Typically, absorption of CW laser of suitable energy is used for forming SCAG at or near the surface. This approach is extended to form 3D SCAG using pulsed fs laser of wavelength that is too long for normal absorption, but its intensity is sufficient to cause nonlinear absorption and heating at the finely focused point inside the glass. In either case the local heating facilitates nucleation of crystalline phase. Controlled translation of the laser can make the nucleus grow into a 1D single crystal line, a 2D layer at the surface by 'stitching' crystal lines, a 3D structure in the interior, etc. Doping the glass with rare earth introduces additional functionality in SCAG. The key challenge for fabrication is how to grow the initial nucleus into a single crystal of desired geometry without forming additional nuclei. Additional challenges appear when the composition of desired crystal phase is diffrent from that of glass, requiring interdiffusion of species as the growth proceeds.

2:20 PM

(ICACC-S14-003-2017) Densification and translucency in RE:Y_2O_3 + MgO nanocomposites for mid-infrared solid-state lasers

V. L. Blair*[1]; N. Ku[1]; Z. Fleischman[2]; L. D. Merkle[2]; 1. US Army Research Laboratory, USA; 2. US Army Research Laboratory, USA

Several challenges exist for mid-infrared laser materials, including 1) the need to dissipate heat generated in lasing; 2) luminescence quenching by multi-phonon relaxation, and 3) 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. A simple co-precipitation technique has been developed to produce intimately mixed nanopowders in situ. These powders compare well with commercially available ceramics (including their Er spectroscopy), even when mixed as a composite, and can be air-fired to ~96% of theoretical density, yielding translucent parts. In this presentation, results of hot pressing the composite powders with varying ratios of MgO/Er:Y_2O_3 will be shown. As the amount of Er:Y_2O_3 increases and the number of scattering sites start to coalesce into large patches, the translucency decreases. If the amount of Er:Y_2O_3 is sufficiently small and dispersed, the yttria grains will be pinned as individuals in a sea of MgO, leading to optimal translucency.

2:40 PM

(ICACC-S14-004-2017) Doping erbium into the lattice of aluminum oxide

N. Ku*[1]; V. L. Blair[1]; A. Rahane[3]; V. Kumar[3]; J. Synowczynski-Dunn[1]; C. Moorehead[2]; R. E. Brennan[1]; 1. U.S. Army Research Laboratory, USA; 2. Drexel University, USA; 3. Vijay Kumar Foundation, India

Alumina offers potential advantages over traditional laser host materials due to its relatively high thermal conductivity. This would enable higher laser output by allowing heat to dissipate quickly to lower the operating temperatures. As a result, it could help to avoid thermal shock of the host material, which is a common cause of failure. In order to effectively replace the current Nd:YAG material with alumina, rare earth cation dopants are important to include. However, the large size mismatch between the Al and rare earth cation necessary for lasing presents problems in the doping of bulk alumina. Previous work has shown that erbium can be successfully doped into alumina by a precipitation process to produce doped powder. This work will investigate the solubility limit of erbium into the alumina lattice using a similar method. Concentrations of the Er range from 0.04 atom% up to 0.6 atom%. The use of co-dopants to increase the solubility limit of erbium will also be studied. These will include Mg and Ca, as DFT modelling has shown that these additives reduce the strain in the lattice caused by the larger Er dopant. This is achieved by increasing the population of vacancy defects within the alumina lattice.

Piezo/Ferro

Room: Tomoka C
Session Chairs: Zuo-Guang Ye, Simon Fraser University; Elvira Fortunato, FCT-UNL

3:30 PM

(ICACC-S14-005-2017) Progress in Growth and Characterization of $Pb(Zr_{1-x}Ti_x)O_3$ [PZT] Single Crystals as High-T_C and High-Performance Piezoelectrics (Invited)

Z. Ye*[1]; 1. Simon Fraser University, Canada

Lead zirconate-titanate $Pb(Zr_{1-x}Ti_x)O_3$ (PZT) has been the most extensively used piezoelectric material in the form of ceramics and thin films due to its good electromechanical properties. Here, we report on recent progress in the growth and characterization of the PZT single crystals. PZT single crystals with a wide composition range across the morphotropic phase boundary (MPB) were successfully grown by a top-seeded solution growth technique. The growth conditions are optimized in terms of the chemical, thermodynamic and kinetic parameters. The properties qualify PZT single crystals a high-performance and high-T_C piezoelectric material useful for high-temperature and high-power electromechanical transducer applications. On the other hand, by advanced characterization and analytical techniques, such as piezoresponse force microscopy, spherical aberration-corrected transmission electron microscopy, neutron total scattering and diffuse scattering and pair-distribution function (PDF) analysis, we have gained new insights into the complex local structure, atomic scale polarization rotation, nano-scale domain structure of PZT, which have provided a better understanding of the relationship between micro-/nanoscopic structure and macroscopic functional properties for this important material, but also for the piezo-/ferroelectric materials in general.

4:00 PM

(ICACC-S14-006-2017) New Challenges for Metal Oxide Nanoparticles: From Optical Sensors to Biosensors (Invited)

E. Fortunato*[1]; R. Martins[1]; 1. FCT-UNL, Portugal

In this paper we refer the work performed within i3N/CENIMAT in the area of functional metal oxide nanoparticles to be used in a wide range of applications, from optical sensors to biosensors. In these work we will refer to the use of a very simple synthesis process based on the hydrothermal method to produce ZnO, WO3 and VO2 nanoparticles, that will be used in optical sensors, biosensors and as thermochromic materials, respectively. During the presentation real examples of applications will be given.

4:30 PM

(ICACC-S14-007-2017) Influence of oxygen partial pressure and Al content on the resistivity and piezoelectric properties of $Ca_3TaGa_{3-x}Al_xSi_2O_{14}$ single crystals

X. Fu*[1]; E. Víllora[1]; Y. Kitanaka[2]; Y. Noguchi[2]; M. Miyayama[2]; K. Shimamura[1]; N. Ohashi[1]; 1. National Institute for Materials Science (NIMS), Japan; 2. The University of Tokyo, Japan

Piezoelectric langasite $Ca_3TaGa_{3-x}Al_xSi_2O_{14}$ (CTGAS) single crystals are are attracting much attention for high temperature sensor applications. So far, the growth of CTGAS with high Al concentration has been reported to be difficult. In this work, CTGAS (x=0-3) single crystals are successfully grown by Czochralski method. The temperature dependence of resistivity and piezoelectric properties are systematically investigated for the first time as a function of the Al content and the oxygen partial pressure during growth. The growth of CTGAS under various oxygen partial pressures indicated that, although the piezoelectric properties were invariant, the resistivity was remarkably higher the lower the oxygen partial pressure. The effect of Al content was also studied. $Ca_3TaGa_3Si_2O_{14}$ exhibits the lowest values for the d_{11} and the resistivity. By the gradual substitution of Ga by Al, both parameters tend to enhance continuously, reaching the highest resistivity and d_{11} for the fully substituted $Ca_3TaAl_3Si_2O_{14}$. In conclusion, the current high resistive $Ca_3TaAl_3Si_2O_{14}$ is the most promising single crystal among the langasite family for HT piezoelectric applications. This work has been partially supported by the Ministry of Education, Culture, Sports, Science and Technology (MEXT) Element Strategy Initiative to Form Core Center of Japan.

4:50 PM

(ICACC-S14-008-2017) A study on temperature insensitive relative permittivity in $Ba\{[Ga_x,Ta_x]Ti_{(1-2x)}\}O_3$ with different concentration of dipole-like dopant substitution

V. Kaliyaperumal Veerapandiyan*[1]; W. A. Schulze[1]; S. Misture[1]; S. M. Pilgrim[1]; D. M. Potrepka[2]; F. J. Crowne[2]; A. Tauber[3]; S. C. Tidrow[1]; 1. Kazuo Inamori School of Engineering, Alfred University, USA; 2. U.S.Army Research Laboratory, Sensors Electron Devices Directorate, USA; 3. As contracted to the U. S. Army Research Laboratory from Geo-Centers Inc.;presently retired, USA

$Ba\{[Ga_x,Ta_x]Ti_{(1-2x)}\}O_3$ materials with x values of 0, 0.005, 0.01, 0.025, 0.05, 0.075 and 0.1 have been prepared by conventional solid-state reaction. Interestingly, $Ba\{[Ga_{0.05,}\ Ta_{0.05}]Ti_{0.9}\}O_3$ presents a flat relative permittivity (ε_r) curve with a ε_r value of 700±16% and dissipation factor <0.1 at 10 kHz on the temperature range [-75°C, 120°C] as well as demonstrates electric-field tuning over the same temperature range. Therefore, concentration dependent charge compensated dipole-like dopant substitutions were investigated to better understand the postulated dipole-like effects on the dielectric properties of the parent material. Specifically, weak-field dielectric measurements have been conducted over the temperature range [-75°C, 800°C] in decade steps [100 Hz, 1 MHz]. In addition, in-situ XRD, including Rietveld refinement has been performed to determine the lattice parameters, coefficient of thermal expansion and phase transition temperature(s) for the temperature range [RT, 1000°C]. Designed ε_r, through varying the concentration of dipole-like substitution, is explored in an effort to increase the capacitance of the material while maintaining the trend of temperature insensitive ε_r.

5:10 PM

(ICACC-S14-009-2017) Epitaxial thin films of ε-Fe_2O_3: A new multifunctional "super-oxide"?

L. Corbellini*[1]; C. Harnagea[1]; C. Lacroix[2]; D. Ménard[2]; A. Pignolet[1]; 1. INRS, Canada; 2. Ecole Polytechnque de Montreal, Canada

ε-Fe_2O_3 is a metastable phase of iron(III)oxide, intermediate between γ- and α-Fe_2O_3. ε-Fe_2O_3 has been investigated mainly because of its magnetic properties, in particular its large magnetic anisotropy resulting in (i) a gigantic coercive field (20 kOe for nanoparticles), and (ii) a ferromagnetic resonance frequency in the low THz range under no applied magnetic field and at RT, which could find potential use in short-range Wi-Fi communications and ultrafast memories. However, given its polar crystal structure, it exhibits pyroelectricity, potentially making it one of the few single phase RT multiferroic materials if its polarization will prove to be switchable. Finally, it has been recently shown how nanostructured ε-Fe_2O_3 can achieve high hydrogen production rates, with performances superior than the one of commonly investigated α-Fe_2O_3. Stabilization of ε-Fe_2O_3 has been obtained mainly by synthesizing it by sol-gel as size-confined nanoparticles embedded in a SiO_2 matrix. Recently however, deposition of epitaxial thin films of ε-Fe_2O_3 as also been shown. Such ability is a key feature towards the development of integrated devices. We report the growth by PLD of epitaxial thin films of ε-Fe_2O_3 and ε-$Al_xFe_{2-x}O_3$ on different single crystal substrates, both oxides (STO, LAO, and YSZ) and non (Si), and discuss the influence of both the substrate and of the Al doping on the structural and functional properties.

5:30 PM

(ICACC-S14-010-2017) Dielectric behavior and electromechanical coupling of Lanthanum modified Strontium Bismuth Titanate for tranducer applications

S. Pasala*[1]; 1. Vardhaman College of Engineering, India

Lanthanum modified Strontium Bismuth Titanate ceramic $SrBi_{3.075}La_{0.025}Ti_4O_{15}$ (SBLT) is synthesized using solid state double sintering method. Single phase formation is confirmed by XRD. SEM micrographs reveal plate like morphology with around 2-3 μm grain size. All peaks in the EDAX pattern matched the stoichiometry of SBLT. Dielectric constant is measured from 50°C to 600°C in frequencies ranging from 1 Hz to 1 MHz. The Curie temperature is found to decrease with addition of Lanthanum. Impedance studies were carried out using a Wayne Kerr LCR meter. Cole-Cole plots are show semicircles in the temperature range 450°C to 600°C. Relaxation time decreased with increase of temperature. SBLT electromechanical coupling factor is calculated from the resonance and anti-resonance frequency. This material is found to have stable behavior up to 500°C thus making the material suitable for transducer applications at high temperatures.

S15: Additive Manufacturing and 3-D Printing Technologies

Selective Laser Processing

Room: Coquina Salon B
Session Chair: Soshu Kirihara, Osaka University

1:30 PM

(ICACC-S15-001-2017) Additive Manufacturing in High-Value Added Ceramic Products Manufacturing Technologies (Invited)

T. Ohji*[1]; 1. National Institute of Advanced Industrial Science and Technology (AIST), Japan

"High-Value Added Ceramic Products Manufacturing Technologies" project has been conducted since 2014 as a part of Strategic Innovation Promotion Program under the sponsorship of the Japanese government, with target of "removing restrictions of conventional manufacturing". This project deals with two principal technologies; additive manufacturing of ceramics aimed for complex shaping, reducing working-processes, saving cost, etc. and hybrid ceramic coating on 3D polymers/metals for enhancing their reliability and functionality. This paper will introduce the backgrounds, objectives, structure, research contents, research schemes, innovation strategy of this project, as well as the latest research achievements. Particular focus is places on the R&Ds on the additive manufacturing, which contains ceramic powder layer manufacturing (powder bed fusion, or indirect selective laser sintering), slurry layer manufacturing (stereolithography) and ceramic laser sintering (direct selective laser sintering) which realizes concurrent forming and sintering. 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)".

2:00 PM

(ICACC-S15-002-2017) 3D printed monolithic catalysts: Development and performance

M. Schwentenwein*[1]; S. Schuh[3]; R. Koopmans[3]; C. Scharlemann[2]; Y. Batonneau[4]; C. Maleix[4]; R. Beauchet[4]; J. Homa[1]; 1. Lithoz GmbH, Austria; 2. University of Applied Sciences Wiener Neustadt, Austria; 3. Forschungs- und Technologietransfer GmbH, Austria; 4. Université de Poitiers, France

Current manufacturing methods for catalysts are still in general 2D based processes such as extrusion with limited design freedom and do not allow the implementation of existing considerations for the improvement of catalyst design and performance. Due to recent developments in the area of additive manufacturing it is now possible to allow 3D design freedom and therefore enable the implementation of new design features to improve performance as well as lifetimes of catalysts. This contribution discusses the manufacturing of 3D printed catalysts using the lithography-based ceramic manufacturing (LCM) process to produce monolithic structures based on various ceramic materials such as alumina, cordierite, magnesia and silicon nitride. Using this lithographic process allows the fabrication of highly detailed structures with defined feature sizes down to 150 μm. Such catalysts provide a high surface to volume area and significantly reduce the pressure drop standard pellet type catalysts suffer from. Moreover, it was shown, that the (thermo)mechanical properties of the printed ceramic materials are similar to/same as conventionally processed ceramics. This also underlines the potential for a future use of 3D printed catalysts, especially in the field of launcher and satellite propulsion where a significant need for capable catalytic solutions exists.

2:20 PM

(ICACC-S15-003-2017) Deformation Behavior of Low Temperature Borosilicate and Aluminophosphate Glass Systems

W. Yu*[1]; F. Wu[1]; 1. National United University, Taiwan

Low temperature glass systems, including borate-, silicate-, and phosphate-based and their mixed types, are frequently adopted for versatile applications in optical fibers, molding glass, and high efficiency lighting encapsulation. The motivation of present study is to understand the softening, wetting, deformation and rheological behavior of the low temperature glass systems under various thermal histories. A Zn and Bi co-doped borosilicate and a Na and Li co-doped aluminophosphate glass systems are fabricated as targets. The borosilicate glass system exhibits a transition and a softening temperature less than 483 and 524°C, while the aluminophosphate system possesses an even lower temperature of 362 and 392°C, respectively. Powders of the two glass systems are mixed and heat-treated under various thermal profiles from 340 to 590°C. The aluminophosphate glass softens and wets the borosilicate powders thoroughly at 540°C. With higher temperature of 560°C, the borosilicate starts to soften and generates a glass mixture with aluminophosphate glass powders. Microstructure evolution and the softening deformation behavior are also discussed.

2:40 PM

(ICACC-S15-004-2017) Laser Shock Processing of Ceramics for Improved Cracking Resistance

B. Cui*[1]; F. Wang[1]; C. Zhang[2]; Y. Lu[2]; M. Nastasi[1]; 1. University of Nebraska, Lincoln, USA; 2. University of Nebraska–Lincoln, USA

Laser shock processing (LSP) is a novel surface modification process which is traditionally applied to metals. This study applied the LSP process to ceramics such as alumina, and aims to understand the fundamental processing-microstructure-property relationship of alumina ceramics in this process. LSP utilizes high-energy laser pulses to irradiate the ceramic surface, which generates shock waves that can penetrate into a depth of more than 1 mm and modify the subsurface microstructures. X-ray characterizations revealed that the compressive residual stresses induced by LSP can extend to a depth of 1.4 mm from the surface. Microstructural characterizations suggested that the elastic deformation dominates the mechanical response of alumina ceramics to laser-driven shock waves. However, intergranular fractures occasionally occurred which showed a limited plastic deformation in the vicinity of grain boundaries. The presence of compressive residual stresses improved the resistance of alumina ceramics to indentation cracking.

Ink Jet Printing

Room: Coquina Salon B
Session Chair: Martin Schwentenwein, Lithoz GmbH

3:20 PM

(ICACC-S15-005-2017) Additive Manufacturing (3D Printing) of Ceramics: Microstructure, Properties, and Product Examples

P. Karandikar*[1]; M. Watkins[1]; A. McCormick[1]; B. Givens[1]; M. Aghajanian[1]; 1. M Cubed Technology, Inc., USA

Additive manufacturing offers significant advantages (over conventional processing) including enabling design freedom, complex shape capability (e.g. cooling channels), near-net shape capability, the resultant ability to eliminate joining and reduce part count, elimination of some machining, reduced raw material consumption, reduced tooling cost, reduced prototyping and production time, and overall lower cost. However, several challenges have to be overcome to make additive manufacturing a viable production process including achieving the same composition, material properties (static, dynamic, mechanical, physical, chemical, electrical, etc.), isotropy of properties, and overall production cost. Also, for some simple shapes (e.g. blocks, ingots) and extremely high volume (100s of thousands/month) small parts, other processes can be more cost effective. In this work, microstructure and properties of additively manufactured (3D printed) reaction bonded (RB) ceramics are presented. The ability to systematically vary process parameters to produce RB ceramics of varying compositions and properties is demonstrated. The properties and microstructures of additively manufactured RB ceramics are compared with the properties and microstructures of the RB ceramics made by conventional processing. Finally, the ability to produce complex-shaped components is demonstrated with some examples.

3:40 PM

(ICACC-S15-006-2017) Effect of additives in oxychloride cement used for the production of lightweight material by high-speed 3D printing

D. Cabiddu*[1]; V. M. Sglavo[1]; 1. University of Trento, Italy

In the present work, the effect of some specific additives in oxychloride cement used as binder for the production of lightweight inorganic by powder-bed inkjet-3D printing is studied. The 3D printing technology considered here consists in the deposition of successive layers of a powder mixture and in the deposition of a magnesium chloride solution by ink-jet nozzles. The powder mix is preliminary produced starting from foamed glass, sands, magnesium oxide and additives selected among calcium oxide, calcium hydroxide, aluminum phosphate and methylcellulose. The inert phases are therefore bonded by the formation of oxychloride cement after the 3D printing process. Samples are produced by using constant processing parameters (thickness of the powder bed, ink-jet printing velocity, curing time). The effect of the additives is analyzed in terms of final resolution, microstructure and composition, density, mechanical strength and water absorption and resistance. It is shown that the final properties of the printed samples strongly depend on the used additive and its relative amount: for example, calcium oxide and calcium hydroxide accelerate the setting time of the cement and increase the final resolution of the samples, as well as methylcellulose does; aluminum phosphate reduces the strength of the samples but increases the retained strength after immersion in water.

4:00 PM

(ICACC-S15-007-2017) Investigation of the Binder Jetting of Agglomerated Ceramic Powders on the Resulting Sintered Density

K. Myers*[1]; 1. rp+m, USA

Agglomerated powders have been used in the injection molding industry for years to take advantage of the high surface area of sub-micron powders that do not flow well, but have great sintering characteristics. The density of the green bodies of injection molded parts are similar to those manufactured from binder jetting additive manufacturing technique. Currently, binder jetting of metal and ceramic powders require a densification step such as mechanical pressing, infiltration, or hot isostatic pressing to achieve sintered densities over 95%. This research, investigates the use of agglomerated ceramic powders are as a densification technique for the binder jetting platform. Polyvinyl alcohol, dextrin, and a phenolic resin were used as the agglomerating binders for sub-micron silica (SiO_2) and silicon carbide (SiC) powders.

Stereolithography

Room: Coquina Salon B
Session Chairs: Tadachika Nakayama, Nagaoka Univ of Tech; Surojit Gupta, University of North Dakota

4:20 PM

(ICACC-S15-008-2017) Fabrication of nanosized 3D structure by two photon absorption lithography and motion control by DC and Nano Pulsed Electric Field

T. Nakayama*[1]; M. Herrera Salazar[1]; N. Matsutani[1]; T. Suzuki[1]; H. Suematsu[1]; K. Niihara[1]; 1. Nagaoka Univ of Tech, Japan

In this project we have developed microstructures using two photon polymerization process, in which two-photon are absorbed simultaneously by the material using an ultrafast laser causing its polymerization. The microstructures fabricated were observed by confocal microscope, this microstructures can be moved by an electric field; the experimental setup used consists in two electrodes (anode and cathode), and a high-voltage source. The voltage applied is 0.5 to 1 kilovolts, the electrons enter in the microstructure through the cathode and get out by the anode; the space between electrodes is 150 to 300 micrometers, this area is filled with a liquid polymer, YE5822 (A). Time staying in the electrode was longer when the move in DC only. By overlapping nanosecond pulsed electric field and a DC electric field, it is possible to move the nanostructure as soon as pressing the switch.

4:40 PM

(ICACC-S15-009-2017) 3D Macro- and Micro- Additive Manufacturing of ceramics from preceramic polymers

J. E. Schmidt*[1]; G. Della Giustina[1]; L. Brigo[1]; P. Colombo[1]; G. Brusatin[1]; 1. University of Padova, Italy

Ceramic structures for various applications were fabricated by additive manufacturing using stereolithography (SLA) and 2-Photon polymerization (TPP) of preceramic polymer solutions. Commercially available polysiloxanes were selected, physically combined and photocrosslinked using the visible light range of the SLA printer for macrofabrication and the IR-wavelength of the TPP printer for microfabrication. Compatible polysiloxanes were combined with one material possessing the necessary photocrosslinkable groups for fabrication and the other a high ceramic yield. The printing process and solution were optimised regarding printing parameters and additives like solvent, photoinitiator and photoabsorber to print 3D structures with accurate geometry in an appropriate time frame as well as preserve the shape after pyrolysis. We present the SLA and TPP fabrication of SiOC ceramic macro- (heights up to several cm) and micro-fabricated parts (features down to hundreds of nm) starting from commercially available preceramic polysiloxanes. Complex ceramic structures, with a chemical composition which is not realizable other than with the preceramic polymer route, were manufactured with ceramic yields up to 47 wt%. In comparison to other additive manufacturing techniques, SLA offers higher resolution and the possibility of achieving dense ceramic parts, while sub-micron resolution can be demonstrated for TPP.

5:00 PM

(ICACC-S15-010-2017) Comparison of Dynamic Mask- and Vector-based Ceramic Stereolithography

S. Baumgartner*[1]; J. Schönherr[1]; M. Pfaffinger[1]; J. Stampfl[1]; 1. TU Wien, Austria

Additive manufacturing (AM) has developed into a promising technology allowing the parallel production of several complex parts with high resolution. Especially AM of ceramics shows great potential in the field of medical and dental applications where personalized aesthetic restorations are demanded. Accuracy is therefore a must to meet the high standards of those fields. At TU Wien we developed a special lithography based manufacturing technology where layer-by-layer a photosensitive slurry with ceramic filler is cured. To get dense ceramics the so called green body is then debinded and sintered. Two printing systems were evaluated: The first system uses a Digital Light Processing (DLP) approach, where a digital mirror device projects visible light (460nm) and triggers polymerization. A second system is based on a diode-laser with 405nm wavelength. In this case a galvanoscanner is used for structuring. The second systems enables a feature resolution down to 20µm. The aim of this study is to compare both of this technologies for multiple ceramics such as aluminium oxide, zirconium oxide, bioglass® and tricalcium phosphate. In order to get the best accuracy, printing parameters such as laser speed, hatching style, exposure time and intensity are varied.

5:20 PM

(ICACC-S15-011-2017) Tribology of Ti3SiC2 Reinforced Polymer Matrix Composites (PMCs) fabricated by Additive Manufacturing

R. Dunnigan[1]; M. Fuka[1]; S. Gupta*[1]; 1. University of North Dakota, USA

It is well known that the manufacturing process where thin layers of material are deposited, one after another, until the designed component is created is collectively referred to as Additive Manufacturing (AM). Fused deposition process (FDP) and Stereolithography (SL) are two types commonly used AM techniques. Currently, complex structures are being developed by FDP and SL. For further deployment in the field, especially for turbomachinery applications, it is critical to understand the tribological behavior of these solids. In this presentation, we will report the tribological behavior of Ti3SiC2 reinforced polymer matrix composites fabricated by FDP and SL. These results will be compared with the base polymer. During this study, the tribological behavior of all the samples will be evaluated with tab-on-disc method.

5:40 PM

(ICACC-S15-012-2017) Additive manufacturing of dense and strong silicon nitride-based ceramics

M. Schwentenwein*[1]; A. A. Altun[1]; B. Melrose[2]; N. Fecitt[2]; J. Homa[1]; 1. Lithoz GmbH, Austria; 2. International Syalons, United Kingdom

Due to problems associated with light absorption lithography-based additive manufacturing (AM) processes for ceramic materials have been primarily focusing on oxide ceramics light in color. This contribution presents recent results concerning AM of silicon nitride-based ceramics (SiAlONs). By using the so-called Lithography-based Ceramic Manufacturing (LCM) process it was possible to print and sinter silicon nitride-based compounds with the same mechanical properties as in conventional manufacturing. With a relative density of 99.8%, a hardness of 1500.5 and a biaxial bending strength of 763 MPa the tested composition is exactly at eye-level with its conventionally processed analogue (hardness of 1500 and biaxial bending strength of 770 MPa). These material properties in combination with the high precision of the LCM process allow the fabrication of highly complex objects that have not been feasible before and that are fully functional. Beside the printing process and the properties of the ceramic material, this presentation will also focus on first case studies using 3D printed silicon nitride parts to demonstrate the capability of this new manufacturing approach but will also highlight current boundaries and limitations of this technology.

6:00 PM

(ICACC-S15-013-2017) Stereolithographic Additive Manufacturing of Bulky Ceramic Components with Micro Geometric Structures to Modulate Energy and Materials Streamlines

S. Kirihara*[1]; 1. Osaka University, Japan

In stereolithographic additive manufacturing, 2D cross sections were created through photo polymerization by UV laser drawing on spread resin paste including ceramic nanoparticles, and 3D composite models were sterically printed by layer lamination. The lithographic system has been developed to obtain bulky ceramic components with functional geometries. An automatic collimeter was newly 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 of the 3D printing, nanometer sized ceramic particles were dispersed in to photo sensitive liquid resins from 40 to 60 % 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 10μm in layer thickness by a mechanically moved knife edge. An ultraviolet laser beam of 355 nm in wavelength was adjusted from 10 to 300 μm in variable diameter and scanned on the pasted resin surface. Irradiation power was changed automatically from 10 to 200 mW for enough solidification depth for 2D layer bonding. The created 3D composite precursor was dewaxed and sintered in an air atmosphere to obtain full ceramic components.

Tuesday, January 24, 2017

3rd Pacific Rim Engineering Ceramics Summit

Current Trends and Future Directions II

Room: Coquina Salon C
Session Chairs: Junichi Tatami, Yokohama National University; Hui Gu, Shanghai University

8:30 AM

(ICACC-PACRIM-008-2017) Novel concepts to design tough and reliable ceramic devices (Invited)

R. Bermejo*[1]; Y. Chang[2]; R. Danzer[1]; G. L. Messing[2]; 1. Montanuniversitaet Leoben, Austria; 2. Pennsylvania State University, USA

The design of ceramic layered composites with weak or strong interfaces can affect the propagation of surface cracks during external loading. The former approach aims to induce crack deflection, thus increasing the failure resistance of the composite. The latter uses residual compressive stresses to arrest or hinder the propagation of cracks. For practical applications, the design of layered-based structural and/or functional ceramic components often requires strong interfaces to guarantee the functionality of the device. In this paper, novel concepts are presented which combine different approaches used in layered architectural design with strong interfaces aiming to obtain highly reliable ceramic materials with high 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 a minimum design strength for the material, below which failure cannot take place. In addition, 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.

9:00 AM

(ICACC-PACRIM-009-2017) Development of Pin-point Controlled 3-Dimensional structure using Switching Nano-pulse Electric Field (Invited)

T. Nakayama*[1]; H. Cho[2]; T. Suzuki[1]; H. Suematsu[1]; K. Niihara[1]; 1. Nagaoka Univ of Tech, Japan; 2. Hanyang Univ, Republic of Korea

BN nanosheets with flake-like structure are exfoliated and the surfaces are modified in order to enhance the filler-to-filler gaps narrower than 1 nm during application of electric field. An electrode with micro-protrusions on the surface is designed by a simulator, MAGIC, followed by fabrication using electron beam lithography. The localization points of linear densely packed BN nanosheets (LDPBNs) on the surface of the electrode are controlled by different design of protrusions, which minimizes the effect of electric field concentration on the tips. A device generating a nano-pulse electric field is used for application of high electric field by overcoming the limitation of breakdown voltage. 3-dimensional analysis of internal structures of the hybrid film is performed by X-ray CT Scanning. The LDPBNs assembly of would be noticeably effective for heat dissipation through out-of-plane of the hybrid films, if the structure anchors the film planes like bridges in the hybrid film, and may pave a way for strategy to solve thermal build-up as next generation technology in the field of modern electronic, optoelectronic, photonic devices and systems of semiconductor industry as a thermal conduction circuits with electric insulation property.

9:30 AM

(ICACC-PACRIM-010-2017) Transient intergranular glassy film to initiate bi-modal microstructure in engineering ceramics (Invited)

H. Gu*[1]; 1. Shanghai University, China

Additive to control microstructure is ideal for ceramists. It is often found additives remain at two-grain junctions to form intergranular glassy film (IGF), especially in Si_3N_4 where IGFs are ubiquitously present while an equilibrium width was repeatedly observed. Such IGFs are independent to crystallographic orientations, hence brought important influences on sintering behaviors and mechanical properties. Recently, IGFs were correlated to abnormal grain growth in Al_2O_3 ceramics via transitions between complexions, or stable grain-boundary phases, adding a strong thermodynamic nature for IGF. However, the chemical analyses reveal different scenarios: IGF is sensitive to grain surface and to neighboring multi-grain junctions. In low-CaO-doped Si_3N_4, basal/non-basal facets exhibit bi-modal dopant segregation in IGF regardless doping level, leaving extra CaO re-partitioned in glassy pockets, followed by abnormal grain growth. In SiC and Sialon ceramics, IGFs were found subject to kinetic processes. In low-TiO_2-doped Al_2O_3 ceramics with minor SiO_2 impurity, preferential segregation of SiO_2 and TiO_2 to basal and non-basal IGFs were correlated with a transient steps of SiO_2 at basal facets to enable abnormal growth on non-basal facets. The transient nature for IGF indicates the equilibrium width is purely a local structural issue.

10:20 AM

(ICACC-PACRIM-011-2017) Functionalization and design of advanced ceramics based on innovative powder processing (Invited)

J. Tatami*[1]; 1. Yokohama National University, Japan

Many kinds of ceramics were fabricated using powder as raw materials. Optical, mechanical, electrical and thermal properties of advanced ceramics can be improved by optimization of the powder processing. Highly concentrated and dispersed slurry resulted in the dense and homogeneous green body to make transparent ceramics. Based on this technique, 3D small component of transparent SiO_2 was obtained using polymer mold prepared by stereolithography. Dense green body leads to transparent and luminescent SiAlON ceramics for high power LED application. Use of nanocomposite particles also important technique to control microstructure and to improve the properties of advanced ceramics. Uniform dispersion of Al_2O_3 to ZnO improved electrical conductivity by firing at a lower temperature and CNT / TiO_2 nanocomposite particles gave electrical conductivity to Si3N4 ceramics. Graphene coated nanocomposite particles resulted in the c-axis orientation of Si_3N_4 ceramics through molding in low and static magnetic field.

10:50 AM

(ICACC-PACRIM-012-2017) Fabrication of High Thermal Conductivity Silicon Nitride Substrates for Power Electronic Device Application (Invited)

Y. Zhou*[1]; H. Hyuga[1]; D. Kusano[2]; K. Hirao[1]; 1. National Institute of Advanced Industrial Science and Technology (AIST), Japan; 2. Japan Fine Ceramics Co., Ltd., Japan

Silicon nitride ceramics were prepared via a route of sintering of reaction-bonded silicon nitride (SRBSN) process from a high purity silicon powder. The ceramics showed obviously higher thermal conductivity compared to the silicon nitride ceramics prepared by the conventional gas pressure sintering of silicon nitride powder. The improvement of thermal conductivity was attributed to the lower content of oxygen impurity dissolved in the lattice of Si_3N_4 grains. The SRBSN ceramics also showed rather high fracture toughness. Thermal conductivities and thermal expansion coefficients of the prepared silicon nitride ceramics were measured at temperatures ranging from -50 to 250°C. These data are necessary for analysis of thermal stresses generated in the metalized silicon nitride substrates. The SRBSN ceramics with both high thermal conductivity and good mechanical properties should be promising substrate materials for the next generation of high-power electronic devices. 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).

11:20 AM

(ICACC-PACRIM-013-2017) Ceramics for Fluorine-based Plasma Environment: Erosion Mechanism and Plasma Resistant Materials (Invited)

S. Lee*[1]; S. Park[1]; Y. Oh[1]; H. Kim[1]; 1. Korea Institute of Ceramic Engineering and Technology (KICET), Republic of Korea

As transistor size in semiconductor industry has scaled down to 10 nm, particulate contamination has becoming of a critical issue. To tackle this, various ceramics have been used including oxides or carbides as sintered body or coating. The ceramics are usually exposed to fluorine- or chlorine-based plasma, experiencing erosion due to the interaction with the plasmas. The surface of the exposed ceramics was found to consist of a nanometer thin top C-F polymer layer and a underlying fluorinated oxide layer which is thermodynamically stable. Thus, this result supports that erosion of Al_2O_3 and Y_2O_3 ceramics under fluorocarbon plasma occurs by a physical removal process with simultaneous surface fluorination which was induced by the interaction with plasma. In these regards, a new dense Y_2O_3 coating is being studied using several techniques. Of these, suspension plasma spray could provide dense Y_2O_3 coating and a smooth surface, which contributes to reducing contamination particles. In addition to oxides, a new oxyfluoride material with various F content are being developed. For instance, YOF coating is believed to suppress the chemical interaction of the coating with the fluorocarbon plasma.

11:40 AM

(ICACC-PACRIM-014-2017) Designing novel strategies for enhancing materials education for undergraduate students (Invited)

S. Gupta*[1]; 1. University of North Dakota, USA

In this presentation, I will report an innovative strategy for designing senior design projects for developing better understanding of sustainable materials manufacturing for both online and campus based students. The author will also present a case study with engineering students where the students are located in different regions of the United States. For example, the critical challenges during the design of online senior design projects are, (a) establishing online communication between students, (b) teaching online students in real time, and (c) creating a challenging project where different online students can participate and contribute synchronously as well as asynchronously. In the second part of the talk, I will present different types of best-practice online strategies, which may be used to enhance Undergraduate (UG) student learning experiences. I will also showcase a Modified Flipped Learning Approach (MFLA) for teaching complicated courses. In addition, the effect of formative evaluation through SGID (Small Group Instruction Design) and informal assessment on course and student development will be discussed. These online strategies may further support student learning and have wider pedagogic applications.

6th Global Young Investigator Forum

Advanced Ceramics and Coatings for Structural, Environmental, and Functional Applications

Room: Coquina Salon F
Session Chair: Valerie Wiesner, NASA Glenn Research Center

8:30 AM

(ICACC-GYIF-010-2017) Joining of Ceramics: The Role of Ceramics Fusion Welding (Invited)

D. King*[1]; G. Hilmas[2]; W. Fahrenholtz[2]; 1. UES, Inc., USA; 2. Missouri University of Science & Technology, USA

The joining of materials to make complex shapes or multi-material components is an important and fundamental engineering challenge, and joining challenges become magnified when working with ceramics due to the high temperatures, and sometimes pressures, required for densification. Therefore, ceramics joining has been an area of interest, with research typically focused on two methods: brazing with a low melting temperature glass or metal, or solid state joining. Similarly, processes like electron beam and laser welding have been utilized to research fusion welding techniques, but their cost is of concern. However, not all ceramics are electrical insulators. With a room temperature electrical resistivity of ~6 μΩ●cm, ZrB_2 is great electrical conductor, better than 304 stainless steel (~72 μΩ●cm), and can be arc welded under the right conditions. The arc welding parameters control the size, shape, and efficiency of the arc welding process, but control of grain growth and microstructure are also important for fabricating a usable joint. While fusion welding is possible, more research is certainly necessary. But can the fusion welding of ceramics be useful, where can it be applied, what is the potential role of ceramics fusion welding?

9:00 AM

(ICACC-GYIF-011-2017) Synthesis and properties of ceramics derived from preceramic polymers

K. Kita*[1]; M. Fukushima[1]; H. Hyuga[1]; N. Kondo[1]; 1. National Institute of Advanced Industrial Science and Technology (AIST), Japan

Ceramics with unique shapes such as membranes and fibers are promising materials that can contribute to development of a gas separator and a novel material such as ceramics matrix composites, which can be obtained easily by using preceramic polymers. It was found that precursor blends containing various preceramic polymers could make ceramic fibers with very fine porosities. In addition, preceramic polymers could be used as a joining agent for alumina substrate. In this study, mixtures of polycarbosilane and some polysiloxane were prepared as a precursor polymer, in which membranes and melt-spun into fibers were formed by those blends. By thermal control, porous membranes with average pore diameter around 600 nm could be obtained.

9:20 AM

(ICACC-GYIF-012-2017) Additive Manufacturing of Dense Ceramic Parts via Direct Ink Writing of Aqueous Suspensions

L. Rueschhoff*[1]; W. Costakis[1]; A. Diaz Cano[1]; J. Youngblood[1]; R. Trice[1]; 1. Purdue University, USA

Additive manufacturing is gaining momentum in all areas of materials research due to the ability to create three-dimensional parts directly from CAD designs through adding materials layer-by-layer, producing near- net shape complex geometries that would not be possible with subtractive manufacturing. In the direct ink writing additive manufacturing approach, a highly-loaded colloidal ink is extruded through a small cylindrical nozzle to create a filamentary element that is deposited in a layer-by-layer sequence to produce three-dimensional components. In this study, a low-cost syringe style 3D printer has been utilized to direct write a variety of previously developed ceramic aqueous suspensions including alumina, boron carbide, and silicon nitride. The suspensions consist of a ceramic powder, dispersant, polymer binder, and water and have yield-pseudoplastic rheological properties that have been optimized for the direct writing process. The effect of ceramic solids loading and polymer concentration on rheology, specimen uniformity, density, microstructure, and mechanical properties was studied. All parts produced through this method were sintered to high densities, with no cracking or delamination between deposited layers observed.

9:40 AM

(ICACC-GYIF-013-2017) Direct ink writing of geopolymers

G. Franchin*[1]; L. Zeffiro[1]; P. Scanferla[1]; H. Elsayed[1]; A. Baliello[2]; M. Pasetto[2]; P. Colombo[1]; 1. University of Padova, Italy; 2. University of Padova, Italy

Geopolymers are based on an inorganic 3D network of alumino-silicate units usually synthesized through reaction of alumino-silicate powders in presence of a silicate alkaline solution. The rheological characteristics of the reactive mixtures and the fact that these systems can consolidate at low or even room temperature, together with their intrinsic micro- and meso-porosity and mechanical properties, are the reason why they are considered for a wide range of applications, such as construction materials, thermal insulation, filters, adsorbers and so on. Our group has been exploring innovative fabrication processes for geopolymer components, focusing on additive manufacturing technologies. We used, for the first time, mixtures based on a geopolymeric binder for 3D printing of components which can be used at room or at high temperature, as they can easily withstand heating up to at least 1200°C. As we developed geopolymeric inks for direct ink writing, particular focus was put on their rheological properties. We investigated the effect of different water content and different type and amount of additives in order to enhance the Bingham pseudo-plastic behavior of the inks. Such optimization resulted in the ability to fabricate components with overhangs and spanning features, including highly porous 3D lattices. Further characterization included SEM and mechanical testing.

Ceramic Materials and Composites for Aerospace, Armor, Biological, Energy and Medical Applications

Room: Coquina Salon F
Session Chair: Go Kawamura, Toyohashi University of Technology

10:20 AM

(ICACC-GYIF-014-2017) $Na_3V_2(PO_4)_3$ as a Cathode Material for Sodium-ion Batteries

A. Murugappan*[1]; M. Law[2]; P. Balaya[2]; 1. University of California, Berkeley, USA; 2. National University of Singapore, Singapore

Due to the existing concerns regarding the lifespan and cost of Li-ion battery technology, the Na-ion battery is a fast-growing candidate to replace the Li-ion battery for large-scale energy storage applications. $Na_3V_2(PO_4)_3$ (NVP) is one of the preferred polyanion-based cathode materials due to its relatively high operating voltage (3.34 V) and high theoretical specific capacity (117.6 mAh g^{-1}). In this regard, we introduce a cost-effective, environmentally friendly and highly scalable cathode material for sodium-ion batteries. Pure NVP is synthesised by a water-rich soft template approach using V_2O_5 as the inexpensive vanadium precursor. FESEM images recorded on the samples show that the particles are of pseudo-spherical morphology. This material tested against Na metal is capable of delivering an initial discharge capacity of 109 mAh g^{-1} at 0.1 C, with an energy storage capacity of ~364 Wh kg^{-1}. At low current rates, the electrochemical performance is comparable to the NVP synthesized using the more expensive method.[1] However, a rapid decrease in discharge capacity is observed at medium to high current rates. With further optimizations, we believe our NVP could be realized as a potential Na-ion battery cathode for grid storage applications.

10:40 AM

(ICACC-GYIF-015-2017) Enhancement of nitridation of silicon with various rare earth oxides addition and the reaction mechanism

T. Okuno*[1]; Y. Zhou[1]; K. Hirao[1]; H. Hyuga[1]; 1. National Institute of Advanced Industrial Science and Technology (AIST), Japan

Silicon nitride (Si_3N_4) as a representative structural ceramic has been used for several decades because of its good mechanical properties, high-temperature durability, and chemical resistance. Among the different classes of silicon nitride ceramics, sintered reaction-bonded Si_3N_4 (RBSN) has been considered as an attractive material due to low cost of raw Si powders and high strength retention at elevated temperature. However, the nitridation of Si powder requires a long time to complete and it is difficult to apply RBSN ceramic industrially due to this obstacle. In order to overcome this obstacle, many researchers have reported the nitridation of silicon with some metals and metal oxides as sintering additives. In this study, some rare earth oxides were used as nitridation enhancing additives for the nitridation of Si powder. The effect of the rare earth oxide additives on the nitridation behaviors of Si powder and the reaction mechanism was investigated.

11:00 AM

(ICACC-GYIF-016-2017) Solution carbothermal processing of nano phase ZrC powders

S. N. Katea*[1]; G. Westin[1]; 1. Uppsala University, Sweden

ZrC is of great interest for cermets due to its high melting point, hardness, and durability. In this work, a new solution based process to nano-phase ZrC based on sucrose-Zr-alkoxide precursors is described. The phase evolution from gel to ZrC was investigated in detail with TG-DTA, XRD, IR and Raman spectroscopy SEM-EDS and TEM-EDS. It was found that an extremely intimate mixing of ca 4 nm sized ZrOx(OH)y particles and carbon as a graphene type coating surrounding the particles was obtained at 200C, and that the Zr-particles were converted to ZrO2 at 600C with retained structure at 600C. The carbothermal carburation reactions started at ca 1200C and were finished at 1500C, resulting in a phase pure ZrC according to XRD, but TEM showed that the nanometre sized particles had a 4-5nm thick shell of a different material than the ZrC bulk.

11:20 AM

(ICACC-GYIF-017-2017) Effect of morphologies on strength and thermal conductivity in gelation-freezing derived porous ceramics

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

Porous ceramics with very high porosity up to 93vol% were fabricated by gelation freezing method. The thermal conductivity of obtained samples exhibited 0.1 to 0.6W/mK at room temperature, and the compressive strength was measured to be 1 -26MPa. The properties measured were strongly influenced by processing conditions of solid contents in the initial slurry, freezing condition, ice binding additives and sintering temperature. The image based modelling for mechanical properties and thermal conductivities, based on microstructures collected by X-ray computed tomography, will be also discussed, in terms of those properties and cellular morphologies created by gelation freezing.

11:40 AM

(ICACC-GYIF-018-2017) Shaping of ceramic microspheres using vibrational droplet coagulation

J. Pype*[2]; B. Michielsen[1]; S. Mullens[1]; V. Meynen[2]; 1. VITO, Belgium; 2. University of Antwerp, Belgium

Ceramic microspheres are widely used in a broad range of applications, all with their own demands on the ideal correlation between material properties and performance, required in the processes. These demands led us to produce ceramic microspheres of different sizes and architectures with a wide range of control. In order to obtain uniform microspheres, the vibrational droplet coagulation was selected as most controlled and versatile. This granulation technique enables the shaping of microspheres with different architectures varying from dense to hollow. The current research stipulates the impact and importance of combining both variations in the chemical and physical parameters controlling the vibrational droplet coagulation technique. By controlling the suspension composition (powder content/type, amount/type of the alginate and presence of additives), a wide variety of microspheres can be shaped. Depending on the requirements of the application, the porosity and mechanical strength can be controlled by adapting the suspension composition in combination with sinter profile and temperature. The technique has been applied to different materials, architectures and in applications of waste valorization, each with other requirements of control and complexity.

FS2: Advanced Ceramic Materials and Processing for Photonics and Energy

Multi-functional Materials I

Room: Halifax A/B

Session Chairs: Gilles Lerondel, University of Technology of Troyes; Muhammet Toprak, KTH Royal Institute of Technology

8:30 AM

(ICACC-FS2- 006- 2017) ZnS-$CaLa_2S_4$ Infrared Optical Ceramics Sintered by a Field-Assisted Technology

Y. Li*[1]; Y. Wu[1]; 1. Alfred University, USA

As a pressure-assisted and current-activated sintering, field-assisted sintering technique (FAST) has been extensively studied for materials processing in the recent years. In this study, high-energy ball milling was employed to mix and grind the commercial $CaLa_2S_4$ powders and ZnS nanopowders synthesized by a wet chemical precipitation method. The ZnS-$CaLa_2S_4$ composite powders were sintered via the FAST. The phase compositions of ZnS-$CaLa_2S_4$ composite ceramics sintered at different temperatures were investigated by XRD analysis. Grain growth kinetics and sintering behaviors of the ZnS-$CaLa_2S_4$ ceramics were discussed to understand the correlations between the microstructures and sintering variables. Mechanical properties were also characterized to analyze the influences of compositions and microstructures on hardness and fracture toughness of the ZnS-$CaLa_2S_4$ ceramics. Optical characterizations of the ZnS-$CaLa_2S_4$ composite ceramics were performed to investigate the infrared optical compatibility of two phases and defects within the ZnS-$CaLa_2S_4$ composite ceramics.

8:50 AM

(ICACC-FS2- 007- 2017) ZnO-PMMA quantum dots nanocomposites for electrical switching applications

M. S. Toprak*[1]; V. Doddapaneni[1]; M. Saleemi[1]; F. Ye[1]; H. Edin[2]; 1. KTH Royal Institute of Technology, Sweden; 2. KTH Royal Institute of Technology, Sweden

One of the key technological components in the smart grid is a circuit breaker, which has been playing a key role at power generation, transmission and distribution levels. It is an automatic mechanical switching device, with a role to safeguard the smart grid from the faults by automatic interruption. During interruption of high fault currents, it generates a high-energetic electrical arc. In order to protect from such electrical arcs, ceramic plates have been widely used in circuit breakers due to their high thermal resistance. Since last decade, polymers have been used to cool down the electrical arcs effectively due to their outgassing nature in addition to the thermal protection. This work reports on the newly engineered polymer inorganic nanocomposites (PINCs) for quenching the electrical arcs and to improve the operational limits of electrical switching applications. We report on the fabrication and electrical arc testing of PINCs composed of polymethyl methacrylate (PMMA) and ZnO quantum dots (QDs). Prepared QDs and PINCs have been characterized in detail using a variety of techniques, including TEM, SEM, FTIR, UV-Vis spectroscopy. The experimental findings shows that the presence of ZnO QDs improved the key obstructive nature of broad range UV radiation absorption of PMMA and the fabricated PINCs revealed improved operational limits in terms of voltage rise and arc interruption capability.

9:10 AM

(ICACC-FS2- 008- 2017) 2D Materials Growth: Applications and Challenges (Invited)

M. Siaj*[1]; 1. UQAM, Canada

Two-dimensional (2D) materials have attracted much attention due to their unique properties. Controllable synthesis of 2D materials with high quality and high efficiency is essential for their large-scale applications. In parallel to the chemical synthesis route, chemical vapor deposition (CVD) has been one of the most important techniques for the synthesis of 2D materials. The present talk will be devoted to the CVD growth of graphene, boron nitride, core-shell nanoparticles@graphene and transition metal dichalcogenides (TMDs) in our research group. The Hydrogen-induced effects during the growth will be discussed. In parallel, we will show that the use of these resulting 2D materials as electrodes leads to an enhancement of the overall reactivity and sensors sensitivity which is favorable for many applications.

9:40 AM

(ICACC-FS2- 009- 2017) Nanostructured semiconducting metal oxides as flexible platform for solar energy exploitation (Invited)

I. Concina*[1]; 1. Luleå University of Technology, Sweden

The design of multifunctional systems for solar energy exploitation, conjugating reasonable efficiency with low environmental and economical impact for fabrication, is among the most intriguing challenges for materials science. This lecture will focus on the synthesis of nanostructured semiconducting metal oxides (ZnO hierarchical nanostructures, SnO_2 nanoparticles and α-Fe_2O_3 quasi 1D structures and thin films), combined with strategies for rational devices engineering, to be applied as photoelectrochemical active materials in solar energy exploitation in the fields of photovoltaics, water splitting and catalysis for water remediation. We will show how a materials-by-design approach holds the potential to empower the functionality of earth abundant materials in relevant applications aimed at exploiting the solar light, while highlighting at the same time some intrinsic limitations in performances. Emphasis will also be given to simple, cheap and green fabrication processes, such as spray deposition, hydrothermal synthesis, water-based wet chemistry, enabling the synthesis of nanomaterials with enhanced functional performances while complying with green chemistry principles.

10:30 AM

(ICACC-FS2- 010- 2017) ZnO as a multifunctional photonic material: emphasis on enhanced optical properties and controllable nanostructuring (Invited)

G. J. Lerondel*[1]; A. Gokarna[1]; K. Nomenyo[1]; A. Gwiazda[1]; C. Chevalier[1]; A. Rumyantseva[1]; 1. University of Technology of Troyes, France

We will review the intrinsic and extrinsic properties that make ZnO a remarkable material for UV and visible photonics when hybridized with other absorbing / emitting materials. Intrinsic properties include high intrinsic quantum efficiency in solution grown material and high optical gain in epitaxial thin film which together with high refractive index can lead to a new kind of optical energy transfer so called GWET. In the second part of the presentation we will show how these intrinsic properties can be enhanced using light confinement in nanostructured ZnO. Nanostructuring includes top-down approach using ebeam lithography and dry etching for enhanced light emisison in 2D planar photonic thin films. Nanostructuring also include combined top-down and bottom-up approaches for enhanced light absorption and antireflection coating in heterostructured materials i.e. ZnO nanorods deposited on mesoscale periodically patterned silicon. We will also introduce a new technique for ZnO structuring namely direct photochemical dissolution. Finaly we will illustrate the multifonctionality of these meso and nanostructured thin films for direct transfer in other materials and especially oxides using ZnO material as a universal mask. Results will be illustrated using several applications such as chemical sensing, lighting and photon-to-electron conversion.

11:00 AM

(ICACC-FS2- 011- 2017) Advanced porous graphene thin films for energy-efficient water filtration (Invited)

G. Fanchini*[1]; 1. University of Western Ontario, Canada

In this presentation, we will review our efforts towards the development of next-generation membranes for water purification based on weakly oxidized and nanoporous few-layer graphene. Our reverse osmosis filtration system minimizes the energy cost of filtration processes because it combines the advantages of single-layer graphene, bored membranes, which are suitable to low differential pressures (~10 KPa) with those of graphene oxide, exhibiting high surface-area and superior performance in terms of impurity adsorption. Application of our system to dead-end and crossflow water filtration processes will be iscussed. We will focus on water purification fro metal ions and we will demonstrate that our membranes with nanometer and subnanometer pore size offer superior retention of common metal ions, including Cu and Fe. Strategies for retention of critical metal ions (including Cd) will be dicussed as well in light off graphene pore functionalization. Possible solutions towards large-area scalability of these membranes and their commercialization will also be presented.

11:30 AM

(ICACC-FS2- 012- 2017) Conducting polymers and polymer-based nanocomposites: Their potential role on energy-related technology (Invited)

V. M. Castano*[1]; 1. Universidad Nacional Autonoma de Mexico, Mexico

A review of the historical development of conducting polymers will be presented, emphisizing the challenges that the pioneers of the field faced, from a technical standpoint, along with a discussion of the current problems in large-scale technologies for energy production, conversion and storage, that could greatly benefit from the physicochemical and engineering properties of macromolecules, both natural and synthetic, provided their electrical and ionic conductivity can match those of the materials currently in use. The details of the molecular limitations of polymer-related materials will be analyzed, as well as the alternatives that have been proposed and/or explored in the last few decades. Three examples of novel conducting polymers for generation, conversión and storage of energy, respectively, will be presented, explaining also the problems that their industrial scaling-up is presenting and the actual results of their practical application.

S1: Mechanical Behavior and Performance of Ceramics & Composites

SiC/SiC Materials

Room: Coquina Salon D
Session Chairs: Masaki Kotani, JAXA; Dietmar Koch, Institute of Structures and Design

8:30 AM

(ICACC-S1-012-2017) The conditions of fiber/matrix interface and matrix formation of the polymer-derived SiC/SiC composite towards the best mechanical properties (Invited)

M. Kotani*[1]; K. Konaka[2]; S. Ogihara[2]; 1. Japan Aerospace Exploration Agency, Japan; 2. Tokyo University of Science, Japan

The effects of major influential factors of a polymer-derived SiC/SiC composite on the tensile properties, such as interface layer thickness, filler additive rate, the kind of precursor polymer and heat treatment temperature etc., were systematically investigated.

9:00 AM

(ICACC-S1-013-2017) Manufacturing of damage tolerant SiC/SiC ceramic matrix composites for long term applications

D. Koch*[1]; B. Mainzer[1]; R. Jemmali[1]; 1. German Aerospace Center, Germany

Silicon carbide fiber reinforced silicon carbide matrix composites (SiC/SiC) are promising materials for long term applications under high temperature conditions as they show corrosion resistance and creep stability under severe conditions. These properties make SiC/SiC attractive for applications in jet engines or micro gas turbines. For manufacturing of SiC/SiC two processing routes are performed, liquid silicon infiltration (LSI) and polymer infiltration and pyrolysis (PIP). The resulting properties of the different SiC/SiC composites are compared. At LSI a phenolic resin is infiltrated in a fibrous preform and converted to porous carbon matrix by pyrolysis treatment. The porous structure is then infiltrated with molten Si which reacts with the carbon to SiC. In order to prevent degradation of the SiC fibers during the aggressive Si infiltration process the fibers must be coated effectively. Furthermore this fiber coating enhances fracture toughness in the final microstructure by effective fiber matrix debonding. For comparison in PIP process a Si-organic precursor is infiltrated, cured, and pyrolized to ceramic (Si-C(-N)) matrix. Due to process induced shrinkage several reinfiltration and pyrolysis steps have to be performed until an acceptable porosity is achieved. The densification process is studied by extended use of μCT analysis after each infiltration step.

9:20 AM

(ICACC-S1-015-2017) Effects of post heat-treatment on the mechanical properties of SiC$_f$/SiC composites fabricated by a hybrid route

P. Fitriani*[1]; A. Sharma[1]; A. Septiadi[1]; D. Yoon[1]; 1. Yeungnam University, Republic of Korea

Continuous SiC fiber-reinforced SiC matrix composites (SiC$_f$/SiC) have been recognized as potential materials for high temperature applications. In recent times, extensive research work is devoted to tailor the fiber–matrix interface of SiC$_f$/SiC composites to improve the mechanical properties. In this work, we examined the effects of variation of hot-pressing temperature (1650 and 1750°C) and pressure (10 and 20 MPa) using different sintering additives, such as Al_2O_3-Y_2O_3, Al_2O_3-Sc_2O_3, and rare earth nitrates. SiC fabrics coated with PyC were used as reinforcement and subsequently infiltrated with SiC matrix using electrophoretic deposition or vacuum infiltration route. SiC$_f$/SiC composites with rare earth (Sc, Er, or Yb) nitrate as sintering additives resulted in densities greater than 95% ρ_{theo} and flexural strength in the range 410 – 450 MPa. A strengthened matrix with ultrafine SiC grains for e.g., 150 nm for Sc-nitrate, 690 nm for Er-nitrate etc. can also be obtained. SiC$_f$/SiC composites with Al_2O_3-Y_2O_3 as sintering additive were subjected to optimized post-sintering heat treatments (1150, 1250 and 1350°C for 5h) showed marked tail extension behavior with respect to as-sintered specimens. Correlation between the microstructural features and fracture modes was established to analyze and explain the observed tail extension behavior in these composites.

9:40 AM

(ICACC-S1-016-2017) Effects of braiding angle on damage mechanisms in SiC/SiC composite tubes characterized by X-ray computed tomography

Y. Chen*[1]; L. Gélébart[1]; M. Bornert[2]; C. Chateau[2]; A. King[3]; C. Sauder[1]; 1. French Alternative Energies and Atomic Energy Commission, France; 2. Laboratoire Navier/ENPC/Université Paris-Est, France; 3. French National Synchrotron SOLEIL, France

The mechanical behavior of 2D-braided SiC/SiC composite tubes depends strongly on the braiding angle. Tubes of three different braiding angles (30°, 45°, 60°) have been observed during in situ tensile tests using synchrotron X-ray tomography. In order to detect the slightly open cracks from heterogeneous microstructures, various image processing techniques have been used and developed. Digital Volume Correlation (DVC) allows the 3D displacement field within the material under loading to be estimated. The crack networks are extracted by a DVC-based image subtraction. The detected cracks are first classified into two groups according to their orientations, and then are characterized in qualitative and quantitative ways. The crack analyses on the three different tubes provide not only a study on the braiding angle effects but also a better understanding on the damage mechanisms of the composite.

10:20 AM

(ICACC-S1-017-2017) Electrical resistivity monitoring of SiC/SiC composites during ageing under oxidizing environments

C. Simon*[1]; G. Camus[1]; F. Rebillat[1]; 1. Laboratoire des Composites Thermostructuraux, France

The introduction of Ceramic Matrix Composites parts in civil aeronautics requires to precisely follow and thoroughly understand their evolution usually encountered under the oxidizing environments present within the engines. The development of non-destructive evaluation techniques such as acoustic emission or electrical resistivity is therefore essential to both tasks. In this respect, the electrical resistivity of a SiC/PyC/[Si-B-C] specimen has been first monitored during a room temperature tensile test. As a good correlation was found with the development of a model between the evolution of the resistance and the damage state of the material (crack density and debonding density), electrical resistivity monitoring has then been performed during ageing tests of a few hundreds of hours, under various environments (450°C, ambient and moist air at 10 kPa of water pressure) and mechanical conditions (maximum stress of 100 MPa under creep or cyclic fatigue). It appeared that the oxidation of an essential constituent of the composite, namely its pyrocarbon interphase, led to an increase of the electrical resistivity of the specimen. Electrical resistivity monitoring is hence a promising technique allowing a real time estimation of the oxidation of the interphase and of the damage state of the material.

10:40 AM

(ICACC-S1-018-2017) Use of Electrical Resistance and Acoustic Emission During Fatigue of Woven SiC/SiC Composites under Different Conditions

Z. Han*[1]; 1. University of Akron, USA

Electrical resistance (ER) and acoustic emission (AE) have been proved to be feasible approaches of real-time monitoring and evaluating damage in silicon carbide fiber-reinforced silicon carbide (SiC/SiC) composites for a variety of loading conditions. In this study, they are used to monitor fatigue tests of two types of woven SiC/SiC composite systems with HNS and SA type fibers. Coupons, both at their pristine and post high-velocity impact states, are cycled under various loading frequencies ranging from 0.01Hz to 1Hz. Temperature effect is also considered, as tests were carried out at room temperature and elevated temperature (1200C). A method based on the slope of the ER evolution has been developed. It allows a critical damage phase to be identified. Mechanical and electrical-mechanical models are also considered and compared with the experimental data.

11:00 AM

(ICACC-S1-019-2017) High Velocity Impact of 3-D Woven SiC/SiC Ceramic Matrix Composites of Varying Architectures at Ambient and High Temperature

M. J. Presby*[1]; R. K. Smith[1]; G. N. Morscher[1]; C. Iwano[2]; B. Sullivan[2];
1. The University of Akron, USA; 2. Materials Research and Design, USA

Foreign object damage (FOD) is of great concern to the airline industry because particles ingested into turbine engines can severely degrade performance and durability which leads to serious safety and financial concerns. Many studies have reported on the damage resistance due to high velocity impact in 2-D woven ceramic matrix composites (CMCs), but there is very little reported on the impact damage tolerance in 3-D woven CMCs. This study aims to understand the impact resistance of three different 3-D woven fiber architectures subjected to high velocity impact at ambient (21°C) and high temperature (1093°C) using a 1.59 mm projectile impinging at a velocity of ~350 m/s. Non-destructive evaluation (NDE) techniques such as electrical resistance (ER) and micro-computed tomography (micro-CT) were used to evaluate the impact damage. Post-impact retained strength was determined using a four-point bend test at room temperature. Modal acoustic emission (AE) and ER were utilized during post-impact retained strength to monitor damage accumulation.

11:20 AM

(ICACC-S1-020-2017) In situ observation of damage in SiC_f/SiC_m composites under load at high temperature

A. Singhal*[1]; Y. Gao[1]; Y. Zhou[1]; E. Maillet[1]; H. Barnard[2]; D. Parkinson[2];
A. MacDowell[2]; G. Henson[1]; 1. GE, USA; 2. Lawrence Berkeley National Laboratory, USA

Ceramic matrix composites are a new class of materials with promise for high temperature structural applications. This work focuses on the observation of damage in melt-infiltrated cross-ply SiC-SiC CMCs. Damage is expected to accumulate in these materials at the length scale of the fiber diameter and fiber coatings, or matrix inhomogeneities in the microstructure. Therefore, it is necessary to observe and capture the sequence of events at this length scale to build a better understanding of the failure mechanisms, and also to validate high-fidelity models. In situ imaging under monotonic tensile loading was performed at the Advanced Light Source (ALS, LBNL) to study the crack patterns, occurrence of fiber breaks and their correlation with the microstructural features. High-resolution images from simple monotonic tests showed the location of the first instance of damage in the interior of the sample followed by a sequence of events, namely matrix damage, fiber bridging and pullout, and damage propagation through the fibers, leading to the final failure of the specimen. Tests on samples with different notch geometries shed light on the effect of stress concentration and microstructure. Acoustic emission (AE) was also monitored in parallel with the tensile tests to correlate damage observed in the tomography images with the recorded AE signals.

11:50 AM

(ICACC-S1-021-2017) Rare earth disilicate fiber coatings for SiC/SiC CMCs

E. E. Boakye*[1]; P. Mogilevsky[1]; T. Parthasarathy[1]; T. Key[1]; M. Cinibulk[2];
R. Hay[2]; S. Opeka[1]; 1. UES Inc., USA; 2. Air Force Research Laboratory, USA

Rare-earth disilicates ($RE_2Si_2O_7$) are considered as potential oxidation-resistant alternatives to carbon or BN fiber coatings for SiC/SiC CMCs. Measurements of their mechanical properties (hardness and modulus), thermal expansion, and the determination of favorable sliding stress and debond energies of SCS-0 SiC fibers in rare earth disilicate matrices support this assertion. However, so far there has not been any experimental demonstration of proof concept that rare earth disilicates will indeed work as a weak interface in fiber-reinforced SiC/SiC CMCs, since to date rare-earth disilicate coatings have not been successfully applied to fine-diameter SiC fiber tows. In this presentation, thermodynamic analysis on the formation of rare earth disilicate coatings on SiC fibers is presented. Based on this analysis, $Y_2Si_2O_7$ was formed on Hi-Nicalon S fibers. The effect of different processing parameters on the formation of $Y_2Si_2O_7$ on Hi-Nicalon S fibers along with TEM/XRD/SEM data on the phase composition of the resulting coatings are presented.

12:10 PM

(ICACC-S1-080-2017) Failure analysis and sub-critical crack growth (SCG) characterization of Pt-Al_2O_3 high temperature co-fired (HTCC) ceramics

R. Tandon*[1]; C. Newton[1]; A. Thom[2]; A. Knudsen[3]; 1. Sandia National Laboratories, USA; 2. Medtronic Inc, USA; 3. Kyocera North America, USA

Miniaturization and integration of multiple functions into one component is desired for device downsizing. HTCC alumina, with its high strength and bio-compatibility, provides an option for integration and higher reliability medical devices. A 92% alumina-platinum via HTCC material was characterized for failure modes and sub-critical crack growth, and modifications to processing were implemented to obtain a material with strength and SCG characteristics similar to bulk alumina. In phase I development, this material had strength equivalent to bulk alumina, except at low stressing rates in saline, body fluid-like environments, where a novel failure mode (circumferential cracking) was encountered. Processing changes implemented for Phase II materials led to failures emanating as radial cracks from the vias with slight loss of strength. Changes made to Phase III materials led to strength and SCG characteristics that were undistinguishable from the base materials. Fractographic evidence indicates that cracking originates tangentially to the via in Phase III material Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000

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

Environmental Barrier Coatings I: Processing & Characterization

Room: St. John

Session Chairs: Kang Lee, NASA Glenn Research Center; Peter Mechnich, German Aerospace Center (DLR)

8:30 AM

(ICACC-S2-010-2017) The water-vapor corrosion behaviors of EBC-coated CMC materials (Invited)

Y. Wang*[1]; 1. Northwestern Polytechical University, China

Advanced ceramic matrix composites (CMCs), in particular silicon carbide fiber reinforced silicon carbide ceramic matrix composites (SiC/SiC), are increasingly used in the aerospace industry for the manufacturing of hot components due to their excellent properties compared to superalloys. In the aero-engine, about 5-10% of the combustion gas is water vapor resulting from the burning of hydrocarbon fuels in air. The water vapor will corrode the CMC components, resulting in their final failure. To protect the SiC/SiC components from corrosion, environmental barrier coatings (EBCs) have been developed to increase their durability in combustion environments. Recently, we found that the durability of EBC coated CMC composites was determined by the thermal grown oxide (TGO) layer between EBC and SiC bonding layer. This phenomenon makes us re-consider the function of EBCs. Moreover, how they perform their function to resist the corrosion to water-vapor is yet clear. In this presentation, we studied the kinetics of the growth of TGO at different temperatures. The oxidation rates and activation energies can be obtained. The corrosion mechanism of EBC-coated CMC is stated accordingly. The test condition for simulation experiments is also discussed in this presentation.

9:00 AM

(ICACC-S2-011-2017) Nano-Structured Ceramic Coatings to Stabilize SiC Against Reaction in High Temperature Steam

A. Hoskins*[1]; C. Musgrave[1]; A. Weimer[1]; 1. University of Colorado Boulder, 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. We propose that the application of nanostructured films grown with atomic layer deposition (ALD) will significantly improve the oxidation resistance of SiC in extreme environments. We have targeted a variety of high temperature coating materials chosen based on thermal properties, structural characteristics, and stability in oxidative environments. We have grown ALD layers of our desired materials on SiC particles using a fluidized bed particle ALD reactor. Using thermogravimetric analysis, it has been shown that these coatings improve the oxidation resistance of SiC. We have applied density functional theory (DFT) to model the performance of these coatings in an oxidative environment. Computational modeling of the chosen coating material properties, as well as oxygen diffusion through these materials can identify promising coatings to further extend the lifetime of SiC. We expect to use this understanding to develop more advanced stabilizing coatings for SiC and the myriad of other applications that require high-temperature oxidation resistant materials.

9:20 AM

(ICACC-S2-012-2017) Quantitative measurement of delamination toughness of oxide EBC layer from SiC/SiC substrate

Y. Aoki*[1]; Y. Kagawa[1]; 1. The University of Tokyo, Japan

A simple experimental method for measurement of delamination toughness in BSAS/(mullite+BSAS)/Si EBC layer on three-dimensional (3D)-woven SiC/SiC substrate system under shear loading mode (phase angle ~80°) has been developed. The method has an advantage of low mechanical loading to 3D-woven SiC/SiC substrate to allow damage free condition of the substrate. By using the developed method, stable crack propagation is clearly observed and steady state delamination toughness is obtained from the steady state delamination stage. Delamination behavior of EBC layer is strongly affected by 3D woven fiber architecture of SiC/SiC substrate and constitute materials and their microstructure of EBC layer. In this study, special attention has been focused on effects of 3D-SiC woven architecture of SiC/SiC substrate on delamination crack path, phase angle and value of delamination toughness.

9:40 AM

(ICACC-S2-013-2017) Development of environmental barrier coatings for ceramic materials

H. Klemm*[1]; K. Schönfeld[1]; W. Kunz[1]; 1. FhG IKTS Dresden, Germany

During recent years, significant progress has been achieved in the development of environmental barrier coatings (EBC) for oxide and non-oxide ceramic materials for gas turbine applications. Based on detailed understanding, several multilayer coating systems with high corrosion stability were fabricated and tested successfully. Several functions have to be considered in the design and fabrication of an EBC, especially as the consequence of the long service time with rapid cooling steps, impurities or particle impact. One of the main challenges was found to be the composition and design of the bond coat between the ceramic base material (monolithic or ceramic matrix composite, CMC) and the upper EBC layer system. Diffusion processes or reaction at this interface (oxidation) led to microstructural changes with the consequence of crack formation and failure of the EBC finally. In the present study, the mechanisms of these processes will be analyzed based on oxidation and hot-gas corrosion experiments. Finally, some idea about the interface design in both, oxide and non-oxide ceramic systems will be discussed.

10:20 AM

(ICACC-S2-014-2017) Environmental Barrier Coatings for Ceramic Matrix Composites – An Overview (Invited)

K. N. Lee*[1]; 2. van Roode[2]; D. Zhu[1]; T. Kashyap[3]; V. L. Wiesner[1]; 1. NASA Glenn Research Center, USA; 2. Mark van Roode & Associates, USA; 3. Pratt & Whitney, USA

SiC/SiC Ceramic Matrix Composites (CMCs) are increasingly being considered as structural materials for advanced power generation equipment because of their light weight, higher temperature capability, and oxidation resistance. Limitations of SiC/SiC CMCs include surface recession and component cracking and associated chemical changes in the CMC. The solutions pursued to improve the life of SiC/SiC CMCs include the incorporation of coating systems that provide surface protection, which has become known as an Environmental Barrier Coating (EBC). The development of EBCs for the protection of gas turbine hot section CMC components was a continuation of coating development work for corrosion protection of silicon-based monolithics. Work on EBC development for SiC/SiC CMCs has been ongoing at several national laboratories and the original gas turbine equipment manufacturers. The work includes extensive laboratory, rig and engine testing, including testing of EBC coated SiC/SiC CMCs in actual field applications. Another EBC degradation issue which is especially critical for CMC components used in aircraft engines is the degradation from glassy deposits of calcium-magnesium-aluminosilicate (CMAS) with other minor oxides. This paper addresses the need for and properties of external coatings on SiC/SiC CMCs to extend their useful life in service and the retention of their properties.

10:50 AM

(ICACC-S2-015-2017) Oxygen Tracer Diffusion in Yttrium Silicates

R. A. Golden*[1]; E. Opila[1]; 1. University of Virginia, USA

The insertion of SiC CMCs in next generation gas turbine engines relies upon EBCs to protect CMCs from volatilization and oxidation. Rare-earth (RE) silicates show sufficient thermochemical stability in high temperature water vapor making them ideal EBC candidate materials. However, the ability of RE silicates to protect SiC CMCs from oxidation is not well known. This study focuses on measuring the oxygen diffusivity in yttrium silicates (Y_2SiO_5, $Y_2Si_2O_7$). Yttrium silicate powders were consolidated by Spark Plasma Sintering (SPS). Specimens were cut and polished for tracer diffusion experiments. $^{16}O_2$-equilibrated specimens were encapsulated inside fused quartz tubes containing $^{18}O_2$. Encapsulated specimens were exposed at diffusion temperatures of 1000-1300°C for times of 10-180 minutes. The ^{18}O diffusion concentration profile was measured for each specimen using Time of Flight - Secondary Ion Mass Spectrometry (ToF-SIMS). Oxygen tracer diffusion coefficients were obtained by fitting the semi-infinite solution of the diffusion equation to the concentration profiles. Oxygen diffusion coefficients in yttrium silicates ranged from 10^{-12} – 10^{-14} cm^2/s. ^{18}O maps showed higher concentrations along grain boundaries than within grains, indicating grain boundary diffusion is more rapid than bulk lattice diffusion. Implications for oxidation protection of SiC composites are discussed.

11:10 AM

(ICACC-S2-016-2017) EBC slurry infiltrated matrix/coatings for woven SiC/SiC composites

J. Zhou*[1]; G. G. Chase[1]; A. S. Almansour[3]; G. N. Morscher[4]; B. J. Harder[2]; 1. the University of Akron, USA; 2. NASA Glenn Research Center, USA; 3. NASA Glenn Research Center, USA; 4. the University of Akron, USA

One of the current challenges for SiC/SiC composites is that moisture limits the component lifetime by reacting with protective a silica layer to form volatile silicon hydroxide ($Si(OH)_4$). Historically, protective oxide environmental barrier coatings are applied to protect the SiC-based substrate via physical vapor or plasma spray deposition approaches. Coating processing can be costly and requires good bonding to the substrate in order to insure protection. Traditional plasma sprayed coatings rely on a physical bond to the surface, and delamination or erosion of this layer can expose the SiC/SiC fiber matrix to the turbine environment. An alternative approach to CMC environmental protection is to infiltrate the environmental barrier coating material as an integral part of the matrix of the SiC composite. In principle this provides a more durable matrix with the moisture barrier within the depth of the SiC/SiC fiber-reinforced skeletal structure. In this study, slurry based mullite, ytterbium silicate multilayers were infiltrated into porous chemical vapor infiltration (CVI) SiC/SiC woven composites with three different porosity contents. The mullite served as a bond layer and the ytterbium silicate as the EBC layer. The effectiveness of infiltration and coating morphology as well as the resistance to high temperature water vapor exposure of the coated systems are discussed.

11:30 AM

(ICACC-S2-017-2017) Tantalum-based Ceramic Coatings for Extremely Corrosive Environments and Fiber Interface Coatings for High Temperature Fiber-reinforced CMCs

J. Stiglich*[1]; B. Williams[1]; D. Gambale[1]; 1. Ultramet, USA

Under NASA funding, Ultramet developed a process to diffuse highly corrosion-resistant tantalum metal into the surface of conventional stainless steels and superalloys to improve the acid corrosion resistance of components used in precision propellant transfer valves. The ability to harden the surface via partial or full conversion to tantalum nitride has been demonstrated. This tantalum surface technology has been applied to industrial applications such as heat exchangers, rupture disks, instrumentation, pumps, valves, fittings, and custom equipment. In other work, Ultramet developed a rapid, low-cost method of applying oxide, nitride, and multiple-layer interface coatings to carbon and silicon carbide fibers at temperatures as low as 200°C, thus avoiding problems associated with fiber damage during conventional high temperature deposition. Coatings have been effectively applied to individual fiber tows as well as woven and braided fabric, and continuous coating reactors have been demonstrated. Corrosion and interface coating processing and test results will be presented.

11:50 AM

(ICACC-S2-018-2017) Environment Stability and Oxidation Behavior of HfO_2-Si and YbGd(O) Based Environmental Barrier Coating Systems for SiC/SiC Ceramic Matrix Composites

D. Zhu*[1]; S. Farmer[1]; T. R. McCue[1]; B. J. Harder[1]; J. Hurst[1]; 1. NASA Glenn Research Center, USA

Ceramic environmental barrier coatings (EBC) and SiC/SiC ceramic matrix composites (CMCs) will play a crucial role in future aircraft propulsion systems because of their ability to significantly increase engine operating temperatures, improve component durability, reduce engine weight and cooling requirements. Advanced EBC systems for SiC/SiC CMC turbine and combustor hot section components are currently being developed to meet future turbine engine emission and performance goals. One of the significant material development challenges for the high temperature CMC components is to develop prime-reliant, environmental durable environmental barrier coating systems. In this paper, the durability and performance of advanced NASA HfO_2-Si and YbGdSi(O) EBC bond coat – top coat systems for SiC/SiC CMC have been summarized. The high temperature thermomechanical creep, fatigue and oxidation resistance have been investigated in the laboratory simulated high-heat-flux environmental test conditions. The advanced NASA EBC systems showed promise to achieve 1500°C temperature capability, helping enable next generation turbine engines with significantly improved engine component temperature capability and durability.

12:10 PM

(ICACC-S2-019-2017) High Temperature Environmental Barrier Coatings Deposited via Plasma Spray- Physical Vapor Deposition (PS-PVD)

B. J. Harder*[1]; D. Zhu[1]; 1. NASA Glenn Research Center, USA

Next generation turbine engines are expected to operate with hot section components at temperatures nearing 1482°C, which will necessitate new environmental barriers and bond coats. To deposit these advanced environmental barrier coating (EBC) systems, Plasma Spray- Physical Vapor Deposition (PS-PVD) can be utilized to tailor microstructures and compositions to optimize performance. Advanced composite bond coats of Si-HfO_2 with a $Yb_2Si_2O_7$ environmental barrier topcoat were deposited on SiC using PS-PVD processing. Coating systems were tested in isothermal and laser gradient heating conditions ranging from 1315 to 1482°C to determine the upper temperature limit of the bond coat. Samples were also thermally cycled for 100 hours in a steam environment. Coating composition and crystal structure were tracked with X-ray diffraction and microstructure was analyzed with electron microscopy.

S3: 14th International Symposium on Solid Oxide Fuel Cells (SOFC): Materials, Science and Technology

Solid Oxide Electrolysis Cell

Room: Crystal
Session Chair: Ayhan Sarikaya, Saint-Gobain

8:30 AM

(ICACC-S3-009-2017) Solid Oxide Fuel Cell Based Technologies for Power Generation and Chemical Production (Invited)

P. Miranda*[1]; N. Minh[2]; 1. Coppe-Federal University of Rio de Janeiro, Brazil; 2. University of California, San Diego, USA

Solid oxide fuel cells (SOFCs) have been developed for clean and efficient power generation from a variety of fuels. The SOFC has also served as the basis for the development of technologies (such as solid oxide electrolysis cells or SOECs and SOFC type cells for cogeneration of electricity and chemicals, referred to as CSOFC) to produce hydrogen and other chemicals. The SOFC offers a number of advantages in terms of cell/stack design, fabrication and operation characteristics (design flexibility, multiple cell fabrication options, and operating temperature choices). The SOFC also presents several technical challenges (such as limited material selections, increased undesirable chemical interactions between components and certain operation restrictions). Significant technological progress has been made in the past several years in the development of SOFCs, SOECs and CSOFCs for practical applications. This paper provides an overview of the status of SOFC-based technologies and discusses selected challenges in the areas of efficiency, reliability and cost to move the technologies toward commercialization.

9:00 AM

(ICACC-S3-010-2017) Critical Evaluation of Dynamic Reversible Chemical Energy Storage with High Temperature Electrolysis (Invited)

D. McVay*[2]; J. Brouwer[2]; F. Ghigliazza[1]; 1. SOLIDpower, Italy; 2. University of California, Irvine, USA

A solid oxide electrochemical cell is capable of operating reversibly in both fuel cell and electrolysis modes with high efficiency compared to low temperature alternatives. However, thermodynamic integration challenges exist for a system that can support a reversible solid oxide fuel cell stack (RSOFC). Key system components must be kept within a given temperature range throughout different modes of operation. A six cell short stack with Ni/YSZ anode-supported cells, YSZ electrolyte, and LSCF oxygen electrode with a GDC interlayer was analyzed experimentally. Results from testing were used to verify a physical model of a 50 kW RSOFC system in order to study thermal integration challenges under dynamic operating conditions that span three modes of operation: fuel cell, electrolysis, and standby. Preliminary results suggest that the three modes of operation can be thermally sustained using novel control strategies specifically in the electrolysis and standby modes. These modes are required to enable RSOFC technology to be dynamically dispatched in a manner that complements intermittent renewable power generation, such as wind and solar power. Using measured renewable power dynamics, the dynamic RSOFC model was able to show load-following operation for realistic demands of RSOFC systems as applied to the electric grid that deploys high levels of renewable power.

9:30 AM

(ICACC-S3-011-2017) Electrochemical In-situ Upgrading of Bio-oil Using Solid Oxide Electrolysis Stack

S. Elangovan*[1]; D. Larsen[1]; E. Mitchell[1]; J. Hartvigsen[1]; J. Mosby[1]; B. Millett[1]; J. Elwell[1]; P. Billen[2]; S. Spatari[2]; D. Santosa[3]; 1. Ceramatec, Inc., USA; 2. Drexel University, USA; 3. Pacific Northwest National Lab, USA

Biomass can be converted to liquid fuels via bio-oil production by fast pyrolysis. Typical pyrolyzer operation produces a mixture of biphasic, oxygenated organic compounds. Only the organic phase is processed by hydrodeoxygenation, leaving behind valuable carbon-containing material in the aqueous phase. A deoxygenation process using solid-state, oxygen conductor based electrochemical cell is under evaluation. The cell is operated at 500 – 550°C to match the pyrolysis temperature for both physical and process integration of the two operations. The electrolysis process will remove oxygen from the oxygenated organic molecule as well from steam to produce hydrogen in-situ. Thus, no external hydrogen is needed for deoxygenation, allowing for a distributed, small scale integrated upgrading unit. Mixtures of model compounds were tested using button cells and short stacks. The product from the electrochemical cell contained a suite of compounds with significantly lower oxygen content. Integrated testing of short stacks is planned at the Pacific Northwest National Laboratory. A slip stream from the pyrolyzer will be directly fed to the stack. The collected product will be analyzed for deoxygenated product species. Test results using model compounds and pyrolysis vapor will be reported. Acknowledgment: This material is based upon work supported by the Department of Energy under Award Number DE-EE0006288.

9:50 AM

(ICACC-S3-012-2017) Solid Oxide Electrolysis Development for Oxygen Production by In-situ Resource Utilization on Mars

J. Hartvigsen[1]; S. Elangovan[1]; J. Elwell*[1]; D. Larsen[1]; T. Hafen[1]; E. Mitchell[1]; L. Clark[1]; T. Meaders[1]; 1. Ceramatec, Inc., USA

Space exploration is among the most challenging of human endeavors, requiring a logistics supply not only of food, fuel and tools, but also sophisticated environmental control with atmosphere revitalization and propellant oxidant production. The cost of launching initial mass into low earth orbit is said to make these supplies worth their weight in gold. The transit, entry, decent and landing on Mars will multiply the mass specific value of supplies landed on Mars. For decades the idea of exploiting local resources, (in-situ resource utilization or ISRU) has been accepted as a foundational concept in space mission planning, but no such system has been demonstrated to date. In 2014, NASA announced the experiment suite for the Mars 2020 mission, a curiosity class Mars rover, including MOXIE, the Mars Oxygen ISRU Experiment. This first non-terrestrial IRSU experiment will demonstrate initial feasibility of solid oxide electrolysis of Martian atmosphere CO_2 as a means of producing oxygen for propellant oxidant. Ceramatec is developing the solid oxide electrolysis stack for the MOXIE program. This paper will summarize Ceramatec's efforts and successes in addressing the unique and challenging constraints associated with operation on Mars using dry CO_2. Acknowledgment: This material is based upon work supported by NASA through JPL's prime contract under JPL subcontract number 1515459.

Thermodynamic Stability

Room: Crystal
Session Chair: S. Elangovan, Ceramatec, Inc.

10:30 AM

(ICACC-S3-013-2017) Thermodynamic Stability Maps for the $La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_{3\pm\delta}$ –CO_2 –O_2 System for Application in Solid Oxide Fuel Cells

S. Darvish*[1]; Y. Zhong[1]; 1. Florida International University, USA

Thermodynamic and electrochemical predictions on the formation of secondary phases in CO_2 containing atmosphere on the $La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_{3\pm\delta}$ (LSCF-6428) surface have been carried out utilizing the CALculation of Phase Diagram (CALPHAD) approach. The effects of temperature, CO_2 partial pressure, O_2 partial pressure as well as the cathode composition on the formation of secondary phases have been investigated and correlated with the previous investigations in the literature. It is predicted that $SrCO_3$ has the possibility to form on the surface as a result of CO_2 exposure to the system. In addition, it is seen that LSCF-6428 experiences higher degradation due to the larger amount of segregated phases than LSM-20 at the same operating condition.

10:50 AM

(ICACC-S3-014-2017) A thermodynamic approach on the chemical stability of lanthanum chromite-based perovskite with yttrium-stabilized zirconia

H. Sabarou*[1]; Y. Zhong[1]; 1. Florida International University, USA

This study is addressing the chemical stability of lanthanum chromite-based perovskite with yttrium-stabilized zirconia (YSZ) as a dual phase composite of oxygen transport membrane (OTM). The chemical stability of the system has been studied separately through two aspects: experimental tests and the CALPHAD approach. The experimental tests include $La_{0\cdot8}Sr_{0.2}Cr_xFe_{1-x}$ (x=0.3,0.5,0.7) synthesis by sol-gel, mixing the perovskite with YSZ, and heat treatment under reducing atmosphere. The chemical stability and morphology of resultant materials have been examined by XRD and SEM. Thermodynamic calculations with similar processing conditions have been separately conducted to reveal the dependence of the structure to Fe concentration variations. Both approaches prove that the chemical stability of dual phase composite OTM including LSCrF and YSZ is mainly depending on Fe concentration as B-site dopant. This study proposes a well-proved thermodynamic model to predict a trend of the chemical stability variations with regard to change in composition, atmosphere, and temperature.

11:10 AM

(ICACC-S3-015-2017) Reactivity of SrO/Sr in SOFCs

X. Yin*[1]; R. Spatschek[1]; L. Bencze[2]; L. Singheiser[1]; 1. Forschungszentrum Juelich, Germany; 2. Eötvös Loránd University, Hungary

$(La,Sr)(Co,Fe)O_3$ is very common as cathode material in SOFC applications. Different compositions of this type of materials have been developed and used in SOFCs. SrO/Sr in this type of cathode material is very reactive to form secondary phase with other oxides, which affect microstructures and properties of the cathode material, CGO layer and ZrO_2-based electrolyte. It is well known that SrO from cathode material will react with volatile CrO_x as well as $CrO_x(OH)_y$, which results in SOFC degradation due to formation of $SrCrO_4$ at the cathode surface. A further process which results in SOFC degradation is the formation of $SrZrO_3$ precipitates in ZrO_2 based electrolyte. To prevent this $SrZrO_3$ formation, diffusion barrier layer based on CGO is applied between the cathode and electrolyte. High resolution investigations indicate that in case of porous CGO layer the formation of $SrZrO_3$ is still possible. Thermodynamic calculations show that in presence of humidity in the air volatile Sr-hydroxides can be formed, which can diffuse through porous CGO layer and react with ZrO_2 based electrolytes to form $SrZrO_3$ precipitates. The Sr-hydroxides pressure depends on temperature and water vapor pressure and can be in the same order of magnitude compared with CrO_x and $CrO_x(OH)_y$ vapor pressures. This work will outline the current understanding of possible poisoning and degradation of SOFCs by volatile Cr-compounds as well as by volatile Sr-hydroxides.

11:30 AM

(ICACC-S3-016-2017) Weight Loss Mechanism of $(La_{0.8}Sr_{0.2})_{0.98}MnO_{3\pm\delta}$ During Thermal Cycles

S. Darvish*[1]; Y. Zhong[1]; 1. Florida International University, USA

A new insight to weight loss mechanism of $(La_{0.8}Sr_{0.2})_{0.98}MnO_{3\pm\delta}$ (LSM-20) using the La-Sr- Mn-O thermodynamic database is proposed with respect to the compound energy formalism (CEF) model, utilizing the CALculation of Phase Diagram (CALPHAD) approach. Quantitative Brouwer diagrams for LSM-20 are plotted according to the defect chemistry analysis of perovskite phase and the charge disproportionation for temperature range of 600-1400°C. The simultaneous DSC/TGA (SDT) experiments on weight loss show the thermodynamic and kinetic effects during the thermal cycles. It is found that the Schottky defects dominate during the thermal cycles for LSM-20.

S4: Armor Ceramics

Materials Modeling

Room: Coquina Salon E
Session Chair: Sikhanda Satapathy, U. S. Army Research Laboratory

8:30 AM

(ICACC-S4-010-2017) Validation and optimization of boron carbide interatomic potentials using XRD (Invited)

S. Coleman*[1]; E. Hernandez-Rivera[1]; D. Taylor[1]; J. Synowczynski-Dunn[1]; M. Tschopp[1]; 1. US Army Research Laboratory, USA

The complex atomic structure and bonding of the various boron carbide polymorphs are a challenge to represent using classical interatomic potentials. ReaxFF potentials have shown promising results; however, identification of the subtle geometric shifts caused by asymmetric atomic relaxation is not clearly discernable in real space. Virtual diffraction characterization of atomistic simulations is opening new routes for model validation and enables for experimental comparison. In this work, x-ray diffraction patterns computed over various boron carbide polymorphous using both density functional theory and classical atomistic simulation data identify even subtle atomic realignments. Comparison metrics between computed x-ray diffraction data are used to help optimize candidate ReaxFF parameterizations generated from surrogate models.

9:00 AM

(ICACC-S4-011-2017) Nanotwins in Boron Carbide and Related Materials (Invited)

Q. An*[1]; 1. University of Nevada Reno, USA

The twin structures and their roles in mechanical properties are extensively examined and well understood for metals and alloys. However, for covalent solids, their structures and response to applied stress are not established. Here we characterize the nanotwins structures in boron carbide (B_4C) and related materials such as boron suboxide (B_6O) and beta boron using quantum mechanics (QM) simulations coupled with transmission electron microscopy (TEM). The "asymmetric twins" have been observed and characterized in B_4C, which arises from the interplay of stoichiometry, atomic positioning, twinning, and structural hierarchy. While the negative interfacial energy in the twinned B_6O and β-B leads to the discovery of new phases of τ-B_6O and τ-B. Then deformation responses of these nanotwins are examined by QM simulations showing the strengthening effects for B_4C and softening effects for both B_6O and β-B, which are are validated by nano-indentation experiments.

The strengthening mechanism in B_4C is the presence of twins suppresses the stress decrease as the B-C bond between icosahedral clusters breaks, while this mechanism does not exist in B_6O and β-B.

9:30 AM

(ICACC-S4-012-2017) Molecular-dynamic modeling of propagation of shock wave in porous ceramic materials

I. V. Kartuzov*[1]; V. L. Bekenev[1]; V. V. Kartuzov[1]; 1. IPMS, Ukraine

The previous work of the authors on computer modelling of shock wave propagation in SiC presented the molecular-dynamic investigation of propagation of shock waves of various intensities in silicon carbide ceramic. It was found out that starting from the certain level a velocity of shock wave does not depend on intensity of loading. This effort is to present the results of systematic investigation of propagation of shock waves in porous SiC samples and ceramic materials on the base of high-boron compounds (AlB_{12}, etc.). The samples with a low porosity (1-3%) and with various geometry and with porosity from 9 to 17% were considered. A detailed discussion of microscopic mechanisms of fracture of the investigated materials under the shock wave action is presented.

Materials Characterization and Mechanical Behavior I

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

10:20 AM

(ICACC-S4-013-2017) Polymorph-level variability and amorphization resistance of boron carbide: Experiments and atomistic modeling

A. P. Awasthi*[1]; C. Kunka[1]; G. Subhash[1]; 1. University of Florida, USA

Motivated by the fact that boron carbide occurs in numerous polymorphs, and TEM observations of random distribution of amorphized regions, this study focuses on analyzing a large number of polymorphs with the goal of collating improved properties for enhanced performance. Using DFT-level simulations, we present analysis of a wide range of permissible atomistic configurations of boron carbide polymorphs and demonstrate links between bonding environments and DFT-Raman spectra. Homogeneous deformation studies performed for both hydrostatic as well as shear modes provide new information about bonding behavior and Raman shifts. Additionally, computational predictions of Raman shifts are compared with experimental observation obtained from line-scan in-situ Raman based on four point bend test. Focus is given to addressing cage spaces inherent in the structure to improve deformation response and eventually obtain amorphization resistance. Different foreign atoms are studied for their relative thermodynamic stability and performance as cage space dopants. The relative performance of different polymorphs is investigated further by constructing molecular dynamics systems using the ReaxFF potential. Response to homogeneous deformation and shock are investigated for different polymorphs, establishing polymorph-level tailorability in boron carbide.

10:40 AM

(ICACC-S4-014-2017) Raman-Active Constituents of Boron Carbide

C. Kunka*[1]; A. P. Awasthi[1]; G. Subhash[1]; 1. University of Florida, USA

Fabricated boron carbide comprises numerous crystal structures with unique atomic arrangements and possibly different stoichiometries. However, no preexisting theoretical or experimental method comprehensively discerns the identity or abundance of these crystal structures. With a recently revised nomenclature and established knowledge of polymorph equivalence, the current study employs quantum-mechanical simulations and a new superposition technique to suggest the Raman-active constituents of an experimental sample. By modeling α-boron and fifty-five Raman-active B_4C polymorphs, most dominant Raman peaks in an experimental spectrum have been matched to specific crystal structures. The presence of $(B_{11}C_p)CBC$, which is the only commonly suggested Raman-active polymorph, is confirmed. The abundance of α-boron in fabricated boron carbide is correlated to the intensity of the 826-cm^{-1} peak. Finally, three-carbon-icosahedra structures, which have not been previously studied due to assumptions on stability, are shown to account for some of the Raman peaks not expressed by $(B_{11}C_p)CBC$ or α-boron.

11:00 AM

(ICACC-S4-015-2017) Grain Boundary Characterization of High-Purity Boron Carbide with Al_2O_3 and SiO_2 Additives

C. J. Marvel*[1]; K. D. Behler[2]; S. D. Walck[2]; J. LaSalvia[2]; M. P. Harmer[1]; 1. Lehigh University, USA; 2. US Army Research Laboratory, USA

The main purpose of this study was to utilize analytical methods to detect and understand the influence of Al, Si, and O excess on the structure and chemistry of grain boundaries in commercial boron carbide powders densified with Al_2O_3 and SiO_2 additives. Achieving full densification of pure boron carbide powders is an enduring challenge and sintering additives are needed to fully densify boron carbide in order to achieve the optimal physical and mechanical properties for armor applications. High-purity commercial boron carbide powders were hot-pressed with 5 vol.% of Al_2O_3 and SiO_2 additives at 1900°C and 2000°C for 3 hours. For this study, dense B_4C, B_4C-Al_2O_3, B_4C-SiO_2, and B_4C-Al_2O_3-SiO_2 materials were processed and analyzed. Aberration-corrected scanning transmission electron microscopy (AC-STEM) was used to characterize the microstructures, including high-angle annular dark field (HAADF) imaging to image the grain boundaries on the atomic scale and X-ray energy dispersive spectroscopy (XEDS) to determine grain boundary chemistry. Overall, it was observed that the systems with Al_2O_3 and Al_2O_3-SiO_2 mixtures exhibited the highest density, and consistently exhibited primarily Si and to a lesser extent Al grain boundary segregation. Experimental procedures and results will be presented.

11:20 AM

(ICACC-S4-016-2017) Meso-scale microstructural flaw quantification in boron carbide using microCT

C. Moorehead*[2]; J. M. Sietins[1]; J. Swab[1]; 1. US Army Research Laboratory, USA; 2. Drexel University, USA

Failure in brittle materials is governed, in part, by the specific microstructure of that material, with heterogeneous microstructures generally leading to reduced performance. In ceramics used for armor, an increase in compressive strength can be an indicator for improved ballistic performance. In hot-pressed boron carbide (B_4C) ceramic plates, failure is often attributed to carbonaceous flakes observed on the fracture surfaces of failed specimens. The density and distribution of these flaws is believed to influence the ballistic performance. Therefore, it is of interest to improve the quantification of meso-scale microstructural flaws so that strategies can be developed to mitigate them. This work is focused on characterization of pressure-aided densification (PAD) B_4C compression specimens using high-resolution micro-computed tomography (microCT). Large clusters of non-B_4C agglomerates, up to 125 μm in diameter, were observed in addition to the typical smaller carbonaceous flakes and aluminum-based phases. Agglomerate size distribution, orientation, morphology, and spacing was quantified both parallel and perpendicular to the pressing direction. This information will be used to inform future testing on compressive strength and ballistic performance in order to investigate the effect of microstructure on the ballistic performance of ceramics.

11:40 AM

(ICACC-S4-017-2017) Diffusion bonding of Boron Carbide to Silicon to study the rate of diffusion of Silicon into Boron Carbide and its effect on mechanical properties

C. Besnard*[1]; A. Bhowmik[1]; T. Giovannini[2]; Y. Patel[2]; P. Brown[3]; F. Giuliani[2]; L. Vandeperre[1]; 1. Imperial College London, United Kingdom; 2. Imperial College London, United Kingdom; 3. Defence Science and Technology Laboratory Porton Down, United Kingdom

Boron carbide is used as a light weight ballistic protection material due to its excellent properties: a high hardness only below that of diamond and cubic nitride, a low density, a high Young's modulus and a high Hugoniot Elastic Limit. However, it has been found that at impact velocities in excess of 900 m.s^{-1} the material loses its strength. This strength loss is currently attributed to the formation of amorphous bands in the material. A limited number of theoretical and small scale studies have indicated that doping boron carbide with silicon reduces the tendency for amorphisation of boron carbide. For large scale production of silicon doped boron carbide powders, diffusion of silicon into boron carbide will be required and therefore in this paper the diffusion of silicon in boron carbide is investigated using diffusion couple experiments in which a wafer of silicon is placed between 2 boron carbide discs and heat treated at temperatures between 1200°C and 1800°C. Afterwards the evolution of composition as a function of distance from the interface is determined to estimate the extent of diffusion and the effect of Si doping on mechanical behaviour is determined by Raman analysis of indentations made along the Si concentration gradient. From this we can confirm Si doped eliminates the amorphisation due to nano indentation.

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

Thermal Energy Conversion and Energy Storage III

Room: Tomoka A

Session Chair: Olivier Guillon, Forschungszentrum Juelich

8:30 AM

(ICACC-S6-009-2017) Quantifying defects in battery materials (Invited)

M. Casas-Cabanas*[1]; 1. CIC energiGUNE, Spain

Disruptions in periodicity are extremely common in all kinds of solid materials and can manifest in multiple ways. Indeed solids often exhibit point defects (such as antisites, interstitials or vacancies), stacking faults, grain boundaries, pores, intergrowths or microstrains, just to name a few. While some technologies require the use of crystals that are nearly perfect, polycrystalline solids are usually the norm in most applications. This is the case of electrochemical storage systems, for which a proper understanding of the underlying thermodynamics and kinetics unavoidably requires the framework of both structure and microstructure. Diffraction techniques (XRD, NPD) are typically used to characterize average structural features of functional materials. Thanks to the recent development of advanced tools for diffraction data treatment (either implemented in Rietveld refinement programs such as FullProf, or available as independent refinement programs such as FAULTS), powder diffraction can now be also used for the quantitative characterization of extended defects. Several examples corresponding to different battery materials will be shown to illustrate how, besides classic structural determination, quantitative information regarding microstructural features such as anti-phase domains, stacking faults, twinning or intergrowths can now be extracted from diffraction data to establish correlations with materials' properties.

9:00 AM

(ICACC-S6-010-2017) Impacts of anionic vacancies on the local and electronic structures of Iron-based Oxyfluoride Electrodes (Invited)

D. Dambournet*[1]; 1. Sorbonne Universités, France

Iron fluoride provides very appealing redox properties such as a high theoretical capacity (237 mAh/g per lithium), a relatively high voltage (around 3.1-3.3V), low cost and a good thermal stability. However, metal fluorides display large band gaps, which means that in their pure form these materials are electronic insulators. Over the years, several approaches have been developed to overcome such a limiting electronic character including the synthesis of carbon/metal fluoride composite or oxygen incorporation. Recently, we have shown that introducing anionic vacancies in an iron oxyfluoride cathode material significantly improves the intercalation properties vs. Li. In this presentation, we will first show how to design an iron oxyfluoride compound featuring anionic vacancies starting from a hydrate phase $FeF_3.3H_2O$. Then, we will show that the controlled introduction of defects can be employed in the design of new battery electrode materials with enhanced properties. Finally, through experimental and computational tools, we will attempt to rationalize the observed enhanced electrochemical properties by investigating the impacts of the presence of anionic vacancies on both the local and electronic structures of these iron-based oxyfluoride electrodes.

9:30 AM

(ICACC-S6-011-2017) Advanced Analysis of Nonequilibrium Phase Transition between $LiFePO_4$ and $FePO_4$ during Battery Operation (Invited)

Y. Orikasa*[1]; 1. Ritsumeikan University, Japan

$LiFePO_4$ is a promising cathode material for high power lithium-ion batteries. The origin of the high rate performance in $LiFePO_4$ can give design principles for further development of electrode materials. Under high rate cycling, a metastable Li_xFePO_4 (x = 0.6–0.75) (L_xFP) forms, which might acts as a buffer layer between LFP and FP. However, the detailed reaction mechanism has not been understood due to short lifetime of L_xFP phase. To investigate the phase transition mechanism, with respect to L_xFP phase, we measured operando time-resolved X-ray diffraction (TR-XRD) at various temperatures. Typical two-phase reaction between the LFP and FP phases is observed in the charge-discharge at 25°C and rate of 1C. However, another diffraction peak is observed in the first charging at −5°C. This result indicates the formation of the L_xFP phase in the first charging. During the discharge reaction, the diffraction peaks of FP phase disappear and the peak of the L_xFP phase at 19.4° appears. These results suggest that the phase transition in the first charge is different from that in the first discharge. The difference of phase transition kinetics between LFP/L_xFP and L_xFP/FP causes the initial irreversible capacity at high rate, and/or low temperature conditions.

Thermal Energy Conversion and Energy Storage IV

Room: Tomoka A

Session Chair: Valérie Pralong, CNRS

10:20 AM

(ICACC-S6-012-2017) Controlling and characterizing defects in electrochemically active MnO_2 nanosheet assemblies (Invited)

S. Misture*[1]; P. Metz[1]; P. Gao[1]; T. Hey[1]; 1. Alfred University, USA

2-D birnessite MnO_2 nanosheets were prepared via exfoliation of high-perfection starting microcrystalline powders and reassembled into 3-D porous structures. X-ray total scattering was teamed with X-ray spectroscopy and related tools to probe both the mesostructure and the atomic defects of MnO_2 nanosheet assemblies perturbed by different aqueous treatments. We define a 'surface Frenkel' defect as critical in controlling the electrochemical charge storage, where the capacitance increased from about 150 F/g to over 350 F/g when we intentionally reduced MnO_2 nanosheets to form a fixed quantity of trivalent Mn ions. Notably, the charge transfer resistance decreased from ~18 Ω to ~3 Ω correspondingly. X-ray PDF studies show unequivocally that the reduced Mn ions form surface Frenkel defects, wherein the Mn ions shift from locations in the planar nanosheet to the nanosheet surface, leaving a Mn vacancy in the sheet. Implications of the use of these characterization tools for study of related disordered 2-D solids and our resulting understanding of atomic scale defects in these 2-D entities will be considered.

10:50 AM

(ICACC-S6-013-2017) Humidity propelled washable and wearable energy storage device using carbon nanotubes (Invited)

C. Subramaniam*[1]; M. Jha[1]; M. Singh[1]; D. Banerjee[1]; 1. Indian Institute of Technology Bombay, India

Wearable devices and technologies constitute $10 billion worldwide market today, with primary applications in fields ranging from military to medical care. Emergence of such a technology demands light-weight, portable, mechanically robust and washable energy storage devices. Herein, convention energy sources such as Li-ion batteries are incompatabile and pose serious health hazard. To overcome this challenge, our current efforts focus towards developing the energy storage component through approaches that allow seamless integration onto clothing and fabrics. Towards this end, we demonstrate a solid-state energy storage device based on single walled carbon nanotubes. Contrary to existing batteries and supercapacitors that degrade tremendously when exposed to higher humidity levels, the device demonstrated here exhibits superior energy storage capability at higher levels of humidity. Further, we demonstrate excellent washability and mechanical robustness of the device, based on the facile packaging technology developed. Specifically we achieve superior performance (energy density of 0.25 mW/cm^2 and power denisty of 51 W/cm^2) of the device compared to any other previously reported, in the context of wearable energy sources. Realising this involved the symbiotic combination of multiple strategies suited for direct and facile integrating on to clothing and fabrics.

11:20 AM

(ICACC-S6-014-2017) Unique crystallization behavior of $Na_2MnP_2O_7$ glass

T. Honma*[1]; M. Tanabe[1]; C. Akatsuka[1]; T. Komatsu[1]; 1. Nagaoka Univeristy of Technology, Japan

$Na_2MnP_2O_7$ is a one of candidates for cathode materials in sodium ion batteries. We are proposing novel fabrication process so-called 'glass-ceramics route' to obtain $LiFePO_4$, $LiVOPO_4$, $Na_2FeP_2O_7$ and so on in previously. In order to clarify the mechanism of crystallization behavior of $Na_2MnP_2O_7$ glass. We examined conventional heat-treatment, morphological characterization by means of electron microscope observation, as well as electrochemical properties. Suface crystallization tendency was confirmed from bulk $Na_2MnP_2O_7$ precursor glass. And we successfully obtained metastable $Na_2MnP_2O_7$ via glass ceramics route.

11:40 AM

(ICACC-S6-015-2017) Design and Processing of Ceramics for Low Cost Sodium Batteries

M. Stelter*[1]; R. Weidl[1]; M. Schulz[1]; 1. Fraunhofer IKTS, Germany

Low cost batteries for Gigawatt scale stationary energy storage can be manufactured from Na-β-Alumina ceramics. Candidate battery cells for this applications are Sodium-Nickel-Chloride (NaNiCl) and Sodium-Sulphur (NaS) batteries. To achieve a total cost of the battery cell of under 100 $/kWh, both the ion conducting ceramic material as well as the design of the ceramic component need to be optimized for mass production. IKTS has achieved several breakthroughs in the shaping of β-Alumina components, such as cold plastic extrusion and tape casting. These components are designed according to a set of rules derived from cost and manufacturing requirements. It is discussed, how electrochemical component design and mechanical requirements interact with shaping methods and the route to β-Alumina phase formation. Several problems need to be solved for high-throughput manufacturing of Na-β-Alumina, such as the very high pH value in the feedstock, the automated shaping of tubes with one end closed and the phase stability during sintering. Material qualities with a phase content of > 96% β“ and conductivities of > 240 mS/cm have been achieved from extrusion at IKTS. Extruded ceramic tubes and sheets have been investigated electrochemically with very promising results. They have undergone electrochemical and thermomechanical testing in IKTS designed real-world battery cells based on Nickel cathodes (NaNiCl design).

S7: 11th International Symposium on Nanostructured Materials: Functional Nanomaterials and Thin Films for Sustainable Energy Harvesting, Environmental and Health Applications

Smart Sensors I

Room: Coquina Salon A

Session Chairs: Yakup Gönüllü, University of Cologne; Sanjay Mathur, University of Cologne

8:30 AM

(ICACC-S7-001-2017) Nano-material based stretchable bio-environmental sensors driven by integrated micro-supercapacitor array (Invited)

J. Ha*[1]; 1. Korea University, Republic of Korea

Recently, interest in stretchable electronics has dramatically increased in accordance with the increasing demand on the mechanically stable skin-attachable and implantable sensor devices under body movements. At the same time, the need for integrated high-performance stretchable energy storage devices for wireless operation has been increased. In this work, we demonstrate the successful operation of stretchable bio-environmental sensors by integrated high performance micro-supercapacitors (MSCs). In order to enhance the performance, a strain sensor of fragmentized graphene foam and two gas sensors with a mixed film of MWNTs and SnO_2 nanowires, and patterned graphene were fabricated. As an energy storage device, an array of solid-state MSCs consisting of spray-coated MWNTs electrodes and patterned ion-gel electrolyte was fabricated. After neck-attachment of the integrated system of bio/environmental sensors and the MSCs on a single stretchable substrate, bio-signals of arterial pulse, voice, swallowing of saliva, and body motion were detected using the stored energy of MSCs. In addition, the gas sensors driven by the integrated MSCs showed a fast response to NO_2 gas exposure under stretching up-to 50% without any deterioration in sensing properties.

9:00 AM

(ICACC-S7-002-2017) Ceramic Gas Sensors to Oxide Nano-heterostructures: A Materials Design (Invited)

S. Akbar*[1]; D. Miller[1]; 1. The Ohio State University, USA

This talk summarizes R&D efforts in the author's laboratory from the development of ceramic-based gas sensors to the fabrication of ordered oxide nanostructures exploiting intrinsic material properties. Our focus has been on the development of high-temperature gas sensors specifically for combustion processes. The underlying theme has been the use of materials science and chemistry to promote high-temperature performance with selectivity. Our recent focus is on surface modification techniques for the fabrication of oxide nanostructures that are highly scalable and do not require use of lithography. One such process creates crystallographically oriented nanofiber arrays of single crystal TiO_2 in H_2/N_2 environment. We have succeeded in converting the 1D TiO_2 nano-fiber grown by thermal oxidation to nano-dendritic titanates by hydrothermal treatment. We developed yet another interesting nano-structure (nanoislands and/or nanobars) during thermal annealing of an oxide on top of another oxide substrate that self-assembles along the softest elastic direction of the substrate. Finally, we have observed a novel step faceting phenomenon indicating that doping can alter the step morphology that opens a new avenue for surface patterning that has huge implications in applications such as adsorption, catalysis, sensing and biomedical applications.

9:30 AM

(ICACC-S7-003-2017) Highly selective and sensitive electrochemical gas sensors based on p-n semiconducting oxide heterostructures (Invited)

C. Sun*[1]; 1. National Energy Technology Lab, USA

Detection of trace gases are relevant for environmental, combustion and health-related applications. Resistive semiconducting metal oxide sensing platforms are extensively studied for trace gas detection. Two important aspects of gas sensing are enhancing sensitivity and selectivity. In this talk, we present a sensor platform with n-type and p-type semiconducting oxides placed side by side with a shared interface. The adjacent placement of the oxides allows for ease of variation of the amount of oxide to be included for making the resistance measurements in the presence of analyte gas. Our focus was to develop a selective electrochemical sensor platform with ppb sensitivity, with potential applications in breath analysis. Fractional exhaled nitric oxide (NO) is associated with inflammatory conditions of the airways in patients with asthma. N-type WO_3 and p-type Cr_2O_3 in adjacent alignment was examined as a possible design for NO-selective sensor. Studies focused on resistance measurements toward NO and CO at 300°C. This sensor design exploits the different majority carriers in these two semiconducting oxides, and the response to CO and NO can be continuously varied by choosing to include different fractions of the metal oxide.

9:50 AM

(ICACC-S7-004-2017) Plasma deposition of metal oxide layers for selective gas sensing application

Y. Gönüllü*[1]; S. Mathur[1]; T. Fischer[1]; J. Leduc[1]; 1. University of Cologne, Germany

In the last decades, atmospheric pollution in urban areas has achieved to critical levels. Processes involving combustion in aircrafts, energy and power production and automobile engines as well as through industrial settlement are the main sources of the pollution. Detection and control of the emission relies on the development of the precious and selective gas sensors. It is known that the transition metal oxides such as SnO_2, TiO_2, WO_3, MoO_3, Ga_2O_3 and Nb_2O_5 are used as sensing electrodes in such chemical gas sensors due to their semiconducting and electrochemical properties. For instance $TiO_{2,}$ is non-toxic and low cost and has excellent chemical stability. It can pose semi-conductive properties on doping, and hence is one of the most important transition metal oxides for this purpose. Nevertheless, TiO_2 based gas sensors still need improvement for the achievement of high-temperature sensitivity, stability or efficiency. An electrochemical reaction occurs, as a gas species, either oxidizing or reducing, reaches on the semi-conductive metal oxide surface. Thus, the quantity of this reaction relies on the availability of the surface area of the metal oxide. In this study, we performed plasma deposition of FeSiOx layer for an alternative gas sensing layers. The obtained results showed that FeSiOx has a selectivity towards NH_3 compared to other gases.

10:10 AM

(ICACC-S7-005-2017) Scalable Nanostructure Integration for Multimode Gas Sensing at High Temperature (Invited)

P. Gao*[1]; 1. University of Connecticut, USA

Using a combination of vapor and solution phase deposition methods, scalable heterostrutured nanowire arrays have been successfully fabricated composed of metal oxide nanowire cores ~100-300 nm in diameter, ~1-30 nm thick shells of perovskite, metal oxide and noble metal nanoparticles on ceramic planar substrates. The nanowire structure features tuned composition, density, geometry, and dimensions through varying the deposition temperature, concentration, pH, substrate temperature, time, etc.. Multi-mode gas sensing platform has been achieved at elevated temperature to include photocurrent, electrical resistance and electrochemical impedance modes. Depending on the selections of comprising materials, dimensions and post-treatment processes, trace amount decoration of perovskite, metal oxide and noble metal nanoparticle shells may enable formations of catalytic filters, sensitizers and hetero-junctions that can drastically enhance nanosensor performance in both oxidative and reductive gas atmospheres. Specifically the materials-synergy effect in Ga_2O_3/perovskite, and ZnO-Au nanowire array sensors are discussed toward CO and NO_2 sensing. Toward mixture gas conditions, this new type of multi-mode nanowire array sensors allows selective and sensitive detection of multiple species in a single-device platform.

Smart Sensors II

Room: Coquina Salon A
Session Chairs: Chenhu Sun, National Energy Technology Lab;
Pu-Xian Gao, University of Connecticut

10:30 AM

(ICACC-S7-006-2017) Metal oxides nanophotonic and plasmonic applications in chemical sensing (Invited)

G. Faglia*[1]; 1. University of Brescia, Italy

As a wide-bandgap semiconductor, ZnO is a very attractive candidate for blue and UV optoelectronics. We investigated the Micro Raman and Micro Photoluminescence (PL) as a function of the gaseous environment of individual ZnO nanowires (NWs), prepared by evaporation condensation technique, dispersed in isopropanol and transferred on different substrates (as SiO2, SiO2/Si p-doped, p-GaN, or p-SiC). The aim was to get a picture how the physico chemical properties of the NWs may affect the functional ones. Two innovative applications have been studied: Optical whispering gallery mode (WGM) resonances can be excited in zinc oxide micro- and nanowire cavities. Based on the resonant process, the performance of the WGM microcavity may become very sensitive to the surface refractive index. By using localized surface plasmons to generate hot carriers in noble metal nanostructures supported on metal oxide nanowires, visible light can produce - in addition to plasmonic heating - energetic hot electrons (or holes) which may drive chemical reactions on the supporting semiconductor. Here we will discuss how hot electrons could boost metal oxide nanowires based chemical gas sensing.

11:00 AM

(ICACC-S7-007-2017) Dry printing of carbon nanotubes for transparent, flexible and stretchable touch sensors (Invited)

E. Kauppinen*[1]; 1. Aalto University School of Science, Finland

We present the direct dry printing (DPP) manufacturing method of single walled carbon nanotube (SWNT) based transparent conducting films (TCF) for touch sensors with electrical properties on par with those of ITO-on-PET, and with optical as well as mechanical properties i.e. bendability, flexibility and especially stretchability better than those of ITO, metal nanowire and metal mesh. In addition, all carbon transparent, flexible and stretchable TFT-FETs have been made using percolating SWNT network as the semiconductor and SWNT TCF as the source, drain, gate and interconnect material. We show that SWNT networks consisting of long, clean and highly individualized SWNTs exhibit substantially improved TCF performance.

11:30 AM

(ICACC-S7-008-2017) Integrated metal oxide nanowires for chemical sensors

T. Fischer*[1]; Y. Gönüllü[1]; S. Mathur[1]; 1. University of Cologne, Germany

Metal oxide semiconductors, such as SnO2, ZnO, TiO2, etc. have been employed and studied in resistive gas sensing for several years. Nonetheless the so called "S-criteria" Selectivity, Sensitivity, Stability, Speed are still an extensive field of research to understand fundamental surface reactions as well as improving device performance. Especially the cross sensitivity with interfering analytes like humidity poses significant limitation to the applicability of metal oxide resistive gas sensors. The presentation will focus on potential pathways of integrating metal oxide nanowires directly on gas sensing substrates. In addition the surface modification of the resulting nanostructured materials for enhanced selectivity and sensitivity is discussed. Moreover different concepts for preconcentration and filtering are being evaluated supported by in-situ diagnostics.

11:50 AM

(ICACC-S7-009-2017) Hydrogen sensing characteristics of highly sensitive and reliable GaN based gas sensor (Invited)

S. Jung[1]; M. Lee[1]; K. Baik[2]; S. Jang*[1]; 1. Dankook University, Republic of Korea; 2. Hongik University, Republic of Korea

Recently, hydrogen has attracted great attention as an alternative energy source and a viable energy carrier. Different from conventional fossil fuels, it is an emission-free fuel that can produce heat combustion of 141.9 kJ/g when reacted with oxygen. Hydrogen gas is colorless, odorless, extremely reactive with oxygen, and has low ignition energy. Unfortunately, when it leaks from a pressurized container, its temperature rises quickly due to its negative Joule-Thomson coefficient. This in turn can induce spontaneous flammable ignition, posing great risk to humans and commercial equipment. Therefore, hydrogen gas detection is a critical issue for hydrogen-related industrial processes and product use. In general, market-driven sensors require a 0.1-10% detection range, less than 5 s response time, -30-80°C operational temperature, and an accuracy reading within 5% of full scale. GaN-based material system is very suitable for constructing hydrogen sensors to satisfy these requirements. In this study, hydrogen sensing characteristics of highly sensitive GaN based hydrogen sensors with various crystal planes was investigated. Also, as a dramatic approach to improve the sensitivity of the device, active platinum nanostructure and photo-chemically etched GaN were employed. In addition, nano-porous membrane was developed for the reliable operation of hydrogen sensor in humid ambient.

12:10 PM

(ICACC-S7-010-2017) Probing the complex surfaces of oxide materials (Invited)

G. Tabacchi*[1]; 1. Università Insubria, Italy

Establishing structure–functionality relationships for titanium dioxide – one of the key materials in technology – would be particularly desirable for improving a plethora of applications. Nowadays, this task can be pursued by analyzing extended surfaces of TiO_2-single crystals with probe microscopies such as, e.g., scanning tunnelling microscopy. Herein, we will show that TiO_2 nanoparticles with well-defined shape can provide the missing link between TiO_2 single crystals and commercial TiO_2 nanopowders with complex morphology. To this aim, we produced TiO_2 anatase nanoparticles with diverse shape and size - for example, truncated bypiramids or elongated rectangular prisms each characterized by Ti sites with different structure, coordination, and physico-chemical properties. These samples of controlled morphology were then used as reference materials for extensive IR-spectroscopy /DFT-modeling investigations on the surfaces of titania-based nanomaterials. IR spectroscopy of adsorbed carbon monoxide (CO), in view of its versatility and high sensitivity, was our choice approach for probing structure and reactivity of the surface sites. Our results will show that theoretical modeling may help tracing a route in this complex scenario by providing microscopic level insight on both structure and properties of the surface sites.

S9: Porous Ceramics: Novel Developments and Applications

Innovations in Processing Methods & Properties of Porous Ceramics I

Room: Coquina Salon G

Session Chair: Sawao Honda, Nagoya Institute of Technology

8:30 AM

(ICACC-S9-010-2017) Highly porous Y-Si-O silicates as high-temperature thermal insulator: Optimal processing and properties (Invited)

J. Wang*[1]; 1. Shenyang National Laboratory for Materials Science, Institute of Metal Research, China

Porous yttrium silicates, including $Y_2Si_2O_7$ and Y_2SiO_5 are promising high temperature thermal insulators in harsh environments due to their superior high temperature environmental corrosion resistance, mechanical durability, as well as the very low thermal conductivity. One critical challenge was to process highly porous Y-Si-O silicates with the balanced high porosity, low thermal conductivity and high strength. The reason was attributed to the trade-off between high sintering activity of Y-Si-O powders (or significant shrinkage of porous Y-Si-O silicate green bodies) and the formation of strong sintering necks at temperatures above 1400°C. We developed in-situ reaction sintering and foam-gelcasting method by using Y_2O_3 and SiO_2 powders as starting materials and non-toxic gelatin as gelling agent to fabricate porous Y-Si-O silicates at 1550°C. The as-prepared sample has unique multiple pore structures, low linear shrinkages, controllable high porosities, relatively high compressive strengths, and low thermal conductivities. The present work ensures the potential high-temperature thermal insulation applications of highly porous Y-Si-O silicates with optimal light weight, low thermal conductivity, and strength.

9:00 AM

(ICACC-S9-011-2017) Experimental investigation and numerical modeling of mechanical behavior in porous carbon materials

E. Kojo*[1]; R. Inoue[1]; Y. Kogo[1]; 1. Tokyo University of Science, Japan

Porous carbon materials are potentially used for light-weight applications such as advanced space craft structures. Experimental investigation has been carried out and mechanical properties of them strongly depend on pore size. In order to investigate pore size dependence, several simple models have been proposed, however, modeling of real structure is difficult because they have quite complicated microstructure. In this study, mechanical properties of porous carbon materials with equal porosity for all pore sizes, namely, 4.5, 4.9, and 9.8 μm were evaluated experimentally. Then, complicated microstructure was modeled in three-dimensions by image based modeling using X-ray CT and FIB-SEM. The elastic properties of them were evaluated by homogenization method. Experimental results showed mechanical properties of porous carbon materials strongly depend on its pore size. In this presentation, the effect of pore size on mechanical properties of a porous carbon material will be discussed based on the experimental results and modeled structures.

9:20 AM

(ICACC-S9-012-2017) Main parameters governing the radiative properties of silicon carbide open-cell foams up to T = 1300 K

B. Rousseau*[1]; 1. CNRS LTN, France

Silicon carbide (SiC) open-cell foams are remarkable porous materials for developing high-temperature energy conversion systems (T~ 1300 K) in which heat transfer and fluid flow must be simultaneously intensified. The thermal contribution due to the thermal radiation is crucial at these expected temperatures so that the knowledge of their thermal radiative properties (TRP) is mandatory. However, the direct measurement of TRP up to 1300 K remains today difficult so that modelling approaches are the only possibility to investigate them. A key challenge to accurately model TRP is to be able to separate what is due to the struts, themselves often constituted of micro scale size grains, of what is related to the 3D porous network. To answer to this question, a foam generator was developed to elaborate a set of realistic 96 open-cell foams on large ranges of porosity (0.4-0.95) and pore nominal diameter (0.4-2.6 mm). The solid part of each foam are endowed with complex indices of refraction, previously extracted up to T= 1300 K on SiC single crystals with known chemical doping level. A Monte Carlo Ray Tracing code is later applied to derive practical laws connecting TRP to both the chemical and textural features of the set of SiC foams. The importance of each contribution on TRP is finally discussed. It opens new developments for studying TRP for temperature higher than 1300 K.

9:40 AM

(ICACC-S9-013-2017) Thermomechanical characterization of porous LSCF with different pore structures and porosity

Y. Zou*[1]; F. Schulze-Küppers[1]; M. Balaguer[1]; J. Malzbender[1];
1. Forschungszentrum Juelich, Germany

Advanced mixed electron ion conducting ceramic membranes for gas separation based on a thin layer concept supported by porous substrates. Substrate porosity as well as pore structure are, therefore, critical parameters for the long-term reliability under the demands of elevated temperature exposure, under strains induced thermomechanically and by stoichiometric changes. Mechanical properties of porous $La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_{3-\delta}$ (LSCF), envisaged as substrate for dense membrane layers, were studied with respect to apparent elastic and creep behavior. Porous LSCF with different microstructures was manufactured by tape-casting, freeze-casting and uniaxial pressing, resulting in different pore microstructure and porosities. Apparent elastic deformation was characterized by indentation tests at room temperature and by bending tests at operation relevant temperatures. Elevated temperature creep behavior of the LSCF samples was investigated via compression tests. Pore structures and porosity were studied using optical and electron microscopy.

Mechanical Properties of Porous Ceramics

Room: Coquina Salon G
Session Chair: Jingyang Wang, Shenyang National Laboratory for Materials Science, Institute of Metal Research

10:20 AM

(ICACC-S9-014-2017) Bending Strength Test of Porous Ceramics using Small Size Specimen

S. Honda*[1]; K. Yasuda[2]; T. Ono[3]; H. Kita[4]; M. Takahashi[5]; Y. Takahashi[6]; S. Tanaka[7]; S. Taruta[8]; T. Mitsuoka[9]; H. Muto[10]; S. Yamamoto[11]; Y. Yoshizawa[12]; 1. Nagoya Institute of Technology, Japan; 2. Tokyo Institute of Technology, Japan; 3. Kyocera, Japan; 4. Nagoya University, Japan; 5. Ehime University, Japan; 6. Noritake Company 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. Asuzac, Japan; 12. AIST, Japan

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. We have been evaluated the bending test data obtained from the round robin tests in Japan, and discussed how to measure the strength distribution of porous ceramics. In recently study, we reported the results of bending strength test of porous ceramic using the large specimen size regulated in the Japanese standard testing method, JISR1664. In this study, we investigated the smaller specimen size regulated in more widely used testing method for dense ceramics, JISR1601. Samples are porous Al_2O_3, and NiO/8mol%YSZ electrode material. The number of specimens is 30 for both materials in each organization. The average bending strength and fracture location among the organization were almost same. There no difference between average fracture strength of three and four point bending test. Both samples showed elastic deformation and followed by brittle fracture. Bending strength data can be expressed by 2-parameter Weibull distribution. The values of bending strength and Weibull shape parameter of small specimen size were higher than these of lager specimen size. This work was supported in part by METI, Japan.

10:40 AM

(ICACC-S9-015-2017) Linearity Condition on Weibull Plot of Bending Strength Data of Porous Ceramics

K. Yasuda*[1]; 1. Tokyo Institute of Technology, Japan

Bending strength is one of the most important characteristics in dense fine ceramics; however, that of porous ceramics also becomes an attractive topic in recent days. For example, porous ceramic electrodes in SOFC are functionally damaged by its fracture due to thermal cycling, and bioceramics are sometimes broken in bending in clinical cases. So, it is important to attain strength reliability in porous ceramics in advanced systems as key devices and components. In this presentation, the author discusses the applicability and validity of Weibull plot to bending strength data of porous ceramics. The numerical strength datasets are generated, and plotted in Weibull plot. For each plot, the square of correlation factor is estimated, and its average and standard deviation are statistically analyzed. From this investigation, the author proposes the linearity condition on Weibull plot, and also makes clear how many specimens should be used for bending test in porous ceramics. This work was supported in part by METI, Japan.

11:00 AM

(ICACC-S9-016-2017) Effects of platelets addition on the microstructure and uniaxial compressive response of ice-templated porous alumina

D. Ghosh*[1]; M. Banda[1]; H. Kang[1]; V. Kamaha[1]; 1. Old Dominion University, USA

Bioinspired hierarchical porous ceramics are a new class of materials that have drawn significant attention for structural applications to energy storage. Processing of such hierarchical ceramics is of significant challenge, however, freeze casting has emerged as one potential technique that relies on the principles of unidirectional solidification of particulate suspensions. In spite of the significant developments on the process-microstructure correlations, very little progress is made on the structure-property relationships and understanding the role of different length-scale components on the mechanical properties. In this presentation, we will show that grain-level anisotropy is an attractive approach to significantly improve the uniaxial compressive response of the ice-templated ceramics, however, without modifying the overall material composition and porosity. To this end, a custom-made device is employed to develop a series of ice-templated cellular ceramics where the particle morphology and freezing kinetics are systematically varied to modify the lamella thickness, interlamellae spacing, interlamellae bridge density and grain-level morphology to understand their influence on the stiffness, compressive strength and energy absorption characteristics.

Preliminary results on the high-strain rate compressive response will also be presented.

11:20 AM

(ICACC-S9-017-2017) Adaptation of the Chevron-Notch Beam Fracture Toughness Method to Diesel Particulate Filters

A. Wereszczak*[1]; O. Jadaan[2]; M. Modugno[1]; G. Hatala[2]; M. Lance[1]; 1. Oak Ridge National Lab, USA; 2. University of Mount Union, USA

The apparent fracture toughness of diesel particulate filter ceramics was estimated using large chevron-notch test specimens inspired by ASTM C1421. The specimens had a nominal cross-section of 20 x 40 mm, an a_0 of ~ 8 mm, and were loaded in 3-point loading with a 140-mm span. Stable crack propagation and classical load-displacement curves for this specimen configuration were consistently achieved. Companion finite element analysis was used to model the specimen compliance change as a function of advancing crack propagation, and an apparent fracture toughness of 0.7 MPa√m was ultimately determined. The specimen geometry, test method, and allied analysis are advocated because of the ease of specimen harvesting and confidence in achieving classical load-displacement response, and are described here. This manuscript has been authored by UT-Battelle, LLC, under Contract No. DE-AC05-00OR22725 with the U.S. Department of Energy.

11:40 AM

(ICACC-S9-018-2017) Comparative Analysis of Synthesis and Mechanical Characterization of Porous Ceramics

M. Mehr*[1]; J. Pineiro-Llanes[1]; J. C. Nino[1]; 1. University of Florida, USA

Highly porous ceramics such as titania and barium titanate are of much interest for a variety of applications such as sensors, catalytic structures, bio applications, and many others. Alternative synthesis and processing methods as well as the mechanical properties of these materials are of much interest. The focus of the work presented here is on the synthesis and mechanical properties. Methods such as direct polyurethane foaming and high internal phase emulsion method will be presented. The corresponding microstructures will be illustrated. Additionally, an alternative sintering process of two-step sintering and its applicability to highly porous ceramics will be discussed. Furthermore, a new testing method for compressive mechanical properties of highly porous (>80%) and brittle materials is proposed. It will be demonstrated that the difficulty in measuring compressive mechanical properties such elastic modulus or collapse stress is the result of imperfect sample platen contact. The use of an epoxy interphase will be shown to improve the compressive testing of these types of materials and produce reliable data.

S10: Virtual Materials (Computational) Design and Ceramic Genome

Modelling - Mechanical behavior

Room: Ponce DeLeon

Session Chairs: Wai-Yim Ching, University of Missouri-Kansas City; Masato Yoshiya, Osaka University

8:30 AM

(ICACC-S10-009-2017) Stochastic Dependence of Microstructure-sensitive Damage Formation/Propagation in Ceramic Continuous Fiber Reinforced Ceramic Matrix Composites (Invited)

A. Samuel[2]; S. Hawkins[3]; C. Blake[4]; S. Bricker[5]; J. Simmons[1]; T. Whitlow[5]; J. Pierce[5]; M. Braginsky[5]; C. P. Przybyla*[1]; 1. Air Force Research Laboratory, USA; 2. University of California, Santa Barbara, USA; 3. Wright State Unversity, USA; 4. University of Texas A&M, USA; 5. University of Dayton Research Institute, USA

Much remains to be understood regarding the fundamental physics of crack formation and propagation in extreme environments of ceramic continuous fiber reinforced ceramic matrix composites (CMCs). Specifically, the relationship between the stochastic material structure at the pertinent scales relative to the operant damage modes is considered here. The distribution of the continuous SiC fibers coated with a BN interphase was studied relative to crack initiation and propagation at the microscale. Detailed microstructure characterization was performed to quantify the individual fibers and the fiber coatings, spatially. Experiments were performed with digital image correlation and acoustic emission to monitor initiating and/or propagating cracks at room and elevated temperatures; correlation between the stochastic microstructure and observed damage were explored. Discrete damage models of damage formation and propagation were developed and compared to experiments.

9:00 AM

(ICACC-S10-010-2017) Extended Finite Element Analysis of the Fracture Behavior of ZrB_2-Carbon Ultra-High Temperature Ceramics

L. Jarvis*[1]; M. Asle Zaeem[1]; W. Fahrenholtz[1]; G. Hilmas[1]; J. Watts[1];
1. Missouri University of Science & Technology, USA

Zirconium diboride (ZrB_2) is an ultra-high temperature ceramic (UHTC) being researched and developed for applications in extreme environments. ZrB_2 has high strength (> 400 MPa) and thermal conductivity (> 100 W/m●K) but low fracture toughness (< 3 MPa●$m^{1/2}$), which limits its use. Processing of ceramics to produce composites with engineered architectures has been shown to increase the fracture toughness via crack deflection and frictional sliding mechanisms. The goal of this research is to predict the fracture behavior and crack propagation in composite ceramics produced with a main phase of ZrB_2 and a secondary phase of Carbon (C). Material properties measured for ZrB_2 and C were combined with the extended finite element method (XFEM), as implemented by ABAQUS 6.14, to predict the fracture behavior for ZrB_2-C composites. Three different composite architectures were simulated under an applied tension: monolithic, laminated, and fibrous monolithic. The monolithic and laminated structures were also simulated in four-point bending. For the laminate ceramics, an interface layer was introduced to study the effects of the interlayer bond strength on crack propagation. The results of the simulations were compared to experimental four-point bending tests to predict architectures that will exhibit crack deflection and graceful failure behavior.

9:20 AM

(ICACC-S10-011-2017) Numerical and Experimental Analysis of a 3D Carbon/Carbon Composite under Four-point Bending

A. Este*[1]; B. Toson[1]; J. Saliba[2]; J. El Yagoubi[3]; S. Morel[2]; E. Martin[4];
1. CEA, France; 2. University Bordeaux, France; 3. University Bordeaux, France; 4. LCTS - CNRS, France

Carbon/Carbon (C/C) composites are used in several industries for their superior thermo-mechanical properties at very high temperatures. In this study, the mechanical behavior of a 3D Carbon/Carbon composite is considered. An experimental investigation in the fracture properties of C/C subjected to four-point bending test has been performed. Crack growth has been monitored using simultaneously several measurement techniques as the digital image correlation (DIC), acoustic emission (AE) and in situ microscopy. Those later allow a continuous and a real time data acquisition and thus can give wealth information on the fracture process and damage mechanisms. The behavior of C/C is modeled at the mesoscopic scale with a damage model associated to a meshing method. The damage model used has been developed and implemented in the finite element modeling software Cast3M. The meshing method consists in projecting materials properties on the shape functions of a finite element mesh, i.e. at each Gauss point, the properties of the meso-constituents (fiber yarns and matrix) are assigned based on the integration point position. The results show a good correlation between load-crack mouth opening displacement (CMOD) curves and AE and DIC analysis. In addition, the numerical simulations show that the mesoscopic modeling reproduces well the failure of C/C composite.

9:40 AM

(ICACC-S10-012-2017) Multi-Phase-Field Modeling of Crack Propagation in Polycrystalline ZrB_2-Based Ceramics

A. Emdadi*[1]; M. Asle Zaeem[1]; W. Fahrenholtz[1]; G. Hilmas[1]; 1. Missouri University of Science & Technology, USA

Initiation and propagation of cracks in heterogeneous brittle materials are governed by their polycrystalline microstructures. Conventional computational models for simulating crack propagation treat cracks as sharp interfaces, which can result in numerical singularities for complex crack topologies where several cracks may agglomerate. Conventional models usually require a fine numerical mesh and an adaptive meshing algorithm during crack growth simulations making them computationally expensive. In this work, a multi-phase-field (MPF) model based on the regularized formulation of the energy functional in Griffith theory was developed to study two-dimensional crack initiation, propagation and branching in polycrystalline zirconium diboride ceramics. To evaluate the basic characteristics of the model, crack propagation was first simulated for a ZrB_2 single crystal. These results were compared with classical fracture mechanics predictions. Then, intergranular and transgranular crack propagation were simulated by considering the effects of grain boundary energy anisotropy and crack surface energy in polycrystalline ZrB_2 and ZrB_2-C multiphase materials. The results showed that the MFP model can reasonably predict crack propagation path in multiphase polycrystalline ceramics.

10:20 AM

(ICACC-S10-013-2017) Tensile and compression experiment on an amorphous zeolitic imidazolate framework (ZIF) model (Invited)

W. Ching*[1]; 1. University of Missouri-Kansas City, USA

Recently, we have constructed a large periodic amorphous zeolitic imidazolate framework (a-ZIF) model of 918 atoms and investigated it electronic and optical properties. Based on this model, we have performed the ab initio computational experiments to investigate the deformation behaviors under compression and expansion. The results are presented in the form of stress vs strain data for strains up to 0.25 for compression and 0.30 for expansion. For compression, it shows an initial linear behavior at small pressure giving a bulk modulus value of about 2.89 GPa but a non-linear behavior at high pressure with a transition region close to 2. GPa to 5. GPa. The tensile deformation under expansion is quite different. It shows a complete nonlinear variation of stress with strain with a maximum stress of about 1.5 GPa around 15% of expansion. These and other results under compression and expansion will be presented and discussed.

10:50 AM

(ICACC-S10-014-2017) Theoretical Investigations on Atomic Structures and Bonding during Plastic Deformation in Ionic Crystals (Invited)

K. Matsunaga*[1]; 1. Nagoya University, Japan

Structural ceramic materials, such as metal oxides are generally brittle at low temperatures, because of the ionic nature of bonding. In order to improve their mechanical toughness and fracture resistance, it is essential to obtain a fundamental understanding of their deformation processes from the electronic and atomic levels. In this study, some of interesting mechanical behaviors in ionically bonded crystals were focused, and their physical origins were investigated by theoretical calculations. As one of typical examples, slip deformation behaviors in rock-salt structured sodium and silver chloride crystals were analyzed. Although these two systems are similar with each other, 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. It was found from first-principles calculations that calculated potential barriers for slip deformation show the limited activation of slip system in sodium chloride and yet the abovementioned slip systems can be activated in silver chloride. From electronic densities of states, it was also found that slip deformation in silver chloride accompanies unexpected formation of covalent like Ag-Ag interactions across the slip planes, which makes multiple slip systems activated even at low temperatures.

11:20 AM

(ICACC-S10-015-2017) Strain effects on energetic responses of oxygen vacancy in $KTaO_3$

J. Xi*[1]; Y. Zhang[2]; W. J. Weber[1]; 1. University of Tennessee, USA;
2. Oak Ridge National Laboratory, USA

Due to lattice mismatch between epitaxial films and substrate, in-plane strain fields are produced in the thin films, with accompanying the structural distortions. Because of the strain profile, defect behavior is changed, which can affect the properties of the thin films. In this work, the response of the oxygen vacancy (V_O) to a biaxial strain field in $KTaO_3$ thin films are investigated by first-principles calculations. In particular, the doubly positive charged oxygen vacancy (V_O^{2+}) is considered, which is proposed to be the main charge state under equilibrium $KTaO_3$. We find that the formation energies for the two symmetrically inequivalent oxygen vacancies are sensitive to the strain field and oxygen position in both compressive and tensile strain fields. The possible mechanisms for these responses are identified, and it is found that the variations of the V_O donor level from deep to shallow during the relaxation are predominant and mainly responsible for the changes in formation energies. In addition, all the possible migration paths are considered, and it is observed that the V_O migration barrier is also affected by the strain fields, resulting in anisotropic migration. Based on above results, oxygen vacancy ordering is expected to occur due to the site sensitivity and diffusion anisotropy of oxygen vacancies in this material.

11:40 AM

(ICACC-S10-016-2017) Theoretical Investigations on the High Temperature Structure and Mechanical Properties of TMB_2 (TM = Ti, Zr, Hf) (Invited)

H. Xiang*[1]; Y. Zhou[1]; Z. Feng[1]; Z. Li[1]; 1. Aerospace Research Insitute of Materials and Processing Technology, China

As promising candidates for ultra high temperature applications, high temperature properties, which are quite rare and fragmentary, have great significance to TMB2 (TM = Ti, Zr, Hf). In this work, thermodynamic and mechanical properties of TMB2 from 0 K to 2000 K were investigated by a combination of first principles calculations and quasi-harmonic approximations. The ground-state properties, including lattice parameters, elastic constants, phonon dispersion and mode-Grüneisen parameters are calculated. The theoretical thermal expansion, elastic and thermodynamic properties at elevated temperatures show good agreement with experiments. By discussing Grüneisen parameters anisotropy, the mechanism for the thermal expansion anisotropy of TMB2 is uncovered. The influence of direction-dependent sound velocities on the anisotropy of thermal conductivity is also discussed.

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

Production Root Technology III

Room: Coquina Salon H
Session Chairs: Ali Erdemir, Argonne National Lab;
Kyoung Il Moon, KITECH

8:30 AM

(ICACC-S11-010-2017) Quantum Mechanically Guided Materials Design for Surface Engineering (Invited)

J. M. Schneider*[1]; 1. RWTH Aachen Univeristy, Germany

The combinatorial approach, combining combinatorial materials synthesis of thin film composition-spreads with high-throughput property characterization has proven to be a powerful tool to delineate composition-structure-property relationships, and hence to efficiently identify composition windows with enhanced properties. The combination of modern electronic structure calculations with the highly efficient combinatorial thin film composition-spread method constitutes an effective tool for knowledge based materials design of hard and wear resistant coatings. Besides the elastic property and phase stability also the interaction of the coating with the ambient can be described based on quantum mechanics. In the talk predictions of the interaction of coated tool surfaces with gases contained in the atmosphere as well as materials to be formed are discussed. Coatings used for forming operations of Al and Polymers are investigated and initial experimental data characterizing these interactions will be discussed.

9:00 AM

(ICACC-S11-011-2017) Friction and Wear Behavior of MXenes under Dry Sliding Conditions (Invited)

G. Ramirez*[1]; S. Kota[2]; O. L. Eryilmaz[1]; Y. Gogotsi[2]; M. W. Barsoum[2]; A. Erdemir[1]; 1. Argonne National Laboratory, USA; 2. Drexel University, USA

MXenes are a new class of 2D materials based on transition metal carbides and/or nitrides. They hold great promise for a variety of industrial applications including energy storage, sensors, electronics, and composites. Lamellar nature of MXenes (similar to that of other 2D materials, like graphene, MoS_2, etc.) also makes them good prospects for mitigating friction and wear in tribological applications. Accordingly, using a pin-on-disk machine, we explored the sliding friction and wear behaviors of Ti_3C_2 in dry nitrogen and open air as a nano-particulate lubricant and as a cold-pressed flat sample. The results from the powder lubrication showed nearly 3 orders of magnitude reduction in wear and 60% reduction in friction compared to the baseline steel in nitrogen. Compared to graphite powders, the reduction in wear was more than two orders of magnitude, while graphite's friction coefficient was low initially (i.e., for about 15 min) but increased to 1. Test results from the cold pressed MXene samples were also very impressive and confirmed more than 50% reduction in friction, while wear was reduced by as much as 2 orders of magnitude compared to baseline steel. Based on the results of microscopic studies, we will explain the mechanisms that were responsible for the much favorable friction and wear behavior of MXene samples.

9:30 AM

(ICACC-S11-012-2017) Flexible ceramic coatings: Role of energy (Invited)

J. Musil*[1]; G. Remnev[2]; 1. University of West Bohemia, Czech Republic; 2. National Research Tomsk Polytechnic University, Russian Federation

The lecture is divided in three parts. The first part reports on the enhanced hardness, the thermal stability of nanocomposite coatings and the formation of the X-ray amorphous coatings with thermal stability and oxidation resistance above 1000°C using sputtering. The second part is devoted to the flexible hard nanocomposite coatings with enhanced toughness and enhanced resistance to cracking. The principle of the formation of flexible hard coatings with enhanced resistance to cracking is explained. It is shown that a key role in formation of the flexible coatings plays the energy delivered during their growth. The flexible hard coatings with enhanced toughness represent a new class of advanced protective and functional coatings with a huge application potential. As examples, the robust, flexible antibacterial coatings with long lifetime and the protective over-layers preventing to cracking of hard brittle coatings are given. Reported results can be used in the development of the flexible ceramic coatings, the surface strengthening of brittle materials, the prevention of (i) cracking of the functional coatings and (ii) the cracks formation on the surface of bended materials. The third part explains the principle of the formation of nanocrystalline and crystalline films on unheated substrates. At the end, trends of next development of the advanced nanocomposite coatings with unique properties are outlined.

10:20 AM

(ICACC-S11-020-2017) Smart powder processing of advanced materials and their recycling (Invited)

M. Naito*[1]; A. Kondo[1]; T. Kozawa[1]; 1. Osaka University, Japan

Recently, various novel powder processing techniques were rapidly developed for advanced material production due to the growth of the high-tech industry. Smart powder processing is a green and sustainable powder processing technique that creates advanced materials with minimal energy consumption and environmental impact. Particle bonding 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. By making use of particle bonding, a new "one-pot" processing method to synthesize nanoparticles without applying extra heat was also developed. Furthermore, one-pot processing achieving both the synthesis of nanoparticles and their bonding with other kinds of particles to make nanocomposite granules was also developed. On the other hand, by carefully controlling the bonding between different kinds of materials, separation of composite structure into elemental components is also possible, which leads to the development of novel technology for recycling composite materials and turns all of them to highly functional applications. In this presentation, the development of novel recycling methods of glass fiber reinforce plastics will be introduced. Besides, its applications for recovering useful elements such as precious metals from waste composites without any heat support will be also explained.

10:50 AM

(ICACC-S11-014-2017) Two-step Loading in Pulsed Electric Current Sintering of MoO_3 for Production of Radioactive Isotopes

H. Suematsu*[1]; S. Sato[1]; M. Seki[1]; M. Nanko[2]; K. Tsuchiya[3]; K. Nishikata[3]; T. Suzuki[4]; T. Nakayama[1]; K. Niihara[4]; 1. Nagaoka University of Technology, Japan; 2. Nagaoka University of Technology, Japan; 3. Japan Atomic Energy Agency, Japan; 4. Nagaoka University of Technology, Japan

For production of ^{99m}Tc, a radioactive isotope used in medical applications, from ^{98}Mo by a nuclear reaction with neutrons in a nuclear reactor, one of the difficulties is to obtain high density MoO_3 pellets which evaporates above 700°C. To overcome this problem, a two-step loading method in pulsed electric current sintering was carried out in this study. Powder of MoO_3 with an average grain size of 12.5 μm was pressed in a graphite die with a diameter of 20mm. Then, the green compacts were initially loaded at 10 MPa and heated in a spark plasma sintering apparatus with heating rates of 100°C/min to 550°C in vacuum. Then, the load was increased to 40 MPa. After holding the temperature for 5 min, the samples were quenched. The sintered samples were characterized by powder X-ray diffraction for phase identifications and scanning electron microscopy for grain size measurements. Relative densities of the sintered MoO_3 bulks are shown in Fig.1. After sintering at 550°C, the sintered MoO_3 bulk had a relative density of 93 %, which was higher than that sintered by a single-step loading at 40 MPa and 550°C(78%). This property is good enough for separation of ^{99m}Tc and recycle of Mo.

11:10 AM

(ICACC-S11-015-2017) Study of Shielding Method to Reduce Leakage Magnetic Field of an Opening in a Magnetically Shielded Room

H. Sugiyama*[1]; 1. National institute of Technology, Kagoshima College, Japan

Technologies that use magnetism and generate a magnetic field have been used in a wide range of fields such as medical care and industry. However, precision instruments are often affected by a magnetic field. Therefore, these precision instruments must use a magnetically shielded room (MSR) to reduce the magnetic noise. The MSR consists of multiple shielding layers, and some openings are installed in a wall and the floor to secure the ventilation, wiring, and plumbing. However, magnetic noise can leak into the MSR from those openings, and anything inside can be influenced by magnetic noise. The shape of the most suitable partition plates to be installed inside the duct was examined by a three-dimensional magnetic field analysis to prevent magnetic fields from entering through the openings. We found that the partition plate should be approximately 0.5 times the opening width of the cell to decrease the amplitude of the magnetic field entering through the openings. When the length of the partition plate is too long, a large amount of magnetic fields from the outside is collected. In contrast, if the length of the partition plate is short, it cannot collect enough magnetic fields from the outside.

11:30 AM

(ICACC-S11-016-2017) Effect of calcium salts on reaction of dicalcium phosphate dihyrdare (DCPD) with fluoride ion

S. Muroyama*[2]; M. Tafu[2]; S. Takamatsu[2]; T. Toshima[2]; Y. Matsushita[1]; T. Fujita[1]; 1. Chiyoda Ute Co. Ltd., Japan; 2. National Institute of Technology, Toyama College, Japan

Dicalcium phospohate dihydrate (DCPD) reacts with fluoride ion and forms stable fluorapatite (FAp). We have applied this reaction to immobilize small amounts of fluoride ion in the environments. However, reaction of DCPD forms phosphate ion that is serious problem for application of eco-solutions. We investigated mechanism of phosphate release from the reaction of DCPD, and method controlling release of phosphate by addition of calcium salts. From results of the investigations, calcium carbonate was effective to control release of phosphate by suppling calcium ion and controlling pH. We also studied effect of mixture of calcium salts on release of phosphate ion from the DCPD reaction and appeared that mixture of calcium sulfates and calcium carbonate was more effective for inhibit of release of phosphate ion.

11:50 AM

(ICACC-S11-017-2017) Nanocomposite Mo-Cu-N and Mo-Cu-X N (X=Cr, Ni, W, B) coatings deposited by reactive magnetron sputtering process with single alloying target

H. Park*[1]; C. Byun[1]; K. Moon[1]; 1. KITECH, Republic of Korea

In this study, it has been tried to make the single Mo-Cu alloying targets with the Cu showing the best surface hardness that was determined by investigation on the coatings with the double target process. The single alloying targets were prepared by powder metallurgy methods such as mechanical alloying and spark plasma sintering. The nanocomposite coatings were prepared by reactive magnetron sputtering process with the single alloying targets in $Ar+N_2$ atmosphere. Also the effects of third alloy elements such as Cr, Ni, W on the Mo-Cu coatings have been surveyed by using single alloying targets. The microstructure changes of the Mo-Cu-N and Mo-Cu-X-N (X=Cr, Ni, B) coatings were investigated by using XRD, SEM and EDS and TEM. The mechanical properties of the coatings were evaluated by using nanoindentor, scratch test, and ball on disc methods. In this study, the nano-composite MoN-Cu coatings prepared using an alloying target was eventually compared with the coatings from the multiple targets.

S12: Materials for Extreme Environments: Ultrahigh Temperature Ceramics (UHTCs) and Nano-laminated Ternary Carbides and Nitrides (MAX Phases)

Materials Design, New Composition and Composites III

Room: Tomoka B

Session Chairs: Surojit Gupta, University of North Dakota; Guorui Zhao, Institute of Metal Research

8:30 AM

(ICACC-S12-010-2017) 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.

9:00 AM

(ICACC-S12-011-2017) ZrB_2-$MoSi_2$ Dual-Scale Composite Architectures: Overcoming the Strength-to-Toughness Trade-off for High Temperature Structural Applications

F. Monteverde*[1]; S. Failla[1]; C. Melandri[1]; D. Sciti[1]; R. J. Grohsmeyer[2]; G. Hilmas[2]; W. Fahrenholtz[2]; 1. CNR-ISTEC, Italy; 2. Missouri University of Science and Technology, USA

Dual composite (DC) architectures have been shown to increase wear resistance while maintaining or increasing fracture toughness in WC-Co composites for room-temperature applications by the use of multiple, discretely segregated engineered microstructures. Composites of zirconium diboride (ZrB_2) and molybdenum disilicide ($MoSi_2$) are candidates for similar dual composite architectures for high-temperature structural use in extreme environments, due to the high-temperature ductility and oxidation resistance of $MoSi_2$. Multiple DC architectures were prepared by hot-pressing pre-sintered granules of known volumetric amount, size and composition that are dispersed in a continuous matrix made of a different loose powder mixture. The powder technology selected to produce granules was the freeze-spray granulation. Conventional composites, using compositions representative of both the granules and the matrices, were also hot pressed for comparison. The joint research effort investigated microstructure development and mechanical properties including elastic modulus, thermal expansion, flexure strength and fracture toughness at room temperature and at 1500°C in air. The results were compared to corresponding traditional particulate sub-composites.

9:20 AM

(ICACC-S12-012-2017) Evidence for the possibility to tune the substitution rate in $[Ti_{(1-y)}Cu_y]_3[Al_xCu_{(1-x)}]C_2$ MAX phase solid solution

S. Dubois*[1]; M. Nechiche[2]; T. Cabioch[1]; V. Brunet[1]; P. Chartier[1]; V. Mauchamp[1]; S. Azem[2]; A. Joulain[1]; 1. PPRIME Institute, France; 2. Université Mouloud Mamerri, Algeria

In this work, Ti_3AlC_2 MAX phase powders were first prepared at 1450°C for 2 hours from 1.9TiC:1.05Al:1.0Ti initial reactant powder mixture. High-energy milling of Ti_3AlC_2 and Cu powders was thus performed during 20 minutes with a ball to powder mass ratio of 5 in order to form large aggregates containing alternative lamellas of Ti_3AlC_2 and Cu. Thus, a $[Ti_{(1-y)}Cu_y]_3[Al_xCu_{(1-x)}]C_2$-Cu Metal Matrix Composite has been produced by sintering the mixture of co-milled Ti_3AlC_2 and Cu powders. In a next step, a selective chemical attack has been performed to extract the metallic Cu. The aim of this work consists in reaching a better understanding of the chemical reaction to synthesize MAX phase solid solutions containing Cu. For that purpose, solid solutions were synthesized from reactant mixtures with Cu volume fractions varying from 10 to 40% and Ti_3AlC_2 volume fraction varying from 90 to 60%. It turns out that aluminium atoms are substituted preferentially compared to titanium atoms. Indeed, copper or nickel atoms can substitute about 50% of the aluminium atoms whereas only a few percent of the titanium atoms can be substituted. Moreover, it is demonstrated that the substitution rate can be tuned by varying the Cu volume fraction; it can reach 50% on the Al site of the Ti_3AlC_2 phase.

9:40 AM

(ICACC-S12-013-2017) Processing and Elevated Temperature Mechanical Properties of ZrB_2-Based Laminates

C. Wittmaier*[1]; W. Fahrenholtz[1]; G. Hilmas[1]; 1. Missouri University of Science & Technology, USA

Elevated temperature flexure strength and fracture toughness were investigated for ZrB_2-based laminates. Both ZrB_2 and mixtures of graphite plus ZrB_2 were blended with thermoplastic polymers. The ceramic laden polymers were then compressed in a heated uniaxial press to produce layers of consistent thickness which were stacked in an alternating pattern and laminated in the green state. The green laminates were pyrolyzed to remove the polymer, and then densified by hot pressing. The microstructures of the laminates were observed, and the flexure strengths and elastic moduli of the laminates were measured at room and elevated temperatures. The microstructures and mechanical properties of the laminates will be compared to traditional ZrB_2 ceramics. The primary focus of this research is to investigate whether this structure improves the fracture toughness significantly compared to conventional ceramics.

Methods for Improving Damage Tolerance, Oxidation and Thermal Shock Resistance

Room: Tomoka B
Session Chairs: Zhe Cheng, Florida International University; Frederic Monteverde, CNR-ISTEC

10:20 AM

(ICACC-S12-014-2017) Tailored Interfaces for Engineered High Temperature Propulsion Materials (Invited)

A. Ghoshal*[1]; M. Murugan[1]; M. J. Walock[1]; B. Barnett[1]; A. Nieto[1]; M. S. Pepi[1]; J. Swab[1]; D. Zhu[2]; W. Gamble[1]; 1. US Army Research Laboratory, USA; 2. NASA Glenn Research Center, USA

This research outlines the preliminary efforts to discover the fundamental relationships between heterophase interface properties and the thermo-mechanical behavior in dissimilar materials, and to create the ability to develop and fabricate materials with targeted macroscale properties via understanding and control of their interfacial properties. The baseline work is based on yttria stabilized zirconia (YSZ) and variations thereof in terms of layered, blended and graded thermal barrier coating deposited onto Inconel substrates with an interfacial bond coat. The modeling of non-discrete interface includes modeling plasticity, grain boundary complexions, and phase field changes due to complex aero-thermal and structural loads in heterophase interfaces. ARL spray deposition technologies of APS and SPPS are utilized to tailor dissimilar interfaces for densification of rigid particles during deposition to generate functionally graded material (FGM) systems. The developed functionally graded ceramic-metal and/or ceramic-ceramic composite materials are tested for thermal durability at the ARL Jet Burner Rig to temperatures up to 1650°C.

10:50 AM

(ICACC-S12-015-2017) Anti-oxidation and mechanical performance of a BN interface in an ultra-high temperature useful C_f/C composite

P. S. Makurunje*[1]; I. Sigalas[1]; 1. University of the Witwatersrand, South Africa

Boron nitride (BN) interfaces for C_f/C composites have mostly been prepared by the polyborazine route when employing liquid techniques. This study presents an ultra-high temperature useful C_f/C composite with a BN interface prepared by the liquid urea-boron infiltration and nitridation route. C_f/C composite specimens with and without the interface were exposed to an oxyacetylene flame around 3000°C and studied by X-Ray diffraction and Raman spectroscopy. The microstructures were examined by scanning electron microscopy and transmission electron microscopy. The mechanical strength contribution of the interface was investigated by the ball-on-three-balls test and the fractured composites examined by X-Ray Computer Tomography. The BN interface is capable of improving the anti-oxidation and structural integrity of C_f/C composites.

Structure Stability under Extreme Environments I

Room: Tomoka B
Session Chairs: Zhe Cheng, Florida International University; Frederic Monteverde, CNR-ISTEC

11:10 AM

(ICACC-S12-016-2017) Compression Creep of HfB_2 and HfB_2-SiC at 1500°C in Argon: Effects of Grain Size and SiC Content

A. DeGregoria[1]; M. Ruggles-Wrenn*[1]; 1. Air Force Institute of Technology, USA

Due to its high thermal conductivity and oxidation resistance at very high temperatures, HfB_2 is being considered for use as leading edge material on sharp-bodied reentry vehicles. In structural applications HfB_2 is likely to operate at elevated temperature. Creep deformation is one of the critical criteria for structural application of ceramics at elevated temperatures in operational environments. Compression creep of HfB_2 was studied at 1500°C in argon at stresses ranging from -25 to -75 MPa. Effects of SiC additive on creep of HfB_2 were examined via testing specimens of HfB_2 with 10, 20 and 30 vol% of SiC. Grain size of HfB_2 specimens was nearly an order of magnitude higher than those of HfB_2-SiC specimens. Such difference in grain sizes afforded an opportunity to assess the effects of grain size on creep. Minimum creep rates were measured. Stress exponents were between 1 and 1.8. Grain size exponent was ≈ 2. Addition of 10% SiC increased creep rates by two orders of magnitude, while additions of 20 and 30% SiC decreased creep rates by nearly two orders of magnitude. It is proposed that larger (20 and 30%) additions of SiC cause formation of a network of point-to-point contacts resulting in lower creep rates of HfB_2 with 20 and 30% SiC. A smaller (10%) addition of SiC is insufficient to produce such network; creep rates of HfB_2-10% SiC are governed by change in grain size.

11:30 AM

(ICACC-S12-017-2017) Evolution of Irradiation Defects in Ti_2AlC during Heavy Ion Irradiation

B. Cui*[1]; F. Wang[1]; Q. Su[2]; M. Nastasi[1]; 1. University of Nebraska, Lincoln, USA; 2. University of Nebraska–Lincoln, USA

Ti_2AlC is a typical MAX phase and a promising material for nuclear fuel cladding applications due to its irradiation tolerance and oxidation resistance in high temperature environments. However, the fundamental mechanism of the defect generation, clustering, and annihilation process in MAX phases such as Ti_2AlC in the irradiation environment is not well understood. This study examines the evolution of irradiation defects under Kr ion irradiation at room and elevated temperatures by using the IVEM-Tandem Facility at the Argonne National Laboratory. The dependence of defect size and density on the irradiation dose and temperature was characterized by in-situ transmission electron microscopy (TEM) experiments. The localized disorder of atomic positions in the irradiated Ti_2AlC was investigated in high-resolution TEM studies. These results suggest that although this material shows exceptional resistance to amorphization over a high level of irradiation dose, the localized nanoscale defect was accumulated in the microstructures which may cluster to larger defects, or annihilate at elevated temperatures.

11:50 AM

(ICACC-S12-018-2017) Ceramic Matrix Composites for Air Cooled Rotating Detonation Engines

D. King*[1]; G. Wilks[1]; T. Parthasarathy[1]; M. Cinibulk[1]; S. Theuerkauf[2]; C. Stevens[2]; 1. Air Force Research Laboratory, USA; 2. Air Force Research Laboratory, USA

Rotating Detonation Engines (RDEs) have the potential to provide remarkable fuel and thermodynamic efficiency over a wide range of speeds (0 to Mach 5), but in RDEs, materials are subjected to intense thermal and acoustic loads. With high efficiencies, RDEs typically operate at temperatures over the melting point of several super alloys, making ceramic matrix composites (CMCs) prime candidates for insertion into air cooled RDEs. Modelling of the thermal effects predict localized thermal stresses in the region close to the site of detonation. To increase the planar and through thickness thermal conductivities, which in turn would aid in the dissipation of detonation induced thermal stresses, HfB_2 particulate additions were incorporated in SiC_f/SiC CMCs for testing in a water cooled RDE. Optical microscopy and X-ray CT were used to compare predicted stresses with observed post-test damage in CMCs that experienced the high thermal gradients in water cooled RDE testing.

S14: Crystalline Materials for Electrical, Optical and Medical Applications

Scintillator

Room: Tomoka C
Session Chairs: Yimin Wang, Radiation Monitoring Devices, Inc.

8:30 AM

(ICACC-S14-011-2017) II-VI and Oxide Materials for Radiation Detection and Conversion (Invited)

J. McCloy*[1]; K. Lynn[1]; 1. Washington State University, USA

At the Center for Materials Research (CMR) at WSU, we have a long history of growth and evaluation of II-VI and oxide crystals for various applications. II-VI semiconductor materials are used in optoelectronic applications from photodetectors to phosphors to photovoltaics. In this presentation we outline the specific characteristics of II-VI materials which lend themselves to this diverse array of applications, and give a few examples of recent research. Our long ongoing research on Cd-Zn-Te crystals for room temperature gamma ray detection has led to recent research on CdTe for solar cells. Additionally, use of II-VI materials for scintillators is being investigated, primarily ZnS and ZnTe for alpha-particle and x-ray scintillation. At the same time, at CMR we have a long history of investigating oxide single crystals for scintillation and laser applications, with much of the work focusing on garnets such as YAG, as well as ferroelectric crystals. Finally, investigation of defect levels materials using a suite of tools at the CMR including thermoluminescence, photoluminescence, and thermo-electric emission spectroscopy (TEES), shows the power and utility of these techniques for understanding II-VI and oxide material behavior.

9:00 AM

(ICACC-S14-012-2017) High Energy Resolution Scintillators for Nuclear Nonproliferation Applications (Invited)

M. Zhuravleva*[1]; A. Lindsey[1]; L. Stand[1]; Y. Wu[1]; M. Koschan[1]; C. Melcher[1]; 1. University of Tennessee, USA

The goal of this work is to develop effective new scintillators that can be used for the detection of illicit radioactive materials. In order to unambiguously identify the specific gamma-ray signatures of radioactive elements, scintillator materials must possess energy resolution approaching 2% at 662 keV. Currently available radiation sensors have either inadequate energy resolution (NaI:Tl), unacceptably high cost (CZT and $LaBr_3$:Ce), or limiting operational burden (HPGe). Using the Bridgman technique, we have demonstrated that crack-free large-size crystals of KSr_2I_5:Eu and $KCaI_3$:Eu up to Ø1.5” can be reproducibly obtained using pyrolytic carbon-coated ampoules and a randomly oriented self-seeded approach. In order to demonstrate feasibility of producing these scintillators in large volume and at lower costs, the in-house constructed Multi-Ampoule-Growth-Station was employed to simultaneously grow up to five Ø1” crystals. The developed technique and equipment can be applied to melt growth of many other halide scintillators offering increased throughput of a conventional one-boule to one-furnace approach. An examination of performance of large crystals has determined that low dopant amounts of 0.5-1.0 at% of Eu can achieve excellent energy resolutions of 3.5% at 662 keV (for Ø1” × 1” cylinders) and 4.5% at 662 keV (for Ø1.5” × 1.5” cylinders) while minimizing deleterious self-absorption effects.

9:30 AM

(ICACC-S14-013-2017) The complementary relationship of scintillation and storage luminescence of inorganic crystalline materials (Invited)

T. Yanagida*[1]; 1. Nara Institute of Science and Technology, Japan

In solid state ionizing radiation detectors, mainly two types of luminescent materials, scintillators and dosimeter materials, have been investigated for a long time. The former one transduces the energy of high energy ionizing radiation (keV-GeV) to thousands of UV-visible photons immediately and the latter storages the energies of irradiated radiation. After the stimulation by visible photons (OSL) or thermal energies (TSL), dosimeter materials release the storage energies by UV-visible emission. Since basic processes, absorption of the energy of radiations and release of absorbed energies by luminescence, are the same, these emission intensities show a complementary relationship in some material systems by assuming the simple energy conservation. In this presentation, after introductions of basic principles of ionizing radiation induced luminescence phenomena in inorganic materials, we present some examples of the complementary relationship.

10:20 AM

(ICACC-S14-014-2017) X-Ray and Neutron Imaging Screens (Invited)

N. Cherepy*[1]; 1. Lawrence Livermore Nat'l Lab, USA

We are developing new polycrystalline ceramic and phosphor screens for ionizing imaging applications. This presentation will provide an overview of the optics and materials science behind optimizing the scintillation and light collection for imaging applications.

10:50 AM

(ICACC-S14-015-2017) Transparent Ceramic Scintillators for X-ray Radiography Applications (Invited)

Y. Wang*[1]; J. Glodo[1]; R. Shawgo[1]; C. Brecher[1]; W. Rhodes[1]; K. Shah[1];
1. Radiation Monitoring Devices, Inc., USA

Modern x-ray radography system use high energy x-ray sources to detect contraband materials in for cargo and trucks. Currently the choices for detector materials that can be used in high-energy radiography are limited. In oder to efficiently detect these high -energy x-ray, transparent scintillator materials with high density and high-atomic-number elements (high x-ray stopping power), low afterglow, high radiation hardness are required in this application. In this study a selection of high-density, high-Zeff sesquioxide scintillator ceramics such as Lu_2O_3:Eu, $(Gd,Lu)_2O_3$:Eu, and Lu_2O_3:Yb were investigated. Nano-sized powders are synthesized by co-precipitation. Phase structures, morphologies of the ceramics are examined by XRD and SEM, EDS. The relationships between processing parameters, powder morphologies, phase structures, microstructures and optical properties are discussed. Scintillation properties (emission, afterglow, light yield etc.) and optical properties of the different candidates are compared.

11:20 AM

(ICACC-S14-016-2017) Development of X-ray Imaging Plates with Sub-micrometer Resolution Based on Intervalence Change of Sm for Synchrotron Radiation Therapy (Invited)

G. Okada*[1]; J. Ueda[2]; S. Tanabe[2]; A. Edgar[3]; N. Kawaguchi[1]; T. Yanagida[1]; G. Belev[4]; T. Wysokinski[5]; D. Chapman[4]; S. Kasap[4]; 1. Nara Institute of Science and Technology, Japan; 2. Kyoto University, Japan; 3. Victoria University of Wellington, New Zealand; 4. University of Saskatchewan, Canada; 5. Canadian Light Source, Canada

Microbeam Radiation Therapy (MRT) is a potential cancer therapy technique that uses an intense X-ray beam produced by a synchrotron. In MRT, a spatially modulated beam, called microbeam, is delivered to a tumour. The microbeam contains considerably large X-ray doses, which are spatially distributed at the micron scale. For the latter reasons, the dosimetry technique in MRT requires (a) a micron-scale spatial resolution and (b) detection sensitivity over a large dose range, from a few to thousands of grays. In the past few years, we have established a new dosimetry technique for MRT applications. We use Sm-doped radiophotoluminescence (RPL) detector plate to detect the dose distribution of microbeam, in which valence conversion of Sm ($Sm^{3+} \rightarrow Sm^{2+}$) takes place by X-ray irradiation. Since the conversion stable and the concentration, in turn photoluminescence intensity, of Sm^{2+} corresponds to the delivered X-ray dose, the dose distribution is recorded on the plate. After the irradiation, the PL signals are read out using a confocal microscope so that the dose distribution is mapped at high resolution. With the use of Sm-doped RPL detectors, we have successfully implemented a dosimetry technique that can measure the dose variation across the microbeam, satisfiying the above fundamental requirements.

S15: Additive Manufacturing and 3-D Printing Technologies

Emerging Additive Manufacturing

Room: Coquina Salon B
Session Chair: Paolo Colombo, University of Padova

8:30 AM

(ICACC-S15-014-2017) In operando non-destructive evaluation techniques for additive manufacturing (Invited)

A. Michaelis*[1]; 1. Fraunhofer IKTS, Germany

Ceramic materials additive manufacturing (AM) offers the possibility to produce components of extremely complex geometry not achievable by conventional shaping routes. The ceramic AM-methods can be divided into powder based and suspension based techniques resulting in porous and dense components, resp. An important demand for all AM technologies is to improve reliability and performance of the manufactured components. Due to the time-consuming manufacturing processes and the layerwise building process it is necessary to control the quality of each layer for enabling the repair of a defect layer or to stop the building process for avoiding waste of time and expensive material loss. Therefore, it is important to access the quality of the printed parts as early as possible. This requires in-operando non-destructive evaluation methods. For this, we present new optical methods such as speckle laser spectroscopy and optical coherence tomography. For a further functionalization of the AM parts, 2D printing technologies can be applied. We present first results on this combination of 3D and 2D paste printing. Furthermore, examples for a combination of dense and porous structures for bio-medical applications are presented.

9:00 AM

(ICACC-S15-015-2017) Additive Manufacturing of Components for Turbine Engine and Electric Motor Applications

M. C. Halbig*[1]; M. Singh[2]; 1. NASA Glenn Research Center, USA; 2. Ohio Aerospace Institute, USA

Additive manufacturing approaches offer new capabilities for rapid proto-typing, final part fabrication, low cost processes, and broadened design envelopes. Components can become more geometrically complex, compact, multi-material, innovatively cooled, multifunctional, and integrated. The additive processes are being applied to components for turbine engines and electric motors. The materials include SiC fiber reinforced/SiC ceramic matrix composites (SiC/SiC CMCs), polymer composite materials, and multi-material systems which offer high payoff in aerospace applications due to such characteristics as light weight, high temperature capability, optimized designs, and tailored and gradient properties. SiC/SiC CMCs are being fabricated using binder jet and laminated object manufacturing (LOM) processes for turbine engine components. A new project which utilizes additive processes to enable new innovative electric motor designs will be presented for achieving much higher power densities and/or efficiencies. The optimization of stators and other motor components is discussed. Direct printing of advanced electrically conductive coils will be demonstrated.

9:20 AM

(ICACC-S15-016-2017) 3D Printing of Kaolinite Clay Ceramics

C. Revelo[1]; H. A. Colorado*[1]; 1. Universidad de Antioquia, Colombia

Additive manufacturing process (AM) by an extrusion method of kaolinite based-clays was used to produce cylindrical samples for compression as other more complex shapes typically made with traditional ceramic methods. Different samples made out of clays were built with different water to clay ratios and parameters. The characterization was conducted by compression, scanning electron microscopy, and X-ray diffraction tests before and after samples were exposed to high temperatures. Viscosity and dimension stability tests were conducted before the high temperature process. Results are very promissory for the fabrication of new and traditional products.

9:40 AM

(ICACC-S15-017-2017) A Novel Cell Laden TiO_2 Nanotube-Hydrogel Composite Bioink for Bioprinting of Dental and Orthopedic Structures

S. Shafiee*[1]; E. Firlar[1]; S. Patel[1]; T. Shokuhfar[1]; 1. UIC, USA

Promoting the calcification of scaffolds or implants in orthopedic and dental implant applications have been the subject of interest and investigation for many years. However, there still exist a lack of a practical method to fabricate a predesigned cell laden scaffold. In this study, we developed a novel composite of TiO_2 nanotubes reinforced gelatin methacrylate hydrogel as a cell laden bioink for 3D bioprinting applications. Controlled mechanical properties of this bioink was ensued by variation of TiO_2 nanotubes concentration within the hydrogel. Using the same hydrogel as the base of the bioink, provided the capability to fabricate a variety of structures with different mechanical properties mimicking the gingival margin. Printability and resolution of bioprinting of this cell laden composite-bioink were examined using a dual head 3D bioprinter. The porosity, rheological behavior and mechanical properties of this bioink were also investigated to optimize the hydrogel/ nanotube ratio to provide the highest possible resolution during the extrusion bioprinting process. Biocompatibility and calcification capacity of this bioink were measured by different methods of microscopy and incubation of samples in artificial saliva, respectively.

Direct Writing & Fused Deposition

Room: Coquina Salon B
Session Chair: Michael Halbig, NASA Glenn Research Center

10:20 AM

(ICACC-S15-018-2017) Direct ink-writing of ceramic matrix composites from a preceramic polymer and fillers

G. Franchin*[1]; L. Wahl[1]; P. Colombo[1]; 1. University of Padova, Italy

We will present a novel approach for the production of ceramic matrix composite (CMC) lattices, which is the application of direct ink-writing (DIW) process to a system comprising a preceramic polymer and fillers followed by pyrolysis and thus ceramization. The incorporation of fibers to a preceramic polymer opens the possibility of generating complex ceramic shapes with enhanced mechanical properties. We will show our latest developments on the DIW of a novel composition for developing a CMC from a mixture containing chopped carbon fibers and SiC powder as fillers. Upon pyrolysis in inert atmosphere (nitrogen), the preceramic polymer (a silicone resin) converts into an amorphous SiOC phase. Suitable formulations were investigated for the extrusion of fine filaments (~400μm diameter) through a nozzle. We will discuss the conditions and requirements for the production of highly porous lattices with spanning features and the importance of gel formation. We will debate the role of the different fillers (carbon fibers, SiC powder, fumed silica) on the rheology, morphology and crushing strength of the highly porous lattices produced.

10:40 AM

(ICACC-S15-019-2017) Tribological Behavior of PLA and ABS-Based Polymer Matrix Composites Fabricated by Fused Deposition Modeling Process

S. Gupta*[1]; M. Fuka[1]; R. Dunnigan[1]; M. C. Halbig[2]; M. Singh[3]; 1. University of North Dakota, USA; 2. NASA Glenn Research Center, USA; 3. Ohio Aerospace Institute, USA

Fused deposition modeling (FDM) process 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 3-D printing process. In this presentation, we report the tribology of novel PLA and ABS matrix composites. More particularly, the effect of critical parameters like printing speed, particulate content, and chemistry will be studied on the tribological behavior of different composites. As a part of this study, a novel method will be presented for studying the tribo-films formed during the tribological process by utilizing detailed SEM and EDS analysis.

11:00 AM

(ICACC-S15-020-2017) Bi-Material Additive Manufacturing: Tensile Study of Additively Manufactured Polymer Composites

G. Acosta Quiros*[1]; M. Singh[2]; M. C. Halbig[3]; B. A. Lerch[3]; 1. University of Miami, USA; 2. Ohio Aerospace Institute, USA; 3. NASA Glenn Research Center, USA

Additive manufacturing has evolved quickly in capabilities, selection of materials, and flexibility in design parameters since its creation in the late 1980s. In seeking to further advance the field of additive manufacturing, a study into the properties of bi-material ABS composites manufactured using Fused Deposition Modelling was undergone. Based on research into homogenous 3D printing of single material filaments, an exploration into the tensile properties of more exotic materials modeled into complex composites was completed. Using novel technologies like 3D printing and photogrammetry, it was discovered that the Elastic Modulus of bi-material composites of ABS and ABS infused with chopped Carbon Fibers was 10-30% higher than that of standard ABS alone. After reviewing the conditions of manufacturing and processing, it was determined that much more research into multi-material printing should be started. It was also determined that a useful model is yet to be created for this form of manufacturing.

3rd Pacific Rim Engineering Ceramics Summit

Energy and Environmental Issues I

Room: Coquina Salon C
Session Chairs: Makio Naito, Osaka University; Wei Pan, Tsinghua University

1:30 PM

(ICACC-PACRIM-015-2017) Smart powder processing of advanced materials for energy and environments (Invited)

M. Naito*[1]; T. Kozawa[1]; A. Kondo[1]; 1. Osaka University, Japan

Smart powder processing is a green and sustainable powder processing technique that creates advanced materials with minimal energy consumption and environmental impact. Particle bonding 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. By making use of particle bonding, a new "one-pot" processing method to synthesize nanoparticles without applying extra heat was also developed. Furthermore, one-pot processing achieving both the synthesis of nanoparticles and their bonding with other kinds of particles to make nanocomposite granules was also developed. The assembling of these composite particles and granules will lead to the control of nano/microstructure of materials and can produce new materials with a simpler and energy-saving manufacturing process. In this presentation, the applications of smart powder processing for energy and environments related area will be introduced. One-pot synthesis of the composite nanoparticles for the cathode materials of lithium ion batteries and that of YAG phosphors for white light emitting diodes will be explained.

2:00 PM

(ICACC-PACRIM-016-2017) An overview of the intelligent self-healing catalyst for automotive emissions control (Invited)

H. Tanaka*[1]; 1. Kwansei Gakuin University, Japan

An automotive emissions control catalyst can call one of the major applications of ceramics. A monolithic substrate of Cordierite plays the very important role to the gasoline engine vehicles. And Ceria-Zirconia complex oxides with the oxygen storage capacity, as well as high heat-resistant g-Alumina, are used widely as the washcoat materials. Since the catalytic reaction occurs at the interface, the precious metal as an active center has been mainly studied. Ceramics have been recognized as auxiliary materials for making the precious metal highly disperse. In the automotive catalyst, the state of exhaust gas, such as composition, temperature, and flow rate, is changed dynamically. In this environment, the ceramic is not only supporting the precious metal, it is possible to conduct itself more active. An intelligent catalyst is introduced here as such examples. It is a perovskite-type catalyst having a self-healing function of the precious metal in the real use environment. The catalyst durability is improved by the self-healing function, the amount of the precious metal can be significantly reduced as a result. Here, together with an overview of its development, it is introduced about the latest analysis of the interaction of the reaction gas, precious metals, and perovskite-type ceramics using synchrotron radiation.

2:30 PM

(ICACC-PACRIM-041-2017) Laser Drilling and Cutting Using Fiber Lasers (Invited)

S. Jiang*[1]; 1. AdValue Photonics Inc, USA

Fiber lasers have attracted intense interest in recent years because of its outstanding performance compared to other types of lasers for industrial application. In this presentation, we report our latest developments of high peak power and high repetition rate green lasers at 515nm wavelength based upon our innovative fiber laser technology. Laser drilling and cutting on ceramics and glasses will be presented.

3:20 PM

(ICACC-PACRIM-018-2017) Development of Low Thermal conductivity Materials by Defect Engineering (Invited)

W. Pan*[1]; 1. Tsinghua University, China

Low thermal conductivity is the key property dominating the heat insulation ability of thermal barrier coatings (TBCs). A reduction of thermal conductivity will surely lead to an improvement of the operating temperature and energy efficiency of gas turbines and jet-engines without further deteriorating the harsh environment faced by the superalloy components, such as blade and vanes. Over the last decade or so, novel fabrication technologies have been developed to optimize the coating architectures and therefore enhance the comprehensive thermophysical properties at high temperature. Yet, reducing the intrinsic thermal conductivity of the TBC materials is still the major topic for developing advanced TBCs. Defect engineering has attracted much attention in seeking better TBC materials since lattice defects play a crucial role in phonon scattering and thermal conductivity reduction, including vacancies, substitutions and interstitials. Among them, oxygen vacancies and substitutions are proven to be the most effective, while the accompanying lattice distortion is also of great importance. In this presentation, recent advances of reducing the thermal conductivity of potential thermal barrier coating materials by defect engineering are comprehensively reviewed.

3:50 PM

(ICACC-PACRIM-019-2017) Microstructure Evolution and Fabrication of Low Thermal Conductivity ZrO_2 Based Composites (Invited)

B. Jang*[1]; Y. Oh[2]; S. Kim[2]; H. Kim[2]; 1. National Institute for Materials Science (NIMS), Japan; 2. Korea Institute of Ceramic Engineering and Technology, Republic of Korea

The thermal efficiency of gas turbine engines in the highest operating temperature is significantly improved by employing ceramic-based thermal barrier coatings (TBCs), which are basically deposited on bond-coat coated superalloy turbine airfoils by means of electron beam physical vapor deposition (EB-PVD) or by air plasma sprayed coating (APS). The state-of-the-art material for these TBCs, partially stabilized zirconia of ZrO_2-4mol%Y_2O_3 (YSZ), exhibits outstanding properties, such as low thermal conductivity, low Young's modulus, and a relatively high thermal expansion coefficient. This work describes the influence of Gd_2O_3 addition on the thermal conductivity of ZrO_2-4mol% Y_2O_3 ceramics is fabricated by spark plasma sintering. Commercial starting powders of ZrO2-4mol% Y_2O_3 (TZ4Y, Tosoh Co.) and Gd_2O_3 were used for fabricating the present samples. The additions of Gd_2O_3 in the ZrO_2 matrix are 0, 1, 3 and 5 mol%. The sintering temperature was 1100°C and 1500°C under a vacuum of 10-2 Pa. Sintering was conducted for 20 min at each sintering temperature under pressure of 30MPa. The thermal diffusivity and thermal conductivity of sintered YSZ decreased with increasing Gd_2O_3 addition.

4:20 PM

(ICACC-PACRIM-020-2017) The study on process variables of SiC Radiant Tube including In-situ Joining (Invited)

Y. Kim*[1]; H. Shin[1]; 1. Inocera inc., Republic of Korea

This study is designed to investigate effects of process variables of SiC radiant tube. As it has many merits like high thermal efficiency and low pollutant emission, RBSC radiant tube will be used gradually for heat treat applications. Joining is inevitable to use RBSC tube for Radiant applications. Starting materials are composed of batch of SiC powders and thermo-set polymers. These powders were mixed with thermo-set polymer in a chamber at low temperature. Pouring it into the casting mold, tube was formed by centrifugal force and heat aid. As the formed bodies were degreased, Joining paste were put on each face and pressed by 10MPa. Joining is done with molten Si infiltration at 1500C in vacuum at the same time. It was studied how process conditions such as casting variables, joining paste, joining method, heat treatment variables and Si infiltration variables affect on materials properties. The flexural strength, density and porosity were measured and microstructures were observed using Optical Microscope and SEM.

4:40 PM

(ICACC-PACRIM-026-2017) Characterization of ZrN, ZrO_2 and β'–$Zr_7O_{11}N_2$ nanoparticles synthesized by PWD

L. V. Do*[1]; H. Suematsu[1]; T. Ogawa[1]; 1. Nagaoka University of Technology, Japan

Nanoparticles of ZrN, ZrO_2 and β'–$Zr_7O_{11}N_2$ were synthesized by pulsed wire discharge (PWD) using Zr wire in the atmosphere containing various O_2/N_2 mixture with different ratios. The syntheses were carried out at relative energy ratio at (K) of 6.4. For the gas mixtures in the reaction chamber, a total pressure of 100 kPa was adopted, O_2/N_2 gas mixtures has the ratio steadily changed from 100% N_2 to 40% N_2 and 60% O_2. Morphology and phases existence analysis were made on these nanoparticles by X-rays diffraction and field-emission transmission electron microscope(FE-TEM). ZrN, Zr_2N were observed in sample synthesized in 100% N_2 reaction atmosphere, formation of β'–$Zr_7O_{11}N_2$ and ZrO_2 was started when O_2 was introduced together with N_2 in the reaction gas mixture, starting at 1% total pressure. By bright field image, detail EELS composition analysis and SAD, nanoparticles of ZrN, β'–$Zr_7O_{11}N_2$ and ZrO_2 are separately characterized. In these syntheses, nanoparticles of β'–$Zr_7O_{11}N_2$ existed in much more smaller size and different shape than the ordinary spherical nanoparticles of ZrN and ZrO_2. In gas mixtures where O_2 contents are larger than 22% (dry air), ZrN was not detected or detected with just a fractional amount compare to 2 major phases of β'–$Zr_7O_{11}N_2$ and ZrO_2. Compare to nitrogen, oxygen species are considered to be much more susceptible to the Zr plasma vapor generated by arc discharge.

6th Global Young Investigator Forum

Magnetic and Electric, Energy Conversion and Energy Storage

Room: Coquina Salon F
Session Chair: Daniele Benetti, Institut National de la Recherche Scientifique

1:30 PM

(ICACC-GYIF-019-2017) Fabrication of metal halide perovskite nanocrystals and their applications in photo- and piezo-electronic device (Invited)

X. Zhang*; 1. Southern University of Science and Technology, China

Recent years, the emergence of metal halide perovskite materials has drawn the increasing attention for their promising characteristics in the application of highly efficient photovoltaic devices for energy harvesting. The perovskite nanocrystal behaves like quantum dots (QDs), which presents high efficiency quantum yield, tunable bandgap, and narrow-band emission. Therefore, the metal halide perovskite is also emerging as a new and excellent semiconductor for various kinds of optoelectronic applications, such as LED, lasing, photodetectors, x-ray diffraction detection and plasmonics. Moreover, we demonstrate the first high performance polymer assisted hybrid composite piezoelectric nanogenerators based on metal halide perovskite nanocrystals.

2:00 PM

(ICACC-GYIF-020-2017) Efficient Processing of Heavily Filled Composites by UV Curing

V. Christensen*[1]; M. Krohn[2]; P. Chan[3]; A. Natarajan[3]; 1. The Pennsylvania State University, USA; 2. GE Oil & Gas, USA; 3. GE Global Research, USA

1-3 piezoelectric ceramic-polymer composites are widely used to make transducers that can be implemented in a variety of applications. A traditional thermal cure epoxy system has been the material of choice for composite fabrication; however this material system has a lot of limitations, ranging from high moisture absorption, high cure shrinkage, and long curing time. We found that UV curing formulations can be very robust and can be used repeatedly to generate consistent results while reducing production time. Understanding the kinetics of UV curable resins is important in determining the optimal cure settings to support the variety of configurations required for 1-3 composite manufacturing. UV formulations of epoxy resins were studied with the addition of five different aromatic hydrocarbon and aromatic ketone photosensitizers to increase the cure depth while minimizing shrinkage. A model relating the UV transmission to the desired cure depth was developed as a tool for manufacturing to ensure proper settings that produce yields with limited scrap. Fourier transform infrared spectroscopy (FTIR) was leveraged to characterize the kinetics of the UV curing and to validate full depth of cure was achieved for a variety of composite configurations. By properly formulating a UV curable resin with the addition of a photosensitizer, production time for heavily filled 1-3 piezoelectric composites was reduced by 96 percent.

2:20 PM

(ICACC-GYIF-021-2017) Colloidal Quantum Dots for Efficient and Durable Photoelectrochemical Hydrogen Production

L. Jin*[1]; H. Zhao[1]; A. Vomiero[2]; F. Rosei[1]; 1. Institut National de la Recherche Scientifique, Canada; 2. Luleå University of Technology, Sweden

The increasing demand in sustainable energy has fueled considerable efforts toward the development of photoelectrochemical (PEC) H_2 generation. Quantum dots (QDs) have been developed as light absorbers to sensitize the wide band gap metal oxides such as TiO_2, to enhance light absorption. However, a major challenge is the high charge recombination and limited photostability due to their surface sensitivity. An elegant solution to address this challenge consists in using core/shell QDs. We developed several PEC photoanode, which is composed of a TiO_2 mesoporous frame, functionalized by colloidal core/shell QDs via electrophoretic deposition (EPD). To facilitate the charge separation, compositions and electronic band structures were suitably tailored. The optimization of charge transfer by suitable engineering of the composition/thickness of the external shell and of the surface capping agents is ongoing to develop a class of QDs optically active in the near infrared region for solar energy and optoelectronic applications.

2:40 PM

(ICACC-GYIF-022-2017) Enhanced Decomposition of Plastic Waste through Photocatalysis

T. Colburn*[1]; T. Toops[1]; 1. Oak Ridge National Lab, USA

Polymer pollution is a dangerous environmental concern that can lead to the spread of toxic chemicals throughout aquatic biomes. Littered plastic from consumer goods, biohazardous medical waste, and products with a limited or single use can be photocatalytically decomposed to avoid an increase in the amount of plastic present in Earth's bodies of water. The photocatalysts, titanium dioxide, zinc oxide, tungsten trioxide, and iron oxide nanoparticles, less than 100 nm particle size, were embedded into polyethylene terephthalate, high-density polyethylene, low-density polyethylene, and polystyrene and exposed under ultraviolet light over two independent phases. The plastics were chosen due to their prolific use in items such as grocery bags and bottles. Photocatalyst particles have solar reactivity due to their threshold energy and release electrons to aid in the decomposition of the polymer backbone. Phase One examined the effects of water and light frequency on polymer decay, and Phase Two examined photodecomposition of polymer thin-films. Low-density and high-density polyethylene thin-films with titanium dioxide decayed approximately 90% faster than control samples. With an estimated price of less than half the cost of the bag for titanium catalyst integration in plastic bags and given research-grade materials, photocatalytic decomposition is a method for the reduction of hydrocarbon build-up in the environment.

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

Room: Coquina Salon F
Session Chair: Isabel Gessner, University of Cologne

3:20 PM

(ICACC-GYIF-023-2017) Impact of Deposition Technique and Process Parameter on Titanium Oxide and BFCO Thin Film Deposition

D. Benetti*[1]; R. Nouar[2]; R. Nechache[1]; H. Pepin[1]; A. Sarkissian[2]; F. Rosei[1]; J. MacLeod[3]; 1. Institut national de la recherche scientifique, Canada; 2. Plasmionique Inc, Canada; 3. Queensland University Of Technology, Australia

We report on characterization of Bi_2FeCrO_6 (BFCO) and TiO_2 thin films deposited with different techniques. BFCO, a bismuth-based inorganic double perovskites, has recently been widely studied due to its interesting proprieties, such as multiferroic and photovoltaic behavior. TiO_2, on the other side, it is one of the most studied photocatalytic materials. The films were synthesized using magnetron sputtering (MS), pulsed laser deposition (PLD) and a hybrid deposition technique that involves the simultaneous use of the two techniques. In view of the scale-up of the process to the industrial level, low power operation of both the laser and the sputtering system were investigated. A multi-technique approach was applied for material characterization, by using atomic force microscopy (AFM), scanning electron microscopy (SEM), X-ray diffraction (XRD) and reflection (XRR) analysis. We compared films deposited by conventional PLD, MS and the new hybrid technique. We show that the new hybrid technique offers an increased deposition rate (+50%) and produces denser films (+20%) than those produced by MS or PLD alone. Large clusters, typically observed in the PLD-deposited films, were also eliminated under optimized processing conditions, resulting in a smoother surface morphology.

3:40 PM

(ICACC-GYIF-024-2017) Gas Phase Synthesis of Nanostructures Materials Using Molecular Precursors

T. Fischer*[1]; Y. Gönüllü[1]; S. Mathur[1]; 1. University of Cologne, Germany

The full potential of Chemical Vapor Deposition (CVD) techniques can be maxed when using tailored molecular precursors with a predefined arrangement of atoms on the molecular level, thus facilitating solid phase formation at relatively low temperatures. Moreover by changeing the ligand sphere in precursor molecules alternate decomposition pathways can be followed resulting in non-stochiometric or even meta-stable phases for coatings and/or fabrication of nanostructured materials. Only a precise control over the reaction conditions (i.e. temperature, pressure, flow, etc.) as well as an analysis of the ongoing reaction in the gas phase as well as at the solid/gas interface gives access to a complete understanding of the whiole deposition processes vital to a full process control. Providing catalyst centers on the substrate, anisotropic growth can be induced, thus resulting in one-dimensional metal oxide nanowires. Tne following presentation will highlight the aspects of reactor as well as process design and control for the synthesis and integration of nanostructured films of various metal oxides and showcase how in-situ diagnostics using i.e. mass spectrometrical anaysis can support the processs development in gas phase deposition reactions.

4:00 PM

(ICACC-GYIF-025-2017) Transition metal (Cr, Mn, and Fe) doping into Ti_3AlC_2 to fabricate doped-MXene

K. Shima*[1]; K. Kawahara[1]; M. Inada[1]; N. Enomoto[1]; K. Hayashi[1]; 1. Kyushu University, Japan

MXene is emerging nanosheet compound produced by selective etching of A element in MAX phase. So far, M for stable MAX has been restricted up to early transition metals, and full substitution of middle transition metals seems to be difficult. This study examines modification of physical and chemical properties of MAX and relevant MXene by doping of the middle transition metals to M. A representative MAX of Ti_3AlC_2 was chosen as the matrix. Partially-substituted Ti_3AlC_2 with compositions of $Ti_{2.72}Cr_{0.28}AlC_2$, $Ti_{2.72}Mn_{0.28}AlC_2$, and $Ti_{2.72}Fe_{0.28}AlC_2$ were synthesized by spark plasma sintering (SPS). These products were soaked in a hydrofluoric acid for 24 h to convert to MXene. Formation of MXene was confirmed by XRD pattern that exhibits widening in basal plane of original MAX, and observation of peculiar accordion-like microstructure by SEM. Residual concentration ratio of Cr, Mn, and Fe to Ti, measured by XRF, was 3.8, 0.6, and 0.2 %, respectively. Marked losses of the dopants are seemingly ascribed to their distribution in co-formed other phases during the SPS and preferred dissolution in the hydrofluoric acid.

4:20 PM

(ICACC-GYIF-026-2017) Stabilization through epitaxial strain of the magnetoelectric ε-Fe_2O_3

L. Corbellini*[1]; C. Lacroix[2]; C. Harnagea[1]; D. Ménard[2]; A. Pignolet[1]; 1. INRS, Canada; 2. Ecole Polytechnque de Montreal, Canada

ε-Fe_2O_3 is a metastable intermediate phase of iron(III)oxide, between γ- and α-Fe_2O_3. ε-Fe_2O_3 has been investigated primarily because of its ferrimagnetic ordering with a T_C of circa 500K. However, given its belonging to the polar space group $Pna2_1$, it is characterized by pyroelectricity, making it a magnetoelectric material. ε-Fe_2O_3 needs to be stabilized at RT, whic has been obtained mainly by synthesizing it by sol-gel as size-confined nanoparticles embedded inside a SiO_2 matrix. Recently however, deposition of epitaxial thin films of ε-Fe_2O_3 on STO (111) has been shown, where stabilization is due to the epitaxial strain. We report the growth by PLD of epitaxial thin films of ε-Fe_2O_3 on different single crystal substrates (STO, LAO, YSZ and Si), and discuss the influence of the substrate on the structural and functional properties. The ability of growing thin films both on hexagonal substrates like STO and LAO (111) and on cubic substrates like YSZ and Si (100) will help to understand the twinning mechanism which was observed resulting from the lower symmetry of the deposited film in comparison with the substrates. Moreover, depositions over vicinal substrates have been performed and successfully promoted the growth along only one crystallographic direction, which represent a fundamental advance given how some application, e.g. memories, require a twin-less film in order to be implemented into devices.

4:40 PM

(ICACC-GYIF-027-2017) Magneto-transport properties of magnet-electric multilayers using ferroelectric tunnnel junction

T. Yokota*[1]; S. Maeda[1]; J. MIyabe[1]; 1. Nagoya Institute of Technology, Japan

Magneto-electric (ME) materials have attracted much attention as a new generation of magneto-electronics. Ferroelectric tunnel junctions (FeTJ) also have attracted much attention to enhance functional properties of tunneling magnetoresistance (MTJ) devices by its spontaneous electric polarization. In this presentation, we prepared a ME-FeTJ which is Cr_2O_3(CO) /$LiNbO_3$ (LNO)/ Cr_2O_3(CO) epitaxial layer. It is expected that it does't only work as a resistance switching devices, but also works as a capacitance switching devices. We also investigate the magneto-transport properties of MFeTJ and discuss the role of FeTJ on the devices. The sample was prepared using a radio-frequency magnetron sputtering method. A Nb-doped $SrTiO_3$ 111 single crystal was used as a substrate. The thickness of each film was changed from 30 to 15 nm for CO and from 5 to 15 nm for LNO. For MFeTJ, we used Fe and $Fe_{82}Ni_{18}$ of 20 nm as a ferromagnetic layer, respectively. The MFeTJ sample showed current jump at around 3.3 x10^{-4} V, which corresponds a polarization switching voltage. By the application of the magnetic field, the voltage was changed and showed hysteresis. The capacitance value of ME-FeTJ is also changed at around the same voltages. It is revealed that the capacitance change can be explained using similar mechanism of the electric and magnetic multiple state of MFeTJ originated from polarization state.

FS2: Advanced Ceramic Materials and Processing for Photonics and Energy

Multi-functional Materials II

Room: Halifax A/B
Session Chairs: Clara Santato, Ecole Polytechnque de Montreal; Farid Akhtar, Stockholm University

1:30 PM

(ICACC-FS2- 013- 2017) Multifunctional Cerium Oxide Nanoparticles (Invited)

E. Traversa*[1]; 1. Xi'an Jiaotong University, China

Cerium oxide nanoparticles (nanoceria) possess the unique feature of tolerating in the fluorite structure large amounts of Ce^{3+} ions, with a compensating large concentration of oxygen vacancies. This non-stoichiometric concentration imparts outstanding properties to nanoceria, especially in the catalysis field. Recently, nanoceria have been reported to show outstanding biomedical activity, acting as well tolerated anti-age and anti-inflammatory agents. The potential pharmacological applications are due to redox changes in the Ce oxidation state (Ce^{4+}/Ce^{3+}) that trigger the abatement of intracellular reactive oxygen species (ROS), hindering the oxidative stress cytotoxic effects. For biomedical applications, well-dispersed nanoparticles are needed, where the dispersibility cannot be obtained with toxic surfactants. In this talk, new synthetic methods that allow the synthesis of nanocrystalline ceria at room temperature will be presented, together with a functionalization that improved colloidal dispersion even improving the biological activity. Applications of nanoceria in biomedical and energy fields will be reported, including as antioxidant UV screen shields and as electrodes in microbial fuel cells.

2:00 PM

(ICACC-FS2- 014- 2017) Design and Engineering 1D/2D materials for clean energy applications (Invited)

D. H. Chua*[1]; 1. National University of Singapore, 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. With this flexibility, researchers are able to fabricate other materials such as nanowires and TMDs respectively. For direct applications on (e.g.) PEM fuel cells or Supercapacitors or Li batteries, one still requires careful materials design to engineer various 1D and 2D carbon and non-carbon-based materials. We will further show and compare how various devices, such as fuel cell properties can vary significantly when specifically designed 2D hybrid materials are used instead of simple mix and match system. 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

2:30 PM

(ICACC-FS2- 015- 2017) Nano-structured Hierarchically Porous Adsorbents for Clean Energy (Invited)

F. Akhtar*[1]; 1. Stockholm University, Sweden

Nanoporous materials hold great potential for catalysis, gas separation and gaseous fuel storage applications. These materials such as Zeolites, metal organic frameworks, activated carbons and aluminum phosphates are high surface area porous materials and are invariably produced in form of powder. These powders are required to be assembled into mechanically strong hierarchically porous macroscopic bodies for gas separation, storage and controlled release of gaseous fuels. In this regard, recent developments on processing of nanoporous powders will be presented to produce hierarchically porous structured materials to overcome the limitations of conventional materials for decarbonization, biomethane production and safe storage of gaseous fuels. We will demonstrate that binderless processing is a versatile tool to structure nanoporous materials and therefore outperforms conventionally structured porous powders in all aspects of post-combustion decarbonization of gas streams including CO_2 uptake capacity, high CO_2 over CH_4 and CO_2 over N_2 selectivity, rapid uptake and release kinetics and mechanical stability. We will discuss structuring of ultra-thin laminates and laminated devices processed by advanced structuring approaches for biomethane production and safe storage and handling of gaseous fuels.

3:20 PM

(ICACC-FS2- 016- 2017) Multifunctional metal oxide films: Exploring the combination of electrochromism and electrochemical energy storage in electrolyte-gated transistors (Invited)

C. Santato*[1]; 1. Ecole Polytechnque de Montreal, Canada

In this paper, we report on our recent observations about the possibility to combine several functional properties in metal oxide films with good transparency in the visible region of the solar spectrum (WO_3, TiO_2 and SnO_2), interfaced to a wide range of electrolytes (aqueous saline solutions, ionic liquids and quasi-solid state ion gels). These properties are: modulation of the electrical conduction in the electrolyte-gated transistor configuration, electrochromism and electrochemical energy storage (supercapacitor configuration). Physical deposition techniques permitted us to controll the stoichiometry of the metal oxides, thus permitting a good control over the surface properties of the oxide, in turn relevant to properly control the modulation of the electrical conductance under electrica bias. Solution-based techniques have been employed for film fabrication to explore large-area applications. Our research advances the fundamental understanding of how charge carrier density is modulated in metal oxide films, paralleled by optical density changes and charge stored/liberated with the aim to demonstrate high performance multifunctional glasses for energy saving and storage.

3:50 PM

(ICACC-FS2- 017- 2017) Photolithographically Patterned Metal Oxide Electrolyte-Gated Transistors (Invited)

F. Cicoira*[1]; I. Valitova[1]; 1. Polytechnique Montreal, Canada

Metal oxides are among the most interesting materials for next generation electronics. We fabricated both solid and flexible electrolyte gated (EG) TiO_2 and SnO_2 transistors making use of high surface area activated carbon (AC), 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 metal oxides have been deposited by thermal evaporation and sol gel techniques and were photolithographically patterned as transistor channel material. To explore the effect of the double layer capacitance on device performance we investigated bottom-contact top-gated transistor where we varied the area of the active layer in contact with electrolyte and area of overlap of source/drain electrodes with gate. To shed light on the doping mechanisms of such transistors we performed electrical measurements, cyclic voltammetry and electrochemical impedance spectroscopy. We demonstrated that patterning improves the performance of electrolyte- gated TiO_2 transistors with respect to their unpatterned counterparts. Patterned EG TiO_2 transistors show threshold voltages of about 0.9 V, ON/OFF ratios as high as 1×10^5 and electron mobility above 1 $cm^2/(V\ s)$.

[3] We believe that these simple architecture devices working at low voltages are promising for low cost, flexible and large area electronics.

4:20 PM

(ICACC-FS2- 018- 2017) Nanoscale interface formation in Graphene and 2D MoS_2 layers based novel 2D junction devices (Invited)

B. R. Mehta*[1]; 1. Indian Institute of Technology Delhi, India

For further improving the efficiency of photovoltaic conversion in single junction devices, use of 2D materials offers unique advantages. It is important to carryout nanoscale investigations of the effect of 2D nature and number of layers on photon absorption, carrier separation and interface formation. In this presentation, results on the KPFM based investigation of graphene-Si and MoS_2-ZnS and MoS_2-WS_2 2D junctions will be presented.. Surface potential changes at the graphene -Si junctions under dark and light conditions have been examined using Kelvin probe force microscopy investigations in surface and junctions configurations. V_{oc} nanoscale maps derived from these measurements show that topographical impurities and wrinkled boundaries on the graphene surface affect junction performance. In a separate study, patterned MoS_2 2D layers having feature size varying from 10 um to 1 um have been grown by a combination of stencil lithography and magnetron sputtering technique. 2D heterojunction having mono and few layers MoS_2 thickness result in high interface photovoltage in comparison to bulk MoS_2 layer. These results confirm active participation of 2D MoS_2 layer in photon absorption and charge separation and presents a step forward towards fabricating 2D junction based devices.

S1: Mechanical Behavior and Performance of Ceramics & Composites

Mechanical Testing

Room: Coquina Salon D

Session Chairs: Jonathan Salem, NASA Glenn Research Center; George Quinn, American Dental Association Foundation

1:30 PM

(ICACC-S1-022-2017) Flaw Type Effects in Slow Crack Growth Testing (Invited)

J. Salem*[1]; 1. NASA Glenn Research Center, USA

Two flaw types are commonly used in estimation of slow crack growth parameters from strength tests of ceramics and glasses: indentation and grinding or polishing damage. In some materials, indentation has the advantage of reducing scatter in the data and easing parameter estimation. However, different and possibly misleading parameters can be derived for coarse grain materials and materials exhibiting little SCG. Grinding damage, when consistently produced, has the advantage of producing numerous flaws for sampling and is thereby likely to produce realistic engineering parameters. Polishing flaws have the latter advantage, but result in more scatter and less confidence in parameters. Flaw type effects are demonstrated for different brittle material systems: those without a microstructure (glass and fused silica) ; one without SCG (Germanium), one with a coarse grain structure (AlON); and one with a finer microstructure relative to the indentation. Ramifications for standardization are discussed.

2:00 PM

(ICACC-S1-023-2017) International Standards for Properties and Performance of Advanced Ceramics: Over Three Decades of High-Quality and Rigorous ASTM Standards

M. G. Jenkins*[1]; J. Salem[2]; S. T. Gonczy[3]; J. D. Helfinstine[4]; G. D. Quinn[5]; 1. Bothell Engineering and Science Technologies, USA; 2. NASA Glenn Research Center, USA; 3. Gateway Materials Technology, Inc., USA; 4. Corning Incorporated, USA; 5. National Institute for Standards and Technology, USA

Mechanical and physical properties/performance of brittle bodies (e.g., advanced ceramics and glasses) can be difficult to measure correctly unless the proper techniques are used. For over three decades, ASTM Committee C28 on Advanced Ceramics, has developed numerous full-consensus standards (e.g., test methods, practices, guides, terminology) to measure various properties and performance of monolithic and composite ceramics and coatings that, in some cases, may be applicable to glasses. These standards give the “low-down” for determining many mechanical, physical, and thermal properties and performance thereby provideing accurate, reliable, repeatable and complete data. Involvement in ASTM Committee C28 includes users, producers, researchers, designers, academicians, etc. who write, continually update, and validate through round robin test programmes, nearly 50 standards over the three decades since the Committee’s inception in 1986. Included in this poster is a pictogram of the ASTM Committee C28 standards and how to obtain them either as i) individual copies (physical or digital) with full details or ii) a complete collection in one volume. A listing of other ASTM committees of interest is included. In addition, some examples of the tangible benefits of standards for advanced ceramics demonstrate their practical application.

2:20 PM

(ICACC-S1-024-2017) Fracture Toughness of Glasses as Measured by the SCF and SEPB Methods

G. D. Quinn*[1]; J. Swab[2]; 1. National Institute of Standards and Technology, USA; 2. US Army Research Laboratory, USA

The fracture toughness of five glasses was measured by the surface crack in flexure (SCF) and single-edged precracked beam (SEPB) methods. Outcomes depended upon the loading rate as well as the environment. At the 2016 ICACC Daytona conference, we presented SCF data that seemed a bit high, and it was suggested that crack healing may have been a factor. We will now present an alternative explanation based on the use of more accurate stress intensity shape factors (Y) devised by Strobl et al. for surface cracks that are not exactly semielliptical in shape. The new Y solutions give better results than the older Newman-Raju solutions. The revised SCF results will be compared to new SEPB results obtained on fragments of the broken SCF test bars.

2:40 PM

(ICACC-S1-025-2017) A New ASTM Test Standard for the Open Hole Tensile Strength of Fiber-Reinforced Ceramic Matrix Composites

S. T. Gonczy*[1]; Y. Katoh[2]; 1. Gateway Materials Technology, USA; 2. Oak Ridge National Lab, USA

Holes, notches, and cut-outs are common features in ceramic components. They are used for thermal management, joining, and fitting. In brittle ceramics these features often act as stress concentration features which significantly reduce the component strength. However, fiber-reinforced ceramic matrix composites (CMC) are commonly much less notch-sensitive, because inelastic deformation typically relieves the stress concentrations. But for component design and qualification purposes the effect of holes on the different CMC material systems must still be physically measured against a wide range of material, geometry and environmental variables. The ASTM C28.07 (Ceramic Composites) subcommittee has developed a new test standard for the open hole tensile strength of fiber reinforced ceramic composites. The test standard is based on the ASTM D5766 Test Standard (PMC Open Hole Tensile Strength -- 6 mm dia.hole in a 36 mm wide, 200mm long tensile bar) and the ASTM C28 C1275 Test Standard (Tensile Strengthof Fiber-Reinforced Advanced Ceramics). The new test standard considers the full range of test factors and procedures – hole size, specimen geometry effects, fiber architecture, interference factors, gripping methods, specimen alignment, test control factors, failure modes and analysis, calculation & interpretation, and high temperature testing.

3:20 PM

(ICACC-S1-026-2017) Notch Variation and Control Mode Effects on Fracture Toughness of Silicon Nitride

B. Huasmann*[1]; J. Salem[2]; 1. Case Western Reserve University, USA;
2. NASA Glenn Research Center, USA

The effects of load rate, machining tolerances, and notch width on the fracture toughness of silicon nitride were determined by using chevron notch test specimens per ASTM C1421. The effects of load rate were investigated in both displacement and strain control modes. Load rate in displacement control did not affect the measured fracture toughness; load rate in strain control exhibited a statistically insignificant trend. Poorly machined notches, in which the notch halves were offset by more than 40% of the notch width (kerf), exhibited increased scatter (about 20%). Notch width for well-machined notches did not exhibit a significant trend, with a scatter of only 6%. The results will be used to confirm the tolerances recommended in ASTM C1421.

3:40 PM

(ICACC-S1-027-2017) Fracture toughness measurement on small ceramic components and specimens

T. Lube*[1]; S. Rasche[4]; S. Strobl[3]; T. Nindhia[2]; 1. Montanuniversität Leoben, Austria; 2. Udayana University, Indonesia; 3. SKF Engineering & Research Centre, Netherlands; 4. MPFA an der Bauhaus-Universität, Germany

State of the art fracture toughness measurements on ceramic materials and components are performed on bar-shaped specimens which are often much bigger than components or the amount of material that is available from lab scale processing units. Suitable specimens thus have to be manufactured separately and often with a different processing route. Their microstructure and therefore their mechanical behavior can vary for this reason. Here, fracture toughness tests are presented, which can be applied to thin disc or plate-shaped specimens (0.5 mm thickness) or small balls. They are based on the ball-on-three-balls (B3B) test or the notched-ball test (NBT), respectively. In analogy to the surface crack in flexure (SCF) method, artificial semi-elliptical surface cracks are introduced into a suitable position of the stress field. Then the specimens are loaded in the way specific for the test. The crack is opened in mode I during loading. After the test, the crack sizes are measured on the fracture surfaces. The evaluation of stress intensity factors is based on finite element calculations. Fit solutions for the fracture stress and the geometric factor calculated for practically relevant ranges of specimens and crack dimensions are provided. The high accuracy of the results is demonstrated using experimental data gained on various materials.

4:00 PM

(ICACC-S1-028-2017) In-Situ Double Torsion Technique on Ultra-High Temperature Ceramics

L. Reyes*[1]; C. Rudolf[1]; P. Nautiyal[1]; B. Boesl[1]; A. Agarwal[1]; 1. Florida International University, USA

In ceramic materials, cracks grow at subcritical levels and usually are referred to as static fatigue. Measuring static fatigue enables life prediction of materials and a fundamental understanding of the origin of failure. The in-situ double torsion technique (inside SEM) provides an alternative to measure crack propagation rate in ceramics. In this work, in-situ double torsion experiments are conducted on ultra-high temperature ceramics such as TaC. This technique accounts for stress intensity factor independent from the crack length allowing for simple and easily adapted specimen loading geometries. Performing this testing inside of a scanning electron microscope (SEM) allows for real-time viewing of slow crack growth using simple load relaxation tests. Fixtures were designed to fit onto a linear, screw driven load frame and were printed out using a 3-D printer to test the validity of the technique. Test samples were prepared by wire EDM off of previously used geometries. A small notch was made in the sample to facilitate the slow crack propagation. From the load versus time curve, crack velocity versus stress intensity factor curves are obtained.

4:20 PM

(ICACC-S1-029-2017) Tensile testing of ceramic balls: A round robin exercise

T. Lube*[1]; O. Schöppl[2]; S. Strobl[3]; R. Morrell[5]; J. Dusza[4]; A. Karakuscu[3]; R. Danzer[1]; 1. Montanuniversitaet Leoben, Austria; 2. SKF Österreich AG, Austria; 3. SKF Engineering & Research Centre, Netherlands; 4. Institute of Materials Research of SAS, Slovakia; 5. National Physical Laboratory, United Kingdom

Ceramic balls are nowadays extensively used in bearings, where their surfaces are heavily loaded. Therefore quality control needs a tensile test for spheres, where high tensile stresses occur in the surface near regions. In conventional tensile testing (e.g. by bending of beams or discs) specimens have to be cut out of the spheres (what is very costly) und high stresses are only applied to material from the inner region of the former spheres. Two methods have been published that provide possible solutions. In the C-Sphere test, much of the ball is machined away leaving a wide notch with a rounded root to create a C-shape. The test-piece is then squeezed at the poles to compress it perpendicular to the notch, creating tensile stresses on the ball outer surface. In the similar notched ball test, a narrower planar notch is machined into the ball, which is a much simpler operation involving the removal of much less material, and the ball is then similarly loaded. Detailed finite element analysis shows that the latter test is robust and readily scalable to balls of a wide range of sizes, from a few mm up to 60 mm. There are recent activities to establish the NBT as an international standard (ISO). A round robin exercise on silicon nitride spheres to test the applicability of the NBT test is carried out by six laboratories in Europe and Japan. First results are communicated in this contribution.

4:40 PM

(ICACC-S1-030-2017) Damage Evolution Behavior of Monolithic SiC and SiC/SiC Composite Hybrid Rod under Compressive Loading

Y. Kagawa*[1]; S. Arai[1]; K. Yonekura[1]; T. Takagi[2]; M. Ukai[3]; M. Uchihashi[3]; S. Suyama[3]; 1. The University of Tokyo, Japan; 2. Ibiden Co. Ltd, Japan;
3. Toshiba Corporation, Japan

Compressive failure behavior of monolithic SiC outer layer and SiC/SiC composite inner layer hybrid-type rod SiC/(SiC/SiC) has been examined. The SiC/(SiC/SiC) rod is ~10 mm in diameter and a total wall thickness of ~800 μm. Under compressive loading, onset of visible fracture behavior starts from failure of the monolithic SiC outer layer with a clear load drop, however, the rod does not completely loose its load bearing capacity because of damage tolerant property of the SiC/SiC composite located as a inner layer of the rod. Failure Compressive damage is localized only near loading edge and the damage does not extend gage section of the specimen. The set of experimental results prove that the hybrid structure of monolithic SiC and SiC/SiC composite is one of effective way to achieve damage tolerant potential of SiC base rod. Discussion is made on micro-failure behavior of the tube.

5:00 PM

(ICACC-S1-031-2017) Study of crack behaviour in layered ceramics with strong interfaces under bending load

L. Nahlik*[1]; K. Stegnerova[1]; P. Hutar[1]; 1. Institute of Physics of Materials Academy of Sciences of the Czech Republic, Czech Republic

The contribution is focused on the description of a specific crack behaviour in layered ceramics. Alumina-zirconia ceramic laminate was used for the study presented. The main aim of the contribution is to clarify crack behaviour in the compressive layer of laminate and provide computational tools for estimation of crack behaviour in the field of strong residual stresses. The crack propagation was investigated on the basis of linear elastic fracture mechanics. Fracture parameters were computed numerically and routines of authors were used for determination of crack behaviour. Finite element models were developed in order to obtain stress distribution in the ceramic laminate containing a crack and to simulate crack propagation. Three different criteria were used for the description of crack behaviour in the field of strong residual stresses. The sharp change of the crack propagation direction was estimated and conditions for crack bifurcation were determined. Estimated crack behaviour is qualitatively in a good agreement with experimental observations. Presented work contributes to the better understanding of the toughening mechanism of ceramic laminates and can be advantageously used for design of new layered ceramic composites and for better prediction of their failure.

5:20 PM

(ICACC-S1-032-2017) Improved methods for in-situ micron scale fracture toughness testing of transparent armor materials

F. Cui*[1]; A. Kundu[1]; R. Vinci[1]; 1. Lehigh University, USA

The usefulness of micron scale in-situ fracture toughness testing has recently been demonstrated using single edge notched microcantilever specimens. Employing such testing methods enables us to analyze the fracture behavior of nanostructures, single grains, single grain boundaries, and doping segregation effects in transparent armor materials like spinel. Bend specimens with a chevron notch can offer the additional advantage of stable crack growth even with brittle materials. However, fabricating and testing an appropriate specimen is challenging at the micron scale. In this study, we developed several fixed-fixed beam designs with a central chevron notch that are capable of achieving stable crack growth during in-situ fracture testing. A Hysitron PI-85 instrument was used to perform a multistep loading and unloading function in order to collect information about compliance change in each loading cycle. A 3D finite element analysis (FEA) was also built to calculate fracture toughness results. Single crystal magnesium aluminate spinel and fused quartz were tested in our study. Both materials have shown a decreasing compliance change, which indicates the stable crack growth.

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

Environmental Barrier Coatings II: Processing and Characterization

Room: St. John
Session Chair: Hagen Klemm, FhG IKTS Dresden

1:30 PM

(ICACC-S2-020-2017) Microstructural Change of Aerosol Deposited Mullite Coating for EBCs under Heat Exposure

A. Iuchi*[1]; T. Mizuno[1]; T. Shibuya[1]; M. Hasegawa[1]; 1. Yokohama National University, Japan

SiC/SiC CMCs with the environmental barrier coatings (EBCs) are one of the candidate for the next generation high temperature structural material. Recent EBCs are composed of silicon, mullite and BSAS layers. Mullite which is processed by conventional air plasma spraying contains metastable amorphous phase due to the rapid cooling of molten mullite during its solidification on a cold substrate. Due to the shrinkage of the coating by sintering and crystallization at high temperature, vertical cracks initiate at EBCs. In order to prevent it, dense ceramic coatings are required from the initial state. Aerosol deposition (AD) method is known to fabricate a dense ceramic coating at room temperature. In this study, mullite which is promising material for EBCs is deposited on the silicon substrate by AD. Microstructure change of mullite coating under heat treatment in a vacuum and exposure in an air is investigated. The mullite coating over a thickness of 10 μm can be fabricated on the silicon substrate by optimizing the formation conditions. XRD analysis indicates the formation of crystalline mullite layer. Oxides in between coating and silicon substrate are not visible in as-deposit state. After heat exposure in air without heat treatment, oxides appear. Heat treatment of the specimen may prevent the oxidation of substrate. However, the decomposition of the mullite layer initiates.

1:50 PM

(ICACC-S2-021-2017) Preparation of Ytterbium Silicate Coatings by Double Electron Beam Physical Vapor Deposition and Their Material Characteristics

T. Yokoi*[1]; N. Yamaguchi[1]; K. Nakahira[1]; S. Kitaoka[1]; M. Takata[1]; 1. Japan Fine Ceramics Center, Japan

Environmental barrier coatings (EBCs) can play a key role in enabling SiC fiber-reinforced SiC matrix (SiC/SiC) composites to be applied as advanced hot-section components in airplane engines, as a means of realizing exceptional fuel efficiency. An EBC must exhibit superior environmental shielding and thermomechanical durability, along with excellent volatilization resistance. Hence EBCs have multilayered structure, namely SiC/SiC/a bond layer/an oxygen shielding layer/a water vapor shielding-volatilization barrier layer/a thermal barrier layer. Yb silicates ($Yb_2Si_2O_7$, Yb_2SiO_5) have excellent volatilization resistance. This property of Yb_2SiO_5 is superior to $Yb_2Si_2O_7$. The thermal expansion coefficient of $Yb_2Si_2O_7$ is close to SiC/SiC, while that of Yb_2SiO_5 is much larger than that. Mullite has higher oxygen barrier properties than Yb silicates. Thus, we designed multilayered EBC including composition-gradient Yb silicate layer (from $Yb_2Si_2O_7$ to Yb_2SiO_5) on mullite oxygen shielding layer. In this study, we investigated coating process of Yb silicates by double electron beam physical vapor deposition. We obtained dense $Yb_2Si_2O_7$ and Yb_2SiO_5 monophasic coatings as well as Yb silicate coating with gradient compositions. Additionally we characterized degree of crystal orientation of these coatings and measured their oxygen permeability constants.

2:10 PM

(ICACC-S2-022-2017) Crack growth tests in air plasma-sprayed yttria coatings for alumina CMCs

S. Hackemann*[1]; M. Hilleringmann[1]; P. Mechnich[1]; M. Bartsch[1];
1. DLR - German Aerospace Center, Germany

Plasma-sprayed yttria coatings revealed to be a favorable choice for alumina based CMC combustor walls for several reasons. The Coefficient of Thermal Expansion of coating and substrate match well, and the resistance of the yttria coating to CMAS corrosion is comparably high to formerly tested materials such as partially stabilized zirconia. The deposition rate of the coating process enabled thicknesses of 1mm and more, which reduced the temperature level of the substrate towards the hot gas side distinctly. Nevertheless, the spraying process incorporates the formation of pores, residual stresses and metastable conditions, which certainly have influence on the crack growth. A rigid bending device, driven by a piezo stack, was designed to characterize the crack-growth in situ. Bending and indentation tests, as well as fracture resistance measurements, were conducted for as-received and aged samples. Furthermore the direction of crack propagation was tested in different directions. The results in stiffness, strength and crack growth resistance show a discernable evolution with respect to the aging conditions. The differing results for different crack propagation orientations reflect well the anisotropic microstructure of the plasma-sprayed material. Different approaches from (eXtended) FEM analysis of the fracture mechanical load parameters and crack growth are discussed for the modelling of the material.

2:30 PM

(ICACC-S2-023-2017) APS YAG Coatings for Al_2O_3/Al_2O_3 Ceramic Matrix Composites

P. Mechnich*[1]; 1. German Aerospace Center (DLR), Germany

Al_2O_3/Al_2O_3 ceramic matrix composites (CMC) are candidate lightweight, damage-tolerant hot-gas leading components of gas turbines. The susceptibility of Al_2O_3/Al_2O_3 CMC to chemical degradation in combustion environments at high temperatures, however, requires the development of protective coatings. Owing to its thermodynamic compatibility and low CTE mismatch to Al_2O_3, $Y_3Al_5O_{12}$ (YAG) is a promising material for such thermal and environmental barrier coatings (T/EBC). YAG coatings were deposited by means of air plasma spraying (APS) on Al_2O_3/Al_2O_3 CMC with a reaction-bonded Al_2O_3 bondcoat (RBAO). APS YAG coatings are mostly amorphous in the as-deposited state and exhibit a good adherence. Above 900°C amorphous APS coatings crystallize instantaneously to YAG without formation of intermediate metastable phases. Crystallization is accompanied by an anisotropic shrinkage and increase of thermal conductivity. APS YAG coatings exhibit a characteristic segmentation crack pattern which is considered beneficial with respect to coating strain tolerance and thermal shock resistance. The durability of APS YAG coatings in harsh, H_2O-rich combustion environments could be demonstrated in a high-velocity burner-rig. However, APS YAG coatings are prone to infiltration by molten CMAS-type deposits which may limit their application in aeroengines.

CMAS-related TBC/EBC Degradation and Mitigation Strategies I

Room: St. John
Session Chair: Douglas Wolfe, Pennsylvania State University

3:10 PM

(ICACC-S2-024-2017) Microstructural investigation of molten sand infiltration and chemical interactions with thermal barrier coatings protecting hot-section gas turbine engines components

M. J. Walock*[1]; B. Barnett[2]; A. Ghoshal[1]; M. Murugan[1]; J. Swab[2]; M. S. Pepi[2]; D. Zhu[3]; R. T. Pegg[5]; C. Rowe[5]; K. Kerner[4]; 1. US Army Research Laboratory, USA; 2. US Army Research Laboratory, USA; 3. NASA Glenn Research Center, USA; 4. US Army Aviation and Missile Research Development and Engineering Center, USA; 5. US Navy Naval Air Systems Command, USA

Advanced gas turbines are used in commercial/military aviation, ship propulsion, and industrial power generation. However, degraded environments that contain sand, dust, ash, soot, and salt can significantly affect their durability and performance. Contaminant accumulation can block cooling holes and reduce air flow through the engine; sand-glazing increases the thermal conductivity and reduces the strain-tolerance of thermal barrier coatings (TBCs). In addition, chemical attack by calcia-magnesia-alumina-silicate (CMAS), formed by the molten sand, can attack and degrade/destroy TBCs. Using atmospheric plasma spray (APS), solution-precursor plasma spray (SPPS), and electron-beam physical vapor deposition (EB-PVD), standard and unique TBC compositions have been deposited onto 1-in diameter Inconel 718 and Rene N5 discs. After exposure to molten sand in a hot-particulate ingestion rig (HPIR), under engine relevant conditions, the discs are cross-sectioned and examined using scanning electron microscopy (SEM), energy-dispersive x-ray spectroscopy (EDS), and scanning Auger electron microscopy (SAEM). This study investigates the microstructural changes and chemical interactions due to molten sand adherence, infiltration, and glassification on TBCs for hot-section components within gas turbine engines.

3:30 PM

(ICACC-S2-025-2017) Thermal cycling performance of thermal barrier coatings with CMAS corrosion: Parameters affecting life

A. Harris*[1]; E. H. Jordan[2]; 1. University of Connecticut, USA; 2. University of Connecticut, USA

As thermal barrier coatings (TBCs) become prime-reliant components in gas turbine engines, understanding factors that drastically reduce coating life becomes critical. Corrosion by glassy deposits of ingested debris, composed primarily of oxides of Calcium, Magnesium, Aluminum, and Silicon (CMAS), is one such factor. While chemical and mechanical interactions between TBCs and CMAS are well characterized, there has been little effort to model the decrease in TBC cyclic life due to CMAS attack. Life debit models will depend on geographic and environmental factors, such as composition and concentration of CMAS, as well as engine operation conditions, such as temperature. Using a custom-built thermal gradient test rig, TBC samples were subjected to thermal cycling with simultaneous CMAS application. Cycling parameters were varied to study their effects on TBC life. The mechanisms behind these effects were studied using material characterization techniques.

3:50 PM

(ICACC-S2-026-2017) CMAS Interactions with Advanced Environmental Barrier Coatings Deposited via Plasma Spray-Physical Vapor Deposition

B. J. Harder*[1]; V. L. Wiesner[1]; D. Zhu[1]; N. S. Johnson[2]; 1. NASA Glenn Research Center, USA; 2. Colorado School of Mines, USA

Materials for advanced turbine engines are expected to have temperature capabilities in the range of 1370-1500°C. At these temperatures the ingestion of sand and dust particulate can result in the formation of corrosive glass deposits referred to as CMAS.

The presence of this glass can both thermomechanically and thermochemically significantly degrade protective coatings on metallic and ceramic components. Plasma Spray- Physical Vapor Deposition (PS-PVD) was used to deposit advanced environmental barrier coating (EBC) systems for investigation on their interaction with CMAS compositions. Coatings were exposed to CMAS and furnace tested in air from 1 to 50 hours at temperatures ranging from 1200-1500°C. Coating composition and crystal structure were tracked with X-ray diffraction and microstructure with electron microscopy.

4:10 PM

(ICACC-S2-027-2017) Effect of Biofuel Impurities on the Hot Corrosion of Yttria-Stabilized Zirconia Thermal Barrier Coatings

J. H. Ramirez Velasco*[1]; H. Kenttämaa[2]; G. Kilaz[3]; R. Trice[1]; 1. Purdue University, USA; 2. Purdue University, USA; 3. Purdue University, USA

The contaminants found in various biofuels are known to include oxides of alkali and alkaline earth metals including calcium, magnesium, sodium, potassium, along with sulfur, phosphorus and silicon oxides. Furthermore, while calcium-magnesium-aluminum silicates (CMAS) are typically ingested as particulate in middle-east theaters, the impurity list in biofuels includes the necessary elements to form CMAS without exposure to any specific environment. This is very significant as CMAS is particularly destructive for operating temperatures above its melting temperature (~1250°C), particularly affecting thermal barrier coatings (TBCs) lifetimes. In the work reported currently, solutions containing the constituents of CMAS are prepared, and sprayed onto TBCs, simulating the deposit of the impurities during combustion. The TBCs are then subject to static and dynamic heating up to 1300°C in a furnace and ablation rig, respectively, and the microstructures evaluated.

4:30 PM

(ICACC-S2-028-2017) High Temperature Interactions between Environmental Barrier Coating Ceramics and Calcium-magnesium-alumino-silicate (CMAS) Glass

L. R. Turcer*[1]; A. R. Krause[1]; H. Garces[1]; N. P. Padture[1]; 1. Brown University, USA

Ceramic-matrix-composites (CMCs) are being considered for replacing current metallic hot-section components, which would allow for higher engine-operating temperatures. To prevent active-oxidation of CMCs in the presence of water vapor, 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-alumino-silicate (CMAS) glass deposits. The molten CMAS glass can penetrate grain boundaries and cause dissolution, which leads to premature failure. New EBC ceramics are needed that are resistant to CMAS attack. A new model has been used to predict the reactivity between CMAS and potential EBC ceramics. Based on this model, several potential EBC ceramics are identified, and their high-temperature interactions with CMAS are studied. The nature of these interactions, damage mechanisms, and their mitigation are elucidated.

4:50 PM

(ICACC-S2-029-2017) Kinetics of Thermal Barrier Oxide Interactions with Molten Silicates

C. S. Holgate*[1]; D. L. Poerschke[1]; C. G. Levi[1]; 1. University of California, Santa Barbara, USA

The degradation of thermal barrier coatings (TBCs) by molten silicates (CMAS) represents a significant barrier to increased operation temperatures of gas turbine engines and thus fuel efficiency. Research toward a CMAS resistant TBC has focused on developing thermodynamic, thermomechanical, and infiltration models to predict TBC behavior under relevant conditions. However, additional quantitative information about the kinetics of TBC-CMAS interactions is needed to refine the models. Specifically, there is insufficient information about (i) the rate of TBC dissolution into the melt, (ii) diffusion of the TBC constituents in the melt, and (iii) the reaction product crystallization rates. Each process is influenced by temperature and the composition of both the coating and the molten deposit. In the present work, single and polycrystalline thermal barrier oxides were placed in a 1-D diffusive contact with model silicate deposits to study the effect of time, temperature, and composition on the reaction product morphology and the cation diffusivity into the melt. Furthermore, the results yield insight into the role of grain boundaries on the reaction dynamics. The resulting kinetic data supports models that predict the in-service behavior and will accelerate the development of new TBC oxides resistant to CMAS degradation.

S3: 14th International Symposium on Solid Oxide Fuel Cells (SOFC): Materials, Science and Technology

Reaction Kinetics and Modeling

Room: Crystal
Session Chair: Harumi Yokokawa, The University of Tokyo

1:30 PM

(ICACC-S3-017-2017) From In-Situ to In-Operando Evaluation of SOFC Cathodes for Enhanced ORR Activity and Durability (Invited)

E. Wachsman*[1]; G. Cohn[1]; Y. Huang[1]; C. Pellegrinelli[1]; 1. University of Maryland, USA

To study the SOFC cathode oxygen reduction reaction (ORR) mechanism and the effects of ambient gas composition on ORR, we used in-situ ^{18}O isotope exchange techniques to probe the exchange of H_2O and CO_2 with two of the most common SOFC cathode materials, $(La_{0.8}Sr_{0.2})_{0.95}MnO_{3-x}$ (LSM) and $La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_{3-x}$ (LSCF). A series of temperature programmed isotope exchange measurements were performed to comprehensively study the interaction of H_2O and CO_2 with the cathode surface as a function of temperature, oxygen partial pressure, and contaminant gas concentration. The experimental data are summarized in a Temperature-P_{O2} diagram to visualize the dominant reactions at each temperature and P_{O2} for the two cathode materials. We further report on the development of a novel in-operando ^{18}O isotope exchange technique by modifying our heterogeneous catalysis system for operation and measurement under electrochemical polarization. The system allows ORR investigations under real time, real life, operating conditions. We will present our studies on the effect of cathodic polarization on LSM and LSCF. The studies show the effects of operating voltage, temperature, and oxygen partial pressure on the oxygen exchange coefficient. We will also present our model for the understanding and analysis of the isotope oxygen exchange profiles under in-operando conditions.

2:00 PM

(ICACC-S3-018-2017) On the Link Between Oxygen Surface Exchange and Bulk Oxygen Anion Transport in SOFC Cathode Materials (Invited)

A. Tomkiewicz[1]; M. Tamimi[1]; A. Huq[2]; S. McIntosh*[1]; 1. Lehigh University, USA; 2. Oak Ridge National Lab, USA

A link between the surface oxygen exchange rate and bulk ion diffusivity of mixed ionic-electronic conductors has been hypothesized in the literature. However, most of data available in the literature is derived from experiments that simultaneously measure both properties. This stems from the complexity of measuring these properties under realistic conditions. We have recently focused on implementing new experimental approaches to independently characterize these properties for SOFC cathode materials. The bulk ionic transport is characterized by in-situ powder neutron diffraction; a powerful technique that enables the simultaneous determination of the position, occupancy, and anisotropic average displacement of the atoms in the unit cell. In contrast to X-ray diffraction, neutron diffraction is sensitive to the oxygen within the material, allowing identification of ionic transport pathways via analysis of site occupancy and average displacement magnitude and anisotropy of the various oxygen sites within these materials. The surface oxygen exchange is measured by pulsed isotopic exchange and complemented by surface composition analysis via high sensitivity-low energy ion scattering (HS-LEIS) and X-ray photoelectron spectroscopy (XPS). This data is then utilized to interpret the electrochemical performance, in terms of the impedance spectra, of the materials as SOFC electrodes.

2:30 PM

(ICACC-S3-019-2017) Determination of Infiltrated Mixed Ionic and Electronic Conducting Nano-Particle Oxygen Surface Exchange Resistance through Finite Element Modeling of 3D Reconstructed Microstructures

T. E. Burye[1]; J. Nicholas*[1]; 1. Michigan State University, USA

Surface-area-normalized oxygen reduction resistance (Rs) measurements of Solid Oxide Fuel Cell (SOFC) Mixed Ionic Electronic Conductor (MIEC) materials are used as a MIEC performance metric and an input for SOFC electrode microstructural optimization modeling. Unfortunately, literature-reported Rs values for nominally identical materials often vary by 2-5 orders of magnitude. Further, the Rs values of infiltrated MIEC materials (which might be expected to be different than bulk materials or thin films due to a high surface curvature, electrochemical spill over onto the supporting material, etc.) are not available in the literature. This talk describes how the Rs values of commonly used MIEC infiltrate materials were determined for the first time using electrode polarization resistance measurements and finite element modeling (FEM) of 3D Focused Ion Beam Scanning Electron Microscopy (FIB-SEM) reconstructions. A comparison of infiltrate, bulk, and thin film Rs values for various MIEC compositions is also presented.

3:10 PM

(ICACC-S3-020-2017) Roles of Oxygen Lattice Defects on the Oxygen Reduction Reaction Kinetics in Solid Oxide Fuel Cell Cathodes (Invited)

X. Liu*[1]; 1. West Virginia University, USA

We report our research on the roles of oxygen defects on ORR kinetics of SOFC cathode. For perovskites, we developed a multi-domain 1-D physical model incorporating multi-step charge transfer to examine the competitive behaviors between the paralleled 3PB and 2PB kinetic pathways. Analyses identified the limitation of surface oxygen ion diffusion as the mechanism for 3PB-to-2PB transition. The model also proved surface reactions are driven predominantly by electrochemical forces at the 3PB, while being controlled by oxygen vacancy concentration variation at regions away from 3PB. For Ruddlesden–Popper (R-P) phases, the governing factors of the ORR are identified as oxygen adsorption and incorporation. The incorporation rate is drastically dependent on the amount of interstitial oxygen. Since the unfilled interstitial sites serve to accommodate the adsorbed oxygen during incorporation, more oxygen interstitials would seem to suppress the kinetics of this process. We proposed a physical model to reconcile the discrepancy between the experimental results and intuitive reasoning. This model illustrates a possibility of how oxygen interstitials works to regulate the exchange rate, and how the contradiction between oxygen vacancies and oxygen interstitials is harmonized so that the latter in the R–P structure also positively promotes the incorporation rate in the ORR.

Air Electrode

Room: Crystal
Session Chair: Eric Wachsman, University of Maryland

3:40 PM

(ICACC-S3-021-2017) Surface Reaction of Cathode Materials for Solid Oxide Fuel Cells (Invited)

K. Yashiro*[1]; H. Chiba[1]; H. Sato[1]; R. Budiman[1]; S. Hashimoto[1]; T. Kawada[1]; 1. Tohoku University, Japan

Cost reduction and high durability of solid oxide fuel cells (SOFCs) are required to widen application fields. Therefore, lower operation temperature is preferable, while an electrode performance becomes worse. Especially cathode performance should be improved for low temperature SOFC. (La, Sr)CoO_3 (LSC) and (La, Sr)(Co, Fe)O_3 (LSCF) have attracted attention as a SOFC cathode, which show both high ionic- and electronic conductivities. We reported that implementation of hetero-interface is one of possible solutions to improve the surface reaction kinetics of LSC. It is also known that LSCF-GDC composite cathode shows higher performance than LSCF. However, the mechanism of improvement is still not clear. In this paper, several evaluation techniques are applied to obtain the comprehensive understandings of surface reaction kinetics of MIEC cathodes.

4:10 PM

(ICACC-S3-022-2017) Tailoring the Nanostructure of LSM to Achieve High Power Output for SOFCs Operating at 600°C (Invited)

E. Traversa*[1]; 1. Xi'an Jiaotong University, China

Essential for the development of low temperature solid oxide fuel cells (SOFCs) is the role of the cathode material and its microstructure. This is due to the fact that since oxygen reduction reaction kinetics is sluggish due to thermally activated processes, polarization losses increase significantly with reducing the temperature. In fact, $La_{0.8}Sr_{0.2}MnO_{3-\delta}$ (LSM), which is the standard cathode material for high-temperature SOFCs, cannot be used at low temperatures because of its low oxygen-ion diffusivity and conductivity at 600°C. Strategies to improve the performance of cathode materials include the deployment of better ionic conducting materials and the increase in triple phase boundary length using nanostructured materials. However, the chemical stability of the best performing low temperature cathode materials is not comparable with the stability of LSM. We report here that nanostructuring LSM using inkjet impregnation we were able to obtain large power output both using proton conducting and oxygen-ion conducting electrolytes.

4:40 PM

(ICACC-S3-023-2017) A-site Non-stoichiometry Lanthanum Strontium Cobalt Ferrite as Cathode Material for Intermediate Temperature Solid-Oxide Fuel Cell

L. Ge*[1]; B. Ingram[1]; D. Carter[1]; 1. Argonne National Lab, USA

Lanthanum strontium cobalt ferrite (LSCF) perovskite has been identified as promising cathode candidate for intermediate temperature (IT) solid oxide fuel cell (SOFC) applications, due to its mixed ionic and electronic conductivity at operating temperatures (500-700°C). In particular, A-site deficient LSCF has shown better electrochemical performance compared to its stoichiometry counterpart. However, non-uniformity in electrical, electrochemical, and morphological characteristics found in different powders used for fabricating cell and stack components has been a continuing challenge for SOFC manufacturers. In this study, a wide variety of physical and chemical characterizations on $(La_{0.6}Sr_{0.4})_{0.95}Co_{0.2}Fe_{0.8}O_{3-d}$ powders were performed to investigate the correlation between the electrochemical performance and materials properties. This effort will help in defining tolerances of key materials properties for optimal cell performance.

5:00 PM

(ICACC-S3-024-2017) Electrochemical Properties and Oxygen Exchange Kinetics of Doped $SrCoO_{3-\delta}$ Cathode Materials

G. Cohn*[1]; J. Wang[2]; C. Pellegrinelli[1]; K. Huang[2]; E. Wachsman[1];
1. University of Maryland, USA; 2. University of South Carolina, USA

Perovskites-type oxides are of considerable technological interest for numerous application including solid oxide fuel cells and solid oxide electrolysis cells. Among the perovskite-type solid solutions, doped $SrCoO_{3-\delta}$ is one of the most promising cathode materials for intermediate temperature due to its high oxide ion conductivity and high catalytic activity for oxygen reduction, which is governed by oxygen surface exchange properties. Here we report on the electrochemical and oxygen surface exchange properties of several Nb, Fe Y and Ta doped $SrCoO_{3-\delta}$, by means of in-situ oxygen isotope exchange. We used our unique heterogeneous catalysis system to determine the oxygen exchange rate coefficients and mechanism for a variety of dopants and doping concentrations. Exchange of $^{18}O_2$ reaches a saturation at 450-500°C, while below 300°C the exchange is very slow. Fitting the exchange results to a model describing the exchange process we were able to extract the temperature dependence of the exchange coefficient as well as the activation energy for oxygen exchange and compared with the literature data. Reaction order, together with the identity of the oxygen species taking part in the reaction are also investigated through variation in the oxygen partial pressure.

5:20 PM

(ICACC-S3-025-2017) Impedance Characterization of Polarization-Induced Activation and Degradation in LSM Based Cathodes

J. Liu*[1]; H. Finklea[1]; M. Yan[1]; H. Abernathy[1]; P. Ohodnicki[1]; G. Hackett[1];
1. National Energy Technology Laboratory, USA

$La_{1-x}Sr_xMnO_3$ perovskite (LSM) is one of the most investigated materials for cathodes in SOFCs because of its high thermal and chemical stability. The activation effect of the cathodic polarization on the activity of LSM/YSZ composite cathodes is of great practical relevance as the SOFC performance. This work demonstrates the application of a two electrode configuration half-cell operated for the impedance analysis of a LSM electrode under load. The counter electrode is LSCF deposited on the YSZ electrolyte by spray pyrolysis. The LSCF counter electrode exhibits a small polarization resistance, typically less than 0.1 Ohm cm^2 at 800°C in air, much smaller than the LSM electrode. The effects of cathodic current density and overpotentials on the polarization behavior of LSM thin film electrode and LSM/YSZ composite cathodes were investigated. The activation response times of the LSM based electrodes are strongly correlated with bias conditions. The reduction of polarization resistance can be over 90%. The pre- and post-activation electrode performances were evaluated at various temperatures and oxygen partial pressures to reveal the nature of the activation mechanism. A threshold polarization condition of reversible and irreversible activation processes was identified and the correlation with activity degradation was also investigated.

5:40 PM

(ICACC-S3-026-2017) The crucial role of cation order in $LnBaCo_2O_{5+\delta}$ perovskite materials used as SOFC cathode

N. Zhang*[1]; 1. West Virginia University, USA

In this work, disordered and ordered $LnBaCo_2O_{5+\delta}$ perovskite powders were synthesized through controlling of the sintering atmosphere and temperature. These powders were characterized by XRD (X-ray diffraction pattern) and SEM (Scanning Electron Microscopy) to discern the phase difference and morphology similarities. Raman Spectroscopy was also adopted to characterize the vibrational state difference between the disordered and ordered powders. The bulk diffusion coefficient D and surface exchange coefficient k values were obtained by ECR (Electrical Conductivity Relaxation) test toward dense $LnBaCo_2O_{5+\delta}$ pellets. EIS (Electrochemical Impedance Spectroscopy) test toward $LnBaCo_2O_{5+\delta}$/GDC/$LnBaCo_2O_{5+\delta}$ symmetric cells were analyzed by CNLS (complex nonlinear least square) fitting. The results show that the ordered cathodes are superior to that of disordered one by 2-3 times reflected in polarization resistance. This significant difference is caused by the increase in local symmetry induced by the oxygen hypo-stoichiometry, the shorten of Ln-O bond length, and the charge and orbital ordering.

S4: Armor Ceramics

Materials Characterization and Mechanical Behavior II

Room: Coquina Salon E
Session Chair: Jerry LaSalvia, U.S. Army Research Laboratory

1:30 PM

(ICACC-S4-027-2017) A Modified Dynamic Expanding Cavity Model for High Pressure and High Strain Rate Response of Ceramics

S. Bavdekar*[1]; G. Parsard[1]; G. Subhash[1]; 1. University of Florida, USA

A modified dynamic spherical expanding cavity model has been proposed to capture the dynamic response of brittle ceramics under constant velocity penetration at low and high confinement pressures and strain rates. The brittle material is assumed to crack when the hoop stress reaches its tensile strength and is subsequently comminuted when the radial stress reaches its compressive strength. The constitutive behavior of the comminuted material is predicted using a modified form of the extended Mohr-Coulomb model, which utilizes an exponential pressure dependence on the shear strength. This model is more accurate in capturing the non-linear response of the material at pressures above the HEL as compared to the conventional Mohr-Coulomb model. The extended model also inherently captures the shear saturation phenomenon observed in comminuted ceramics at shock pressures above the HEL, without the need for experimental data. The new expanding cavity model is further extended to capture the effects of amorphization observed in boron carbide subjected to shock loading and high pressures.

1:50 PM

(ICACC-S4-019-2017) Dynamic Failure of Damaged Advanced Ceramics

C. Lo[1]; E. Krimsky[2]; T. R. Walter[3]; T. Sano[3]; B. Schuster[3]; K. T. Ramesh[2];
J. Hogan*[1]; 1. University of Alberta, Edmonton, Canada; 2. Johns Hopkins University, USA; 3. US Army Research Laboratory, USA

An understanding of the dynamic failure of damaged ceramics is important in protection applications, where the interaction of the projectile with cracked material and granular materials have been shown to be important governing material performance. In this presentation, we will discuss research conducted on the dynamic compressive behavior of pre-cracked advanced ceramics. Pre-cracks are introduced in the material through thermal shocking, and X-ray computed tomography coupled with an in-house Matlab-based code was developed to analyze internal crack length, orientation, and spatial distribution. This information is used to reconcile experimental and numerical definitions of damage, and these results are linked with measurements of failure, strength, and fragmentation. It is hoped that the insight gained in this study can inform computational models and the design of improved security materials.

2:10 PM

(ICACC-S4-020-2017) Compression Strength of Armor Ceramics

J. Swab*[1]; C. Meredith[1]; W. Gamble[1]; 1. Army Research Laboratory, USA

The compression strength of an armor ceramic influences its ballistic performance, but determining the intrinsic compression strength is very difficult. 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 ballistic performance. Thus it is imperative that the compressive strength of candidate armor ceramics be properly and accurately measured. Several candidate 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.

2:30 PM

(ICACC-S4-021-2017) TEM Characterization of Deformation Mechanisms in Several Commercial Silicon Carbide Variants Subjected to Knoop Indentation

S. D. Walck*[1]; J. LaSalvia[1]; S. G. Hirsch[1]; K. D. Behler[1]; 1. Army Research Laboratory, USA

Understanding the deformation and damage mechanisms under compression in ceramic materials is crucial for developing improved ceramic materials for lightweight body and vehicle armor systems. To achieve insight into the mechanistic response of polycrystalline armor ceramics to large contact stresses, transmission electron microscopy (TEM) was used to examine the cross-sections of the inelastically deformed regions beneath 1 kgf Knoop indents. Because of extensive cracking, a multi-step sample preparation technique was developed to preserve the cross sections intact. The steps included the use of a masked ion milling system, scanning electron microscopy (SEM), epoxy vacuum infiltration, and the focused-ion beam (FIB) milling. Results for pressureless-, pressure-assisted, and liquid-phase sintered silicon carbide variants will be shown. In general, TEM characterization of the inelastically deformed regions revealed stacking faults, dislocations, micro-cracking, and macro-cracking. The results depended strongly on the initial microstructure and processing of the material. Experimental procedures, results, and analyses will be presented.

2:50 PM

(ICACC-S4-022-2017) Investigation of Indentation-Induced Amorphization in $B_{4+x}C$ (x = 0.2… 4.8)

V. Domnich*[1]; M. Schaefer[1]; K. Kuwelkar[1]; R. A. Haber[1]; 1. Rutgers University, USA

The extent of indentation-induced amorphization and the temperature stability of the amorphous phase were investigated in boron carbides with varying B:C ratios. The samples spanning the carbon concentration range of 10.2 at.% ($B_{8.8}C$) to 19.1 at.% ($B_{4.2}C$) were prepared by hot pressing the powder mixtures of boron carbide and amorphous boron. Arrays of Berkovich nanoindents (50 – 500 mN) were placed on the polished sample surfaces and Raman spectroscopy was used to assess the degree of amorphization inside each individual indent. Further, the nanoindents were heat treated from room temperature to 1000°C under vacuum and in inert gas atmosphere and in situ Raman spectroscopy was used to monitor the disappearance of the signature of the amorphous phase. The findings are critically examined against the available theoretical predictions for the amorphization of boron-rich boron carbide.

Materials Characterization and Mechanical Behavior III

Room: Coquina Salon E
Session Chair: Steven Kilczewski, U. S. Army Research Laboratory

3:30 PM

(ICACC-S4-023-2017) In situ visualization of crack propagation in Boron Carbide (Invited)

A. F. Leong*[2]; A. K. Robinson[2]; K. Fezza[3]; T. Sun[3]; B. Schuster[4]; D. Casem[4]; P. K. Lambert[1]; T. C. Hufnagel[1]; 1. Johns Hopkins University, USA; 2. Johns Hopkins University, USA; 3. Argonne National Lab, USA; 4. US Army Research Laboratory, USA

Boron carbide (BC) has properties that make it suitable for armor against low-velocity threats. Current materials suffer a dramatic loss of performance against high-velocity threats, but although there is the potential for significant improvement, processing BC to high densities can produce unwanted defects and inhomogeneities that become sites for crack nucleation. A detailed understanding of the behavior of these cracks becomes important in optimizing BC for use in military and industrial related applications. At the Advanced Photon Source (Argonne National Laboratory), we employed propagation-based phase contrast x-ray imaging (PCI) of hot-pressed BC undergoing dynamic loading using a three-point bend Kolsky bar apparatus to induce fracturing. PCI enables the nucleation and propagation of cracks within the sample to be directly visualized by rendering the difference in refractive indices between air and BC as intensity variations; that is, it enhances the boundaries of the cracks. We will present our findings that includes interpretation of the sequence of images of BC fracturing in real-time. While PCI increases the contrast of cracks, one of its other desirable properties is it encodes information such as the density and size of cracks. We will discuss our approach to decoding this information, which will be useful as inputs for and validation of fracture models.

4:00 PM

(ICACC-S4-024-2017) Which One Has More Influence on Fracture Strength of Ceramics: Pressure or Strain Rate?

G. Subhash*[1]; 1. University of Florida, USA

Data for a range of structural ceramics subjected to confined uniaxial stress at arrange of strain rates, uniaxial strain, and shock loading experiments are compiled from a range sources and analyzed to identify the relative influences of pressure and strain rate. Normalized shear stress versus hydrostatic pressure plot for several structural ceramics revealed that despite the differences in material properties, test methods, and strain rates, the normalized failure strength data fall in a narrow range. The overall deformation behavior of structural ceramics and their strain-rate dependence of strength in multiaxial loading at high pressure can be represented by a unified exponential pressure dependence in the Mohr-Coulomb model. Further analysis of intact strength data based on Johnson-Holmquist (JH-2) model constants for these ceramics revealed that pressure alone plays a deterministic role in defining the constitutive response of all brittle ceramics.

4:20 PM

(ICACC-S4-025-2017) Static and Dynamic Compression and Hardness response of 3D-printed Alumina

M. Banks*[1]; M. DeVries[1]; G. Subhash[1]; 1. University of Florida, USA

With the recent development of 3D printing as a means of manufacturing alumina it is of interest to investigate the material and mechanical properties of the alumina produced, in comparison to ceramics produced in more traditional manufacturing techniques. While traditionally-manufactured alumina has been extensively characterized in the literature, there is little to no information available for 3D printed alumina due to the novelty of the production process. The properties of interest for this investigation are quasi-static and dynamic Vickers hardness, compressive strength, and fracture toughness. Furthermore, as evidenced in 3D-printed plastics, directionalities resulting from the printing process can play impactful roles, and as such the potential role of directionality in the 3D printed ceramics is investigated. By producing test specimens in various orientations, these possible directionality effects on mechanical behavior are investigated. Micro-Raman spectroscopy is then utilized to investigate the potential development of residual stresses in the microstructure as a result of the manufacturing techniques.

4:40 PM

(ICACC-S4-026-2017) Failure of Granular Boron Carbide under Extreme Loading

M. Serge[1]; M. Homel[2]; J. Loiseau[3]; T. R. Walter[4]; P. Motamedi[1]; C. Lo[1]; E. B. Herbold[2]; A. J. Higgins[3]; B. Schuster[4]; T. Sano[4]; J. Hogan*[1]; 1. University of Alberta, Edmonton, Canada; 2. Lawrence Livermore National Laboratory, USA; 3. McGill University, Canada; 4. US Army Research Laboratory, USA

Understanding the failure, fragmentation, and subsequent behavior of granular materials is important in ballistic applications. To date, considerable attention has been given to intact forms, with less attention given to granular behavior. In this presentation, we investigate the dynamic behavior of granular boron carbide. Powdered boron carbide was first sieved between 125 and 150 μm, then tested via the thick-walled cylinder (TWC) technique using a tunable explosive in the form of a gelled nitromethane mixture. This method is well-described in literature and serves as a benchmark experiment in the study of material response and failure under simultaneous high shear and pressure conditions. Here, an attempt was made to establish domains of granular response and damage across two distinct loading conditions (global strains). Recovered samples were analyzed and characterized via optical and scanning electron microscopy. X-ray computed tomography imaging was used in conjunction with steel tracer particles mixed in with the boron carbide powder to capture average particle motion over the course of the experiment, which were then compared to detailed mesoscale simulations. Post-mortem characterizations of the boron carbide powder were compared to simulations and experimental geometries to determine dominant granular failure mechanisms in this loading regime.

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

Thermal Energy Conversion and Energy Storage V

Room: Tomoka A
Session Chair: Claude Delmas, ICMCB

1:30 PM

(ICACC-S6-016-2017) Understanding the Effect of Structure and Chemistry on Alkali Conductivity (Invited)

S. Ong*[1]; 1. University of California, San Diego, USA

The facile conduction of alkali ions is the fundamental mechanism underpinning the operation of rechargeable alkali-ion batteries. Poor alkali transport in any part of a battery – the electrode, the electrolyte or the interfaces between them – leads to reductions in rate capability, practical capacity and cyclability. In particular, conductivity requirements are even more stringent in cutting edge all-solid-state batteries utilizing superionic conductor solid electrolytes due to the extended solid-state diffusion length scales both in the bulk as well as the grain boundaries. In this talk, I will provide an overview into the fundamental insights that we have gained into the effect of structure and chemistry on alkali conduction in solid-state bulk materials and interfaces from first principles calculations. I will discuss universal relationships between various topological descriptors with alkali migration, as well as how chemistry modification may be used to enhance both bulk and interfacial conductivity.

2:00 PM

(ICACC-S6-017-2017) First-principles study of superconcentrated electrolytes for electrochemically stable and fast-charging lithium-ion batteries (Invited)

K. Sodeyama*[1]; 1. Japan Science and Technology Agency (JST), Japan

Li-salt concentration has been recently proposed as an important control parameter of reduction stability of electrolytes and high ion conductivity in Lithium-ion batteries. For example, highly concentrated (HC) $LiN(SO_2CF_3)_2$ (Li-TFSA) salt in acetonitrile (AN) electrolyte shows strong electrochemical stability against the reductive decomposition, though in low concentration (LC) solution AN is easily decomposed. However, the atomistic origin of the improved reduction stability and high ion conductivity has been still an open question. In this study, we investigated the mechanism of the improvement of the reduction stability and Li-ion diffusion mechanism depending on the salt concentration by using first-principles density functional theory (DFT) molecular dynamics (MD) calculations. For the reduction stability, we found that TFSA anion sacrificially accepts reductive electron and decomposed in the HC systems, because specific chained network structure is formed and the electron affinity of the anion shifts lower. For the diffusion mechanism, we confirmed that in HC system the Li-ions are coordinated both by solvents and by anions arranged in the network structure, which results in high viscosity. We found Li-ion hopping between the oxygen atoms of the anions and concluded that the hopping can be an origin of the high Li-ion conductivity in the HC system.

2:30 PM

(ICACC-S6-018-2017) Superconcentrated electrolytes for lithium batteries (Invited)

Y. Yamada*[1]; A. Yamada[1]; 1. The University of Tokyo, Japan

An ever increasing demand for better batteries (with high voltage, high capacity, and high safety) has set extraordinarily high standards for electrolyte materials, which are far beyond the realm of conventional nonaqueous electrolyte design based on ca. 1 mol dm^{-3} $LiPF_6$ and ethylene carbonate (EC). Generally, further increasing salt concentration over the conventional 1 mol dm^{-3} standard results in decreased ionic conductivity and increased viscosity, both of which are unfavorable for battery electrolytes in terms of rate performance. Recently, however, various unusual functionalities were discovered at superhigh concentrations (over 3 mol dm^{-3}), and superconcentrated (or highly concentrated) solutions are emerging as a new class of liquid electrolytes for advanced lithium batteries. In this invited talk, I will present our original works on unique features of superconcentrated electrolytes, which are not shared by conventional dilute electrolyte solutions, from the viewpoint of their peculiar ion-solvent coordination structure, and discuss their future contributions to advanced battery technologies.

Thermal Energy Conversion and Energy Storage VI

Room: Tomoka A
Session Chair: Yuki Yamada, The University of Tokyo

3:20 PM

(ICACC-S6-019-2017) Defect chemistry of lithium ion conducting oxides: $Li_{3x}La_{2/3-x}TiO_3$ and $Li_7La_3Zr_2O_{12}$ (Invited)

X. Guo*[1]; 1. Huazhong University of Science and Technology, China

Solid state lithium ion conductors are favorable due to the high safety performance and electrochemical stability; however, they also suffer from the relatively low lithium ion conductivity. In most cases, solid state lithium ion conductors are present in the form of polycrystals; therefore, grain bulk and grain boundaries are the two crucial parts of the microstructure. In this presentation, we evaluate the lithium ion conductivity of $Li_{3x}La_{2/3-x}TiO_3$ (LLTO) with perovskite structure and $Li_7La_3Zr_2O_{12}$ (LLZO) with garnet structure, with the ionic conductivities of the grain bulk and grain boundaries being addressed from the point of view of defect chemistry. We try to give answers to the following questions: How to improve the lithium ion conductivity? How to suppress the electronic conductivity? Does the oxygen ion conductivity play any role? What is the physical origin of the low grain boundary conductivity?

3:50 PM

(ICACC-S6-020-2017) Rechargeable solid state lithium batteries based on garnet ceramic electrolytes (Invited)

X. Guo*[1]; 1. Shanghai Institute of Ceramics, Chinese Academy of Sciences, China

Solid state lithium batteries (SSLB) offer higher energy density, longer cycle life, and better safety than traditional Li-ion batteries, which have attracted a great deal of attention. To develop the high-performance SSLB, the priority is to produce the solid state electrolytes, which are higlhy ionic-conducting, chemically and electrochemically stable, and easy to process. Recently, we have succeeded on large-scale fabrication of the garnet Li7La3Zr2O12 (LLZO)-based powders, based on which the high-dense ceramics show the relative density above 99% and the ionic conductivity above 10-3 S/cm at room temperature. Using the LLZO nanoparticles, the flexible membrane electrolytes with PEO have been fabricated, which show the ionic conductivity of 2.1×10-4 S cm-1 at 30°C and 5.6×10-4 S cm-1 at 60°C. The solid state LiFePO4/PEO:LLZTO/Li and LiFe0.15Mn0.85PO4/PEO:LLZTO/Li cells exhibit large energy density and good cycle performance. Using the dense ceramic disks as the electrolytes, the Li-air batteries with the porous carbon-based air cathodes and the Li metal as the anodes work for many cycles under ambient air, especially at elevated temperatures. In this presentation, the scientific and technical details for these batteries will be given. The fundamental issues in particular with the interfacial issues and promising potential for application will be addressed.

4:20 PM

(ICACC-S6-021-2017) Mechanical Degradation of All Solid-State Batteries: Mesoscale modeling of energy storage materials (Invited)

G. Bucci*[1]; Y. Chiang[1]; W. C. Carter[1]; 1. Massachusetts Institute of Technology, USA

All-solid-state rechargeable lithium-ion batteries (ASSBs; i.e., those using solid-state electrolytes SSE) have attracted much interest because they have features particularly favorable for large-scale application. The replacement of organic liquid electrolytes with non-flammable inorganic SSEs simplifies the battery design and improves safety and durability. However, the mechanical behavior of such electrodes will be considerably different, since ASSBs impose an external elastic constraint on electrode particles. During charge and discharge of Li-ion batteries, anode and cathode materials expand and contract as they intercalate or de-intercalate Li. In ASSBs constraint-induced stresses develop during cycling, potentially reaching values in the order of magnitude of 1 GPa. To address the question of reliability in ASSBs, the present research focuses on the development of a nonlinear continuum model and a computational framework based on large deformation theory and in a thermodynamically consistent fashion. Crack formation and propagation is predicted by means of a cohesive zone model. The extension of damage is computed in relation to material properties such as the fracture energy and Young's modulus of the electrolyte and the chemical expansion of the active material. The link between mechanical degradation and battery performance is analyzed.

4:50 PM

(ICACC-S6-022-2017) Direct preparation of composite granules composed of cathode and electrolyte particles for all-solid-state Li-ion batteries by mechanical method

T. Kozawa*[1]; A. Kondo[1]; K. Fukuyama[1]; M. Naito[1]; H. Koga[2]; H. Iba[2];
1. Osaka University, Japan; 2. Toyota Motor Corporation, Japan

All-solid-state Li-ion batteries (LIBs) have strongly received much attention because these batteries have obvious advantages over traditional organic-liquid-based batteries in terms of high safety, high energy density, and flexibility. However, the disadvantage of all-solid-state LIBs is a ensuring of conductive paths of Li^+ ions and electrons. The build-up structure of composite particles consisting of active material and solid-state electrolyte in which each particle is homogeneously dispersed and well connected is one of the answers to overcome this problem and considered to be a suitable electrode for all-solid-state LIBs. Here, we report the direct preparation of composite granules for using as the cathode of all-solid-state LIBs by mechanical treatment. The mechanical treatment of cathode particles and solid-state electrolyte nanoparticles by using an attrition-type mill can provide the homogenously dispersed composite granules with the size of several tens of micrometers. When $LiCoO_2$ and $Li_{1.3}Al_{0.3}Ti_{1.7}(PO_4)_3$ were used as the model materials for active material and electrolyte, respectively, the first discharge capacity of 50 mAh/g was obtained. The capacity retention was 90% after 20 cycles. This method will become a low-cost fabrication method of electrode particles for all-solid-state LIBs.

S7: 11th International Symposium on Nanostructured Materials: Functional Nanomaterials and Thin Films for Sustainable Energy Harvesting, Environmental and Health Applications

Solar Fuels I

Room: Coquina Salon A
Session Chair: Teresa Andreu, Catalonia Institute for Energy Research

1:30 PM

(ICACC-S7-011-2017) Air-Stable High Photocurrent Perovskite Solar Cells Based on $CH_3NH_3PbI_3$-NiO Nanoparticles Composite (Invited)

Y. Hahn*[1]; 1. Chonbuk National University, Republic of Korea

A simple eco-friendly one-step microwave-assisted reduction (MWAR) method was developed for the production of silver nanoparticles- reduced graphene oxide (Ag-rGO) composites. The field-effect transistor fabricated with the Ag-rGO composite showed p-type behavior with high mobility of $3.3x10^5$ $cm^2V^{-1}s^{-1}$ and conductivity of $9x10^6$ S/m which is one-order of magnitude greater than pristine graphene (i.e., $1.59x10^5$ S/m). As-synthesized Ag-rGO composite was introduced into the active layer of bulk heterojunction solar cell based on P3HT:PCBM. Compared to the P3HT:PCBM only device (i.e., control device), the Ag-rGO implemented device showed a power conversion efficiency (PCE) of 4.23 %, which is about 42 % increase over the control device (i.e. PCE=2.98 %). This dramatic increase in PCE was found to be mainly due to an increase in short-circuit current (J_{sc}) from 9.55 to 12.76 mA/cm^2 (about 33 % increase), suggesting that the incorporation of p-type Ag-rGO into the active layer enhances the charge carrier generation and fast extraction of holes to the electrode. Furthermore, the Ag-rGO composite based solar cells without encapsulation showed remarkable air stability with retaining ~90 % of its original PCE and ~93% of J_{sc} for 30 days under ambient environment, attributed to gas barrier feature of the randomly distributed graphene sheets.

2:00 PM

(ICACC-S7-012-2017) The impact and diversity of surface chemistry and functionalization on membrane separation technology (Invited)

V. Meynen*[1]; 1. University Antwerp, Belgium

Ceramic materials are applied in a wide diversity of applications depending on their structural and physico-chemical properties. However, although their surface properties can be varied, controlling them is challenging. Moreover, the diversity of chemical interactions is much more limited than in organic chemistry. Hence, surface modification of ceramics with organic functional groups combines the diversity of organic materials with the structural benefits of ceramics, driving technological improvement. Not only the organic group but also the surface functionalisation mechanism creates a large matrix of control, adjustable to the specific requirements of the application. Adding an extra dimension of chemical recognition and interaction in ceramic membranes is rapidly expanding their field of application, showing the huge potential of surface control to drive process intensification. Furthermore, the impact of surface control on the molecular level on the performance in applications such as water purification and solvent filtration is demonstrated, revealing the unique features at the top layer, creating new opportunities. The span of control, advantages and drawbacks of tailoring structural features and surface properties will be discussed.

2:30 PM

(ICACC-S7-013-2017) Hybrid Perovskite Photodetectors (Invited)

T. Wu*[1]; 1. KAUST, Saudi Arabia

Hybrid perovskites, such as $CH_3NH_3PbX_3$, where X is Cl, Br or I, in high-performance photovoltaic research has aroused lots of interest from the materials science community. Their exceptional properties, such as low-cost solution processing, tunable direct bandgap, high light absorption and long carrier diffusion length, might lead to paradigm-shifting technologies in the near future. I will discuss the perspective of applying hybrid perovskites in photovoltaic and optoelectronic devices, with a focus on photodetectors and phototransistors. Our experiments on perovskite phototransistors provided direct evidence on the ambipolar transport in perovskite films with balanced electron and hole mobilities. As an approach towards high-mobility devices, we found that carbon nanotubes embedded in perovskite films can serve as one-dimensional transport channels and significantly enhance the charge mobility to a level comparable to common semiconductors such as crystalline silicon. Furthermore, two-dimensional metal dichalcogenides were used to boost the performance of perovskite-based photodetectors. Finally, I will discuss the application of perovskite microcrystal films as photodetectors that operate efficiently in both broadband and narrowband regimes.

3:00 PM

(ICACC-S7-014-2017) Boron nitride based nanostructured materials for energy, environmental and health applications (Invited)

P. Miele*[1]; 1. Université de Montpellier, France

The development of new materials for near-future technological challenges is dependent on the elaboration of ceramics with suitable morphologies, shapes and properties. This goal can be achieved by both the utilization of non-conventional chemical methods and the related preparation of tailored precursors. In the case of non oxide ceramics, the pyrolysis of preceramic precurors is a useful process for preparing materials with a controlled chemical composition and in complex shapes. Several examples of Polymer Derived nano-Ceramics will illustrate this elegant method as well as their use in energy applications, particularly for Hydrogen storage. We will focus on boron-based PDCs displaying various forms and sizes. 2D material such as graphene and boron nitride nanosheets are very attractive for many applications particularly as sensors, electronics and catalysis devices. In the case of BN, novel properties can arise from BN nanosheet due to the high surface area and edge structures. We used the exfoliation as the main tool for the creation of controlled two-dimensional nanostructured interfaces in order to investigate their performances in energy, environmental and health applications. We will show examples of how these methods can be used to create membranes for osmotic energy harvesting and water treatment, optical sensors and biosensors, and bionanocomposites materials for packaging.

Solar Fuels II

Room: Coquina Salon A
Session Chair: Yang Yang, University of Central Florida

3:30 PM

(ICACC-S7-015-2017) Enhancing Photon Harvesting via Plasmonic Nanostructures (Invited)

D. Ma*[1]; 1. INRS, Uni. Quebec, Canada

Harvesting solar photons from UV to visible and further to near infrared (NIR) represents an attractive approach to improve the power conversion efficiency of photovoltaics, since most of them are only able to efficiently capture photons in the UV and visible ranges. The same dilemma exists in photocatalysis. With unique, size- and shape-tunable surface plasmon resonance, plasmonic nanostructures have recently been explored for enhancing the efficiency of photovoltaics and photocatalysis via improved light scattering, strong near field effect and/or hot electron injection. Herein, I will present some of our recent development in Au and Ag plasmonic nanostructures and their applications in solar cells (e.g., quantum dot based solar cells) and photocatalysis. One example is about our recent synthesis of a new class of materials: plasmonic Au nanoparticle decorated upconverting- NaYF4:Yb3+, Er3+, Tm3+ -core@porous-TiO2-shell microspheres with uniform size distribution, high surface area and excellent stability and reusability. This nanohybrid catalyst exhibits broadband absorption from UV up to NIR and excellent photocatalytic activity. Different mechanisms were found to dominate under different excitations. In addition to yielding novel and interesting materials and properties, the current work also provides physical insights that can contribute to the future development of plasmon-enhanced broadband catalysts.

4:00 PM

(ICACC-S7-016-2017) Highly Efficient Solar Driven Catalytic Processes at Semiconductor Metal Oxide Photo-Electrodes (Invited)

R. Solarska*[1]; 1. University of Warsaw, Poland

Solar driven processes like PEC water splitting, activation of CO_2 or biocatalytic detection of biomolecules require long-term stability in line with their high solar to energy conversion efficiency. These two conditions are the critical issue that determine use of semiconductor materials in photo-electrochemical devices. Recent advances regarding tungsten oxide, owing the band gap of 2.5 eV, consist of in identification of new molecular oxygen evolution catalyst, active in acidic media and moderate doping of WO_3 with sodium ions. These two approaches allowed to enhance visible light absorption of tungsten trioxide and, consequently, its photocurrent conversion efficiency. The stable and reproducible water splitting photocurrents reached 4.5 mA cm^{-2} under standard conditions. Incorporated in small amount into electrolyte polyoxometalates: $[PMo_{12}O_{40}]^{3-}$ or $[PW_{12}O_{40}]^{3}$ have been found to act as highly effective in PEC devices molecular OER catalysts leading to very large enhancement of water oxidation photocurrents at the WO_3 photo-anode. To our best knowledge, this is the first example of non-noble metal PEC water splitting catalyst stable in acid media. Dissolved in the electrolyte, the POMs transparent to visible light, can easily penetrate in-depth over extended internal photo-active surface area and thus are particularly well suited for application with nanoporous photo-anodes.

4:30 PM

(ICACC-S7-017-2017) Colloidal core/shell QDs for solar driven hydrogen production (Invited)

H. Zhao*[1]; 1. INRS, Canada

Colloidal core/shell QDs have attracted much attention due to their unique size-tunable optical properties. In this talk, we focus on the synthesis of colloidal core@shell QDs including the PbS@CdS, CdSe/CdS and CuInSSe/ZnS QDs, the investigation the photoelectron transfer rate from QDs to wide bandgap semiconducting mesoporous films and further fabrication of photoelectrochemical (PEC) devices. The as-synthesized QDs were loaded into the mesoporous metal oxide thin film by a link-assisted methods. The different electron affinity of the oxides (SiO_2, TiO_2 and SnO_2), the core size and the shell thickness allow to fine tune the electron injection rate by determining the width and height of the energy barrier for tunneling from the core to the oxide. The results demonstrate the possibility of obtaining fast charge injection in NIR QDs stabilized by an external shell. We further developed a new hybrid photoanode architecture for PEC H_2 generation by using core/shell QDs. The optimized photoanode results in a remarkable saturated photocurrent density of 11.2 mA/cm^2, which is the highest reported value in a PEC system using NIR PbS/CdS QDs as sensitizers. In addition, we also use the green NIR CuInSSe/ZnS core/thin shell QDs and "giant" core/thick shell QDs for hydrogen production.

5:00 PM

(ICACC-S7-018-2017) Fabrication of perovskite solar cells using oxide nanostructured arrays

J. Wu*[1]; S. Lin[1]; H. Cho[1]; S. Lu[1]; 1. National Cheng Kung University, Taiwan

Perovskite-oxide nanostructured array solar cells, which active layer is composed of $MAPbI_3$ scaffolded by oxide nanodendrite (ND) and nanorod (NR) arrays, have been fabricated in this work. Compared to the perovskite-TiO_2 nanorod (NR) solar cell, the enhancement of PCE through improved controls of both electron collection and light propagation has been achieved in the perovskite-TiO_2 ND solar cell. The TiO_2 ND array in the active layer not only performs as efficient electron collector but also induces significant light trapping in the perovskite solar cells due to the three dimensional configuration. Compared to the $MAPbI_3$-TiO_2 NR solar cell, the 18% and 25% enhancements in the average Jsc and PCE are respectively attained in the $MAPbI_3$-TiO_2 ND solar cells. The performances of the perovskite-oxide nanostructured array solar cells will be discussed in this presentation.

5:20 PM

(ICACC-S7-019-2017) Effects of A and B Sites Doping on the Properties of Hybrid Perovskites for Photovoltaic Applications (Invited)

L. Malavasi*[1]; P. Quadrelli[1]; C. Milanese[1]; M. Patrini[2]; 1. INSTM and University of Pavia, Italy; 2. University of Pavia and CNISM, Italy

Hybrid organic-inorganic perovskites of general formula ABX_3, where A is a protonated amine, B is a divalent metal (usually Ge^{2+}, Sn^{2+}, Pb^{2+}) and X is a halide, are known since long time in the field of solid state chemistry. Such hybrid structures open important possibility of combining useful properties coming from the two different chemical entities. In addition, the extension of the well-established tuning strategies of inorganic perovskites also to the hybrid systems, may pave the way to fascinating and intriguing structure-properties modulations in the organic-inorganic perovskites. In the present contribution, we explored the use of various protonated amines as cation A in the $APbI_3$ perovskite structure, thus investigating the impact of branched amines, aromatic amines and heterocyclic amines on the structural and optical properties. In addition, substitution of Sn for Pb has been exploted in several compositions of hybrid perovskites. Samples have been synthesised with new and optimized protocols we have developed, and characterized for what concerns their structure and optical properties. Finally, mixed MA/FA systems were object of investigation with the aim of correlating the exact cation stoichiometry with structural and functional properties.

S8: 11th International Symposium on Advanced Processing and Manufacturing Technologies for Structural and Multifunctional Materials and Systems (APMT11)

Novel Ceramic Processing I

Room: Coquina Salon B
Session Chairs: Takashi Goto, IMR Tohoku University;
Valerie Wiesner, NASA Glenn Research Center

1:30 PM

(ICACC-S8-001-2017) Closed-loop Sustainable Processing of $CaCO_3$-SiO_2[$CaSiO_3$] Composites (Invited)

R. Riman*[1]; K. Blinn[1]; R. Anderson[1]; D. Kopp[1]; 1. Rutgers University, USA

This presentation will detail sustainable methods for the synthesis and processing of calcium silicate composites. We will describe how calcium silicate is synthesized from a low temperature hydrothermal reaction between limestone and silica. These cementitious powders are used to make green bodies that are densified by gas-assisted reactive hydrothermal densification (g-rHLPD). The densification process utilizes carbon dioxide (CO_2) to carbonate $CaSiO_3$ into a $CaCO_3$-SiO_2 mixture. The carbonation step creates high strength concrete whose materials whose performance exceeds that of traditional Portland cement concrete materials. More recently, our efforts have demonstrated the capability to make carbonate cement from indigenous waste and mineral streams all over the world. This breakthrough enables carbonate cement concrete to be prepared in virtually any part of the world without the high capital costs associated with traditional cement mills. Factoring in the reduction in energy and CO_2 emissions, this technology enables the concrete industry to make the largest reduction in CO_2 emissions in the history of industrial chemical manufacturing. The biggest challenge for this vital technology is creating a widely distributed CO_2 supply chain. Several approaches will be described that enable businesses to create a widely distributed profitable CO_2 network.

2:00 PM

(ICACC-S8-002-2017) Processing of SHS Derived Ti_2AlN Active Precursors Powders: Materials and their Properties (Invited)

J. Lis*[1]; L. R. Chlubny[1]; C. Kapusta[2]; 1. AGH University of Science and Technology, Poland; 2. AGH University of Science and Technology, Poland

Ti_2AlN is one of the compounds in the Ti-Al-C-N system which belongs to the interesting family of materials called MAX phases. These heterodesmic materials with mixed chemical bondings has nanolaminate structure which leads to the sets of extraordinary properties which are characteristic for both metals and ceramics. The Self-propagating High-temperature Synthesis (SHS) using combustion in nitrogen atmosphere was applied to produce Ti_2AlN active precursor powders using various reaction stoichiometry and nitrogen pressures. Such obtained powders were hot-pressed in various temperatures. The phase evolution of samples was observed and optimal sintering conditions were established. Basic mechanical and electrical properties of selected samples containing high amount of MAX phases were presented with the discussion directed to the applicability of such materials.

2:20 PM

(ICACC-S8-003-2017) Finite Element Analysis of Self-propagating High-temperature Synthesis of Nitrides

V. Doddapaneni*[1]; J. Lin[1]; A. Hiranaka[2]; T. Akiyama[2]; S. Lin[1]; 1. Lamar University, USA; 2. Hokkaido University, Japan

Self-propagating High-temperature Synthesis (SHS) is a cost effective way to synthesize advanced ceramic materials including oxides and nitrides. In previous works, synthesis parameters for nitrides production (e.g., Si + N_2 ---> Si_3N_4 and Al + N_2 ---> AlN), including stoichiometry, particle size, thermal conductivity, ignition time and additives were investigated to optimize the production of nitrides. In this work a 3-D time dependent dynamic mathematical model based on finite element analysis is developed to study the effects of these parameters on the SHS process. Momentum, heat, and mass transfers and reaction kinetics are integrated in this model. The interactive effects of thermal conductivity, ignition time and ignition flux on the initiation of the self sustained reaction, temperature history, reaction conversion and velocity of the reaction front movement are calculated using this model. The results obtained from this model are compared with experimental data.

2:40 PM

(ICACC-S8-004-2017) Highly flexible and transparent TiO_2 Nanowire UV detector via electrospinning assembling

Y. Wang*[1]; 1. TsingHua University, China

Here, a thin, transparent and nanometer-scale TiO_2-based ultraviolet (UV) photodetector has been fabricated by electrospinning. Well aligned TiO_2 nanowires were first collected on a quartz plate. Then, the nano-device was simply assembled by depositing interdigitated Pt electrodes on the surface. It was found that the deposited Pt electrodes form good ohmic contact with the underneath TiO_2 nanowires. Upon UV illumination, the photosensitivity of this device is up to over three orders of magnitude with relatively fast and stable response speed. During on and off cycles, this detector remains almost the same, indicating the reversibility and stability of the TiO_2 nanowires. These characters of TiO_2 nanowire-based UV detector make it a promising candidate for high-performance UV photodetector for applications in many field.

3:20 PM

(ICACC-S8-005-2017) Electrospun separators for structural battery applications (Invited)

J. Liang*[1]; 1. WPI, USA

Structural battery is a new approach that employs multifunctional material concept to use Lithium-ion battery (LIB) with load-bearing capability for structural applications. LIB usually consists of cathode, polymeric separator, and anode. The separator has been known as the weakest part of the cell. This work aims at creating electrospun polymer membranes with improved mechanical properties. Electrospinning (ES) employs the electrostatic force to control the production of nano-fibers from polymer solutions. Process parameters, such as type of polymer, concentration of polymer solution, ES voltage, and ES duration, have been studied to achieve the desirable membrane property. Many characteristics of electrospun polymer membrane would affect the performance of it as the separator in LIB, including surface morphology, microstructure, mechanical property, electrochemical performance, and thermal stability. In this study scanning electron microscopy, electrochemical testing, mechanical testing, and dynamic scanning calorimetry have been used to characterize the electronspun membranes and to optimize the process parameters for an improved separator for structural batteries. By controlling the composition of polymer solutions and the electrospinning parameters, Poly(Bisphenol A-co-epichlorohydrin) (PBE) memberanes demonstrating promising improvements were fabricated and tested.

3:40 PM

(ICACC-S8-006-2017) Synthesis of lithium titanate hydrate nanotubes by planetary ball milling and their conversion to $Li_4Ti_5O_{12}$ anode for Li-ion batteries

S. Suzuki*[1]; T. Kozawa[1]; T. Murakami[1]; M. Naito[1]; 1. Osaka University, Japan

Li-ion batteries (LIBs) are widely used as rechargeable batteries because of its high energy density. The lithium titanate oxide, $Li_4Ti_5O_{12}$ (LTO), is one of the most promising anode materials for LIBs. LTO exhibits excellent cyclability and stability. According to the previous reports, the morphology of LTO powders plays an important role to enhance the anode properties. These morphology-controlled LTO particles with a high performance are prepared by the solution reactions instead of the conventional solid-state reaction. The hydrothermal method is a useful method to prepare the morphology-controlled particles. The preparation of LTO particles via the hydrothermal method is usually conducted through the synthesis of $Li_{1.81}H_{0.19}Ti_2O_5 \cdot xH_2O$ (LHTO) and the thermal transformation of LHTO. Although the synthesis of LHTO by hydrothermal reaction can control the particle morphology, this is a relatively high-cost method than the solid-state method. Here, we report the wet mechanical method for preparing morphology-controlled LHTO powders. We synthesized LHTO powders in wet process by using planetary ball mill without heating. The synthesized LHTO powders had nanosheet and nanotube morphologies. After the thermal transformation of LHTO into LTO, the nanotube morphology was maintained. The prepared LTO nanotubes were operated as an anode for LIBs.

4:00 PM

(ICACC-S8-007-2017) Improvement of thermal conductivity and transmittance in AlN by microstructure control

T. S. Suzuki*[2]; K. Imai[1]; T. Nishimura[2]; H. Kiyono[1]; Y. Sakka[2]; 1. Shibaura Institute of Technology, Japan; 2. National Institute for Materials Science (NIMS), Japan

The thermal conductivity and transmittance of AlN is expected to be improved by controlling crystalline orientation, because AlN has hexagonal crystal structure and the thermal conductivity along the c-axis is higher compared with other directions, and the optical axis is aligned to be effective in transmittance. When the ceramics particles dispersed in the solvent are located in the strong magnetic field, the easy magnetization axis of particles rotates to the direction of the magnetic field by a magnetic torque. In previous studies, we reported that the a-axis oriented AlN could be prepared by a static magnetic field. On the other hand, sintering additives are needed to obtain dense AlN because of its low sinterability. In this study, we tried to make the c-axis oriented AlN with high thermal conductivity and possession of translucency by a rotating magnetic field and SPS. Ethyl alcohol slurry of AlN with CaF_2 as a sintering additive was prepared. Dense AlN was prepared by a slip casting in a rotating 12T magnetic field followed by SPS at 2073K. The thermal conductivity of the c-axis oriented AlN was improved compared with the random AlN. Translucent AlN can be fabricated with additions of 2 and 3 wt% CaF_2. The thermal conductivity and transmittance can be improved by high temperature annealing due to reduction of grain boundary phase caused by CaF_2.

4:20 PM

(ICACC-S8-008-2017) Towards Developing Comprehensive Understanding about Tribological Behavior of MAX Reinforced Metal (MRM) Composites

F. Al-Anazi*[1]; S. Ghosh[1]; S. Gupta[1]; 1. University of North Dakota, USA

$M_{n+1}AX_n$ (MAX) phases (over 60+ phases) are thermodynamically stable nanolaminates which has unusual and unique, properties. The MAX phases are highly damage tolerant, thermal shock resistant, readily machinable, and with Vickers hardness values of 2–8 GPa, are anomalously soft for transition metal carbides and nitrides. MAX phases display nonlinear, hysteretic, elastic behavior due to kink band formation in the basal planes. The composites of MAX phases with metals (MAXMET) are also important from both fundamental and applied perspective. Recently, we showed that lower concentrations of MAX Phases can also reinforce metal matrix and improve its tribological behavior. In this study, recent results on mechanical and tribological behavior of MAX-Metal (Bi, Ag, and In) composites will be demonstrated.

S9: Porous Ceramics: Novel Developments and Applications

Innovations in Processing Methods & Properties of Porous Ceramics II

Room: Coquina Salon G
Session Chair: Carolina Tallon, Virginia Tech

1:30 PM

(ICACC-S9-020-2017) Fabrication and properties of piezoelectric composites with various oriented microstructures created by freeze casting route

M. Fukushima*[1]; T. Fujiwara[2]; T. Fey[3]; K. Kakimoto[2]; 1. National Institute of Advanced Industrial Science and Technology (AIST), Japan; 2. Nagoya Institute of Technology, Japan; 3. University of Erlangen-Nuremberg, Germany

Piezoelectric materials that can transfer electric energy to mechanical energy and vice versa have been used for electromechanical transducers, in which polymer-impregnated piezoelectric ceramics defined as the composites of varied orientations and connectivities of the active ceramic and passive polymer phase have been widely studied. In this study, piezoelectric ceramics/epoxy composites with well-ordered 3-1 and 3-2 types of channel structure were fabricated by tert-butyl alcohol (TBA) based freeze-casting technique and followed by the impregnation of polymer, and the effects of channel orientations and the size on the dielectric and piezoelectric properties were investigated.

1:50 PM

(ICACC-S9-021-2017) Thermomechanical Properties of Novel Composite Materials, containing Fine Ceramic Fibres, designed for use as Diesel Particulate Filters

A. J. Houston*[1]; J. Dean[1]; B. Clyne[1]; 1. University of Cambridge, United Kingdom

Ongoing concerns about adverse health effects of carbon particulate in Diesel engine exhausts continue to drive the quest for improved performance from Diesel Particulate Filter (DPF) systems. Two of the main areas in which improvements are being sought are enhanced filtration efficiency of very fine particles (<~50nm), particularly immediately after regeneration (the periodic removal of accumulated particulate via combustion), and improved thermal shock resistance. One approach to achieving these aims is to create novel composite materials via the introduction of ceramic fibres. This has the potential both to enhance the fracture toughness, by promoting fibre pull-out, and to improve the filtration efficiency by creating multi-scale structures, with some gas flowing through very fine channels, while the presence of other coarser pathways ensures that the overall permeability remains acceptably high. This presentation covers the creation of novel DPF structures containing fine ceramic fibres and measurement of their porosity and permeability. Work is also presented on tomographic capture of DPF structures and simulation of the flow through them of hot gas containing fine carbon particulate (using COMSOL packages). It is concluded that there is scope for significant improvement in overall DPF performance via the incorporation of fine fibres.

2:10 PM

(ICACC-S9-022-2017) Microstructure and property of porous mullite with whiskers framework by sol-gel process

J. Yang*[1]; D. Zeng[1]; 1. Xi'an Jiaotong University, China

In order to combine relatively high strength and high porosity, porous mullite with whiskers framework has been fabricated by sol-gel process using tetraethylorthosilicate as precursor and nanosized α-Al2O3 and AlF3 powders as Al source. The effects of heating temperature and F existence on porosity, formation of mullite phase, microstructural feature and compressive strength were reported.

After heating at temperatures from 1100 to 1600°C, porosities were kept in range of 80 to 84 %, and interlocked mullite whiskers were formed and became elongated at high heating temperature due to the fast vapor-solid reaction rate. A compressive strength of as high as 16.1 MPa was obtained for the mullite ceramics heated at 1600°C, which was attributed to strong bonding among the smooth whiskers.

Innovations in Processing Methods & Applications of Porous Ceramics

Room: Coquina Salon G
Session Chair: Manabu Fukushima, National Institute of Advanced Industrial Science and Technology (AIST)

3:20 PM

(ICACC-S9-023-2017) Analysis of novel combustion and chemical conversion processes within porous ceramics (Invited)

D. Trimis*[1]; 1. Karlsruhe Institute of Technology, Germany

Combustion and chemical conversion processes in porous ceramics offer significant advantages in terms of power density, process stability and temperature control. Open cell foams from composite SiC based materials is a typically used ceramic structure. Ceramic structures fabricated with additive manufacturing techniques, e.g. at the template level, provide new opportunities with tailored heat and mass transport properties. In this paper numerical and experimental investigations on 3 exemplary novel combustion and chemical conversion processes in SiC based porous ceramics are presented. In the first case of porous burners, the flame structure within an open cell foam is compared to free flames. Laser induced fluorescence (LIF) is employed to visualize the flame zone, utilizing the hydroxyl radical (OH). In the second case, investigations on a partial oxidation process for synthesis gas production within an open cell foam are presented, providing axial species and temperature profiles within the structure. Superadiabatic effects due to the internal heat recuperation show a significant beneficial influence on the chemical conversion process. In the third case a catalytically assisted oxidative steam reforming process in porous ceramics is presented. The differences between open cell foams, cubic-cell-, Kelvin-cell- and Octet-cell-based structures are numerically and experimentally investigated.

3:50 PM

(ICACC-S9-024-2017) Overcoming Challenges in Processing and Near-Net-Shaping of Multi-Scale Porous UHTCs (Invited)

C. Tallon*[1]; 1. Virginia Tech, USA

The control of processing conditions is key to achieve the desire amount, size and shape of porosity in materials for any application. However, when particles with irregular shape, large size or differences in surface chemistry are to be used, finding the right processing technique becomes crucial to ensure the desired properties, especially when there is not information about the actual performance in the application. This work describes how these challenges were overcome to develop multi-scale porous UHTCs for high temperature insulation. The exhaustive control of interparticle forces and understanding interaction between additives and powder surfaces resulted in multi-scale highly porous ceramics using different processing routes (replica, particle stabilized foams plus gelcasting, ice templating and partial sintering). These colloidal techniques can render the required thermal conductivity across the component with a combination of different tailored porosities. Additionally, large complex-shape components with the same customized porous microstructure were prepared. The successful multi-scale microstructure was captured via x-ray tomography. The treated images were stacked to produce a virtual 3D microstructure, which is used to model and predict the material thermal properties to take the first steps towards predicting material performance and design of microstructure.

4:20 PM

(ICACC-S9-025-2017) Approaching high thermal stability and low thermal conductivity in porous SiC nanostructures

P. Wan*[1]; J. Wang[1]; 1. Institute of Metal Research, Chinese Academy of Sciences, China

The bottle-neck of current nanoscale thermal insulation materials, such as Al_2O_3 and SiO_2 aerogel composites, is their unsatisfied thermal stability (<1000°C). SiC exhibits superior thermal stability and mechanical properties up to high temperatures, however, it also possesses high intrinsic lattice thermal conductivity. So this carbide is never on the candidate list of high temperature thermal insulators. We herein show the ultra-low thermal conductivities and high thermal stability of porous β-SiC nanostructures by tailoring the microstructural characteristics. β-SiC nanoparticle packed beds show thermal conductivities of 0.068 ~ 0.1 W m^{-1} K^{-1} with porosities of 73.5 % ~ 62.9 %. And porous nano-SiC ceramics sintered at 1500°C using graphite flakes as fugitives exhibit thermal conductivities as low as 0.14 W m^{-1} K^{-1} with porosity of 76.3%. The breakthrough is realized by the introduction of multi-enhanced phonon scattering sources in SiC nanostructures including defects, interfaces and grain boundaries. We also report the raw SiC nanoparticle (~35nm) can retain the excellent thermal stability up to 1500°C. Our work provide new ideas to discover ultra-low thermal conductivity in advanced ceramics with excellent thermal stability, regardless their high intrinsic lattice thermal conductivities.

4:40 PM

(ICACC-S9-026-2017) Foam-reinforced Thermal Insulation for High Temperature and Cryogenic Temperature Applications

J. Stiglich*[1]; B. Williams[1]; V. Arrieta[1]; 1. Ultramet, USA

Ultramet has developed a highly insulating and lightweight thermal protection material composed of open-cell carbon or ceramic foam with an ultralow-density aerogel filler. The foam serves as an easily machinable structural reinforcement for the low-strength aerogel insulator and defines the shape of the component. The aerogel exists in discrete cells and is supported by the foam skeleton. The combined density of the composite insulator is as low as 0.1 g/cm^3, and the thermal conductivity is <1 W/mK at 2000°C. Aerogel-filled foam has also been shown to be beneficial for cryogenic insulation applications. Single panels up to 30″ square are feasible and can be press-fit over complex features. The benefits of reinforcing chopped fiber phenolic ablators with structural foam has also been demonstrated in arcjet testing to heat flux levels of >1000 W/cm^2 in which low erosion rates and heat transfer were indicated. The foam helps retain the char layer by physical reinforcement, and the network of passages allows pyrolysis gases to escape with minimal disruption of the surface char layer.

S10: Virtual Materials (Computational) Design and Ceramic Genome

Molecular Dynamics and Nuclear Materials Modeling

Room: Ponce DeLeon
Session Chairs: Haixuan Xu, Oak Ridge National Lab;
William Weber, University of Tennessee

1:30 PM

(ICACC-S10-017-2017) Integrating Computational and Advanced Electron Microscopy Techniques to Characterize Ion Tracks in Ceramic Oxides (Invited)

W. J. Weber*[1]; R. Sachan[2]; E. Zarkadoula[2]; D. S. Aidhy[3]; M. F. Chisholm[2]; Y. Zhang[2]; 1. University of Tennessee, USA; 2. Oak Ridge National Lab, USA; 3. University of Wyoming, USA

The structure and properties of nanoscale ion tracks created by fast ions in complex oxides are investigated. High angle annular dark field imaging and molecular dynamics (MD) simulations show that atoms in the shell structure surrounding the amorphous track core in $Gd_2Ti_2O_7$ are disordered with relatively larger cation-cation interspacing, suggesting the presence of tensile strain. Static pair-potential calculations show that planar tensile strain lowers the oxygen vacancy migration barriers, leading to enhanced oxygen ion conductivity in the strained shell structure. Electron-beam irradiation or thermal annealing of nanoscale ion tracks in $Yb_2Ti_2O_7$ induces restructuring of the ion tracks, indicating that locally controlled electron beam irradiation or thermal annealing can further modify the strain and properties of ion tracks. These results suggest that strain engineering can be used to the tailor transport properties of ion tracks. In Gd_2TiZrO_7, dramatic radial variations in ion track size are observed experimentally and confirmed by MD simulations. These large variations are due to differences in ionic radii, local strain effects, and the stochastic competition among Gd, Ti, and Zr to occupy the cation site in the defect-fluorite structure during recrystallization of the track shell structure. This work was supported by the U.S. DOE, BES, MSED.

2:00 PM

(ICACC-S10-018-2017) Atomistic to Mesoscale Modeling of Microstructural Evolution and Thermal Transport in UO_2 based Nuclear Fuels (Invited)

X. Bai*[1]; M. R. Tonks[2]; Y. Zhang[3]; J. Hales[3]; 1. Virginia Tech, USA; 2. Pennsylvania State University, USA; 3. Idaho National Laboratory, USA

UO_2 is used as the primary nuclear fuels in light water reactors. The harsh environment in reactors creates complex microstructural evolution in UO_2 and the microstructural evolution affects its physical properties such as thermal conductivity significantly. To model this multiscale process, in this work atomistic modeling such as molecular dynamics is used to provide science-based mechanisms for mesoscale modeling; Mesoscale modeling such as phase field incorporate these mechanisms to study microstructural evolution and physical property changes. To demonstrate the importance of using such a multiscale approach, two examples will be presented. First, we study how the grain growth is affected by the presence of a large thermal gradient in UO_2 fuels. We use molecular dynamics simulations to study the thermal gradient driving force and derive a science-based analytical equation. The analytical equation is used in phase filed modeling to study the grain growth behavior under a strong thermal gradient. Second, we incorporate the atomistic-level phonon scattering mechanism induced by Xe fission gas in mesoscale heat conduction simulations to study the thermal conductivity of UO_2 over a wide range of microstructural parameters such as gas bubble porosity, grain size, and concentration of dispersed Xe atoms.

2:30 PM

(ICACC-S10-019-2017) What makes the thermal conductivity of UO_2 reactor fuel so low, and how can we improve it? (Invited)

M. R. Tonks*[1]; C. Stanek[2]; D. Andersson[2]; F. Hilty[3]; 1. Pennsylvania State University, USA; 2. Los Alamos National Lab, USA; 3. Pennsylvania State University, USA

The purpose of a nuclear reactor is to generate electricity from heat resulting from nuclear fission in the fuel. Thus, the thermal conductivity of the fuel has a significant impact on the efficiency of the reactor. The fuel for light water reactors is UO_2, however UO_2 has a low thermal conductivity that gets even lower due to fission product formation and irradiation. In this presentation, we summarize efforts to use multiscale modeling and simulation to obtain a quantitative model of the UO_2 thermal conductivity and its changes during reactor operation. We also present our efforts to quantify the impact of higher thermal conductivity additives on the UO_2 thermal conductivity and investigate possible negative effects of the additives.

3:20 PM

(ICACC-S10-020-2017) Energy deposition and dissipation in coupled electronic and atomic subsystems in SiC (Invited)

Y. Zhang*[1]; H. Xue[2]; E. Zarkadoula[1]; R. Sachan[1]; W. J. Weber[2]; 1. Oak Ridge National Laboratory, USA; 2. University of Tennessee, USA

Understand, predictively model, and ultimately control the dynamic response of ceramic structures to energy deposition from irradiation at the level of electrons and atoms is a grand challenge in material science in diverse fields. To understand the energy deposition and dissipation processes in interconnected electronic and atomic subsystems in SiC, energetic ions are utilized to couple and decouple ionization effects from the electronic energy deposition and displacement damage from nuclear energy deposition. Ions with selected energy and mass deposit energy with desired density per unit ion path and preferred ratio between electronic and atomic subsystems. The corresponding ionization effect and displacement damage are investigated in a controllable manner to reveal coupled competing effects that are correlated both in time and length space. Defect dynamics resulting from separated and integrated accumulation of displacement damage and prolonged electronic energy deposition are investigated from both irradiation experiments and molecular dynamics simulations.

3:50 PM

(ICACC-S10-021-2017) Ab Initio Molecular Dynamics Investigation of Low Energy Recoils in Nuclear Materials (Invited)

B. Liu*[1]; Y. Gao[1]; W. J. Weber[2]; 1. Shanghai University, China; 2. University of Tennessee, USA

The microstructure evolution, including point defects, dislocation loops and voids, under irradiation generally provides detail information of the structural stability of nuclear materials. During last decades, simulations are a preferable choice to study the mechanism of cascades, especially at the atomic scale, due to the lack of experimental facilities. In this study, ab initio molecular dynamics are performed to study the low energy recoils in several nuclear materials, i.e. $SrTiO_3$, ThO_2, Ni, NiCo and Ti_3SiC_2. The threshold displacement energies which is important input parameter of SRIM code for calculating the defect production rate, are determined. More importantly, the underlying mechanism, including the effect of charge transfer, pre-existing defects, anisotropic structure and chemical bonding, the dynamic processes and the nature of final defects are clarified. These results provide fundamental science to advance the understanding of the irradiation processes.

4:20 PM

(ICACC-S10-022-2017) MEAM Parameter Space Optimization for Elemental Boron

E. Hernandez*[1]; M. Tschopp[1]; M. Baskes[2]; 1. U.S. Army Research Lab, USA; 2. Mississippi State University, USA

Boron is one of the least fundamentally understood elemental systems. In fact, until relatively recently, the ground state configuration for this element was contested among the community. This lack of understanding stems from the complex microstructure, where boron inherently desires to form icosahedra based configurations. Not surprisingly, most of the computational work relating to boron has focused on first principles calculation since developing high fidelity interatomic potentials has been challenging. While first principles can provide useful insights, its computational limits prohibits in-depth studies of the elemental system. In order to address this, we parameterized the MEAM potential enabling longer and larger scale simulations. We perform a systematic parameter domain study to identify which parameter influence the formation of the pervasive boron icosahedra. Furthermore, we perform design of experiments to understand the global interaction between these fitting parameters.

4:40 PM

(ICACC-S10-023-2017) Quantifying Similarities Between Gaussian–based Spectral Responses and its Application to Virtual Materials Characterization

E. Hernandez*[1]; S. Coleman[1]; M. Tschopp[1]; 1. US Army Research Laboratory, USA

We implemented a set of 49 similarity metrics, outlined by Cha, to analyze and quantify similarities between different Gaussian–based peak responses. To understand how these metrics performed at capturing expected peak changes (e.g. peak shift), we apply a methodological scheme that considers 6 expected peak behavior. To illustrate how these metrics can be used to characterize atomic simulations, we implemented the outlined procedure to study convergence of XRD pattern as calculated with LAMMPS' diffraction compute. We analyzed three microstructures and show how convergence is highly dependent on symmetry. Lastly, we show how even very simple systems showing negligible predicted structural differences can lead to substantially different spectral responses. These differences could in turn be quantified by the methods developed, adding a dimension to force field validation.

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

Production Root Technology IV

Room: Coquina Salon H

Session Chairs: Hisayuki Suematsu, Nagaoka University of Technology; Junichi Tatami, Yokohama National University

1:30 PM

(ICACC-S11-018-2017) Collapse Probability of Granules in Ceramic Powder Compact during Cold Isostatic Pressing (Invited)

K. Yasuda*[1]; S. Tanaka[2]; M. Naito[3]; 1. Tokyo Institute of Technology, Japan; 2. Nagaoka University of Technology, Japan; 3. Osaka University, Japan

Cold isostatic pressing (CIP) is widely used to produce dense and homogeneous ceramic products for advanced engineering applications. However, it is suggested that there still exists some fluctuation in density across the powder compact after CIP. This is caused by stochastic nature of granule collapse together with the dynamic change in stress distribution in the compact during CIP. In this presentation, the authors formulate the granule collapse probability as a function of radial coordinate in the powder compact under CIP pressure. We assume a spherical powder compact and its dynamic change in the radial stress distribution, thereby giving the joint probability density function for applied CIP pressure and radial coordinate by fracture location theory. From this function, we derive the point collapse probability function, suggesting that granule collapse occurs from the surface to the center of the compact. This research was partly supported by Grant-in-Aid for Scientific Researches (C) (25420706) and (B)(15H04129) from Japan Society for the Promotion of Science.

2:00 PM

(ICACC-S11-019-2017) Corrosion Measurements on High-Temperature Alloys under Humid Gas Atmospheres by Thermogravimetry and Mass spectrometry (Invited)

E. Post*[1]; 1. NETZSCH Geraetebau GmbH, Germany

A thermobalance equipped with a water-vapor furnace was employed to investigate zirconium alloy and steel samples under a water vapor atmosphere to 1200°C. The evolution of hydrogen due to the reaction of Zr with water was detected by a coupled quadrupole mass spectrometer (QMS). The design of the water-vapor furnace allows up to 100% water vapor at the sample, although it is limited to a maximum temperature of 1250°C. In this contribution, the setup of the measurement arrangement of the water-vapor furnace coupled to the QMS will be described. Results on zircaloy BCR-276 and steel samples will demonstrate the high efficiency of the thermoanalytical equipment employed. Higher temperatures also under high water-vapor flow are sometimes desirable to simulate, for example, the reactions of cladding materials (zircaloy) during a nuclear accident more appropriately. A modified standard SiC furnace for water-vapor applications up to 1500°C will be presented and first results will be shown.

2:30 PM

(ICACC-S11-013-2017) Motion control of the 3D nanostructured printed materials with electric field (Invited)

T. Nakayama*[1]; M. Herrera Salazar[1]; H. Suematsu[1]; K. Niihara[1]; 1. Nagaoka Univ of Tech, Japan

In this project we have developed microstructures using two photon polymerization process, in which two-photon are absorbed simultaneously by the material using an ultrafast laser causing its polymerization. The microstructures fabricated were observed by confocal microscope, this microstructures can be moved by an electric field; the experimental setup used consists in two electrodes (anode and cathode), and a high-voltage source. The voltage applied is 0.5 to 1.2 kilovolts, the electrons enter in the microstructure through the cathode and get out by the anode; the space between electrodes is 150 to 300 micrometers, this area is filled with a liquid polymer, YE5822 (A). The microstructures material is a polymer produced by a commercial photoresists (IP-G780) using a nanoscribe 3D printer, where the movement of the laser is directed by coordinates previously established by the user, which may be based on the imitation of a computer aided design (CAD) via STL files. In this work we demonstrated that polymer microstructures fabricated by two photon polymerization can be manipulated by an electrical field for its movement control, this phenomenon can be used for applications in micromechanics and microrheology. Microstructures fabricated by two photon polymerization driven by electrical field have not been reported previously to our knowledge.

3:20 PM

(ICACC-S11-021-2017) New Multifunctional Ceramic Coatings: Properties and High-Temperature Behavior (Invited)

P. Zeman*[1]; J. Vlcek[1]; J. Musil[1]; 1. University of West Bohemia, Czech Republic

Development of new thin-film materials with multiple functionalities and unique properties in a single material system are presently of great scientific and technological interest. Our research is focused on the design, preparation and characterization of new multifunctional ceramic coatings based on multielement nitrides, borides or oxides exhibiting a combination of several beneficial properties, e.g., high hardness, high optical transparency, high or low electrical and thermal conductivity, high thermal stability and oxidation resistance, low stress, high resistance to cracking, antibacterial activity, etc. The ternary, quaternary and/or quinary coatings allow us to control their elemental composition, structure and consequently their physical and functional properties. These coatings are deposited by reactive magnetron sputtering. The talk will be aimed at several coating systems including (1) amorphous Si-B-C-N coatings with an exceptionally high thermal stability and oxidation resistance, very high optical transparency and low thermal conductivity and expansion, (2) Zr-B-C-N coatings with very high hardness, high electrical conductivity and good oxidation resistance, (3) Hf-B-Si-C(-N) coatings with very high thermal stability of high electrical conductivity in air, and (4) ternary oxide-based coatings with high optical transparency, high thermal stability and high resistance to cracking.

3:50 PM

(ICACC-S11-022-2017) Developing the root technology for the future of advanced ceramics in Japan (Invited)

H. Sato*[1]; T. Shiozawa[1]; 1. Japan Fine Ceramics Association, Japan

Japan Fine Ceramics Association (JFCA) has 80 members including major advanced ceramics manufacturers and users in Japan. According to an industrial trend survey, the total domestic production of advanced ceramics reached approximately 2.5 trillion yen in 2015. To meet its members' needs, JFCA strives to develop the root technologies for the future of advanced ceramics in Japan and offers a variety of projects which includes developing international standards, fostering international cooperation, hosting domestic forums and developing an industrial roadmap. So far JFCA has successfully established more than 130 JIS and a number of ISOs related to advanced ceramics. In this presentation, we will take the SOFC related technologies as an example and discuss the efforts being made in Japan to develop the root technologies for advanced ceramics.

4:20 PM

(ICACC-S11-023-2017) Control of Particle Size and Copper Content by Electrical Circuit Parameter on Pulsed Wire Discharge in Liquid

T. Murayama*[1]; Y. Tokoi[1]; 1. National Institute of Technology, Nagaoka College, Japan

Pulsed wire discharge (PWD) is one of the methods for the preparation of nanosized powders using metal vapor/plasma. In this method, the vapor/plasma is generated by rapid Joule heating of thin wire due to a pulsed large current which is generated by a simple RLC electrical circuit. The vapor/plasma is rapid cooled by ambient gas or liquid, and it forms nanosized powders. In the previous research of PWD, coarse particles of several micrometers are contained in prepared powders. The coarse particles are generated by liquid droplets due to unequal heating of the wire. In this research, wire heating rate is controlled by an inductance (L) of the RLC electrical circuit and we report the relation of the wire heating to prepared powders on PWD in liquid. The experimental condition is as follows. The wire of Cu with 25mm in length and 0.1mm in diameter was installed in a chamber filled with liquid (water). The inductance of RLC electrical circuit was changed in 0.78 ~ 1.90 μH, and 10 μF capacitor was charged to 3 kV. From the XRD analysis, the prepared powders at all conditions were Cu and Cu2O. Cu content was decreased with increasing L. Additionally, crystallite size of Cu was decreased with increasing L. Therefore, we consider that the wire heating is very important factor for phase and crystallite size of prepared nanosized powders by PWD in liquid.

4:40 PM

(ICACC-S11-024-2017) Preparations and Optical Evaluations of the Tin Phosphate Glass

K. Motoki*[2]; S. Kamada[1]; T. Kozai[2]; T. Fujihara[2]; T. Konishi[2]; N. Uehara[3]; M. Kamano[2]; 1. Tokushima university, Japan; 2. National Institute of Technology, Anan College, Japan; 3. National Institute of Technology (NIT), Japan

In recent years, white color LEDs have been becoming widespread uses of luminaire devices. The present major method of white color LED is manufactured from blue LED and yellow fluorophor. This method has a problem that color rending properties are poor. The solution of the problem is to manufacture LED with high color rendering properties from ultraviolet LED and fluorescent glass. Then, we have been focusing on Tin Phosphate Glass, which has high flexibility in material selection among fluorescent glasses. Evaluation method on thermophysical properties of fluorescent glasses is required toward practical use. As the evaluation method, there is Photothermal Divergence Method. In this study, we have made several glass samples with different additive amount of Cu2O. The purpose of this study is to evaluate the effect of additive amount of Cu2O in Tin Phosphate Glass on light-emittings and thermal characteristics through several measurements including Photothermal Divergence Method. As a result, the increase of additive amount of Cu2O is not led to the increase of light emission intensity and fluorescence lifetime. However, it has an influence on the propagation speed of the heat.

5:00 PM

(ICACC-S11-025-2017) Self-crack healing and strength improvement in $Yb_2Si_2O_7$/SiC nanocomposites

S. T. Nguyen*[1]; T. Nakayama[1]; H. Suematsu[1]; T. Suzuki[1]; K. Niihara[1]; 1. Nagaoka University of Technology, Japan

For the first time, $Yb_2Si_2O_7$/SiC nanocomposite was fabricated by a two-steps processing. Yb_2O_3/SiO_2/SiC powder mixture were firstly sintered by hot-pressing at 30 MPa, 1550°C, 1 h in argon to obtain a $Yb_2Si_2O_7$/Yb_2SiO_5/SiC composite. Vickers indentation was applied on the surface of the composite to create a pre-crack defect. To activate the surface crack healing ability, composite was heat-treated at 1250°C in air for 2 h. The flexural strength measured by four-points testing method indicated that the composite strength was enhanced considerably. XRD analysis confirmed that the residual Yb_2SiO_5 formed during the sintering mostly transformed into $Yb_2Si_2O_7$. It is revealed that the mechanism for crack-healing behavior and silicate transformation is the oxidation of silicon carbide. The findings of this research suggest a potential method to synthesize $Yb_2Si_2O_7$/SiC composite for high temperature applications, such as environmental barrier coatings in gas turbine engine.

5:20 PM

(ICACC-S11-026-2017) Firing Processes of Ceramics measured by Thermal Analysis Instruments coupled to Evolved Gas Apparatus

E. Post*[1]; 1. NETZSCH Geraetebau GmbH, Germany

Thermal analysis methods like TGA-DSC or dilatometry are common and efficient tools for studying the sintering behavior and/or binder burnout of ceramics. Combining these methods with an evolved gas analysis method increases the information and allows often a better interpretation of the thermal analysis data and helps to improve the quality of the product, less energy consumption or environmental impact. Depending on the coupled EGA instrument (MS, FTIR, GC-MS) the detection/identification of the evolved gases can be optimized. In this contribution TGA-DSC and dilatometer measurement data were shown together with the results from evolved gas analysis on several oxide ceramics. The advantages/limitations of the different EGA methods will be explained.

S12: Materials for Extreme Environments: Ultrahigh Temperature Ceramics (UHTCs) and Nano-laminated Ternary Carbides and Nitrides (MAX Phases)

Structure Stability under Extreme Environments II

Room: Tomoka B

Session Chairs: Ryan Grohsmeyer, Missouri University of Science and Technology; Eric Wuchina, Naval Research Lab

1:30 PM

(ICACC-S12-019-2017) Cyclic Ablation of High Emissivity Sm-Doped ZrB_2/SiC Coatings

A. Brenner*[1]; A. Pena[1]; A. Stubbers[1]; R. Trice[1]; 1. Purdue University, USA

Samarium-doped ZrB_2/SiC (ZBS) coatings possess the dual properties of high emissivity and excellent ablation performance. The emissivity, measured to be 0.9 at 1600°C for 5 mol.% Sm dopant, increases radiation heat transfer during hypersonic flight, ultimately cooling the surface. The Sm dopant improves ablation performance by forming a stable oxide scale of $Sm_{0.2}Zr_{0.8}O_{1.9}$. Of interest in the current study is how repeated cyclic ablation effects the development of the scale, including the phase assemblage, microstructure, and surface roughness. Three different levels of Sm-doped ZrB_2/SiC (ZBS) coatings will be assessed. XRD and standard microscopy techniques will be used to assess the microstructures after 1, 5, and 10 thirty second ablation cycles, with temperatures reaching 1700°C.

1:50 PM

(ICACC-S12-020-2017) Oxidation behavior of Ti_2AlC powders in flowing air

F. Kong*[1]; K. Feng[1]; N. Li[1]; X. Qi[1]; Y. Zheng[1]; X. He[1]; R. Wang[1]; Y. Bai[1]; 1. Harbin Institute of Technology, China

The oxidation behavior of nonstoichiometric Ti_2AlC powders synthesized by combustion synthesis in flowing air was investigated by means of simultaneous TG-DTA, XRD, and SEM/EDS to reveal the intrinsic oxidation mechanism of this potential high temperature material. The onset temperature of oxidation was 300°C. The fine Ti_2AlC powders with the size of about 1 μm showed the largest rate of oxidation at 580°C and completing oxidation at 980°C, while with increasing the powder size around 10 μm the corresponding temperature was 920 and 1040°C, respectively. In isothermal oxidation, the changing oxidation mode made that the final mass gains of the oxidized fine-Ti_2AlC-powders at a higher temperature of 600°C was significantly greater than those at 400 and 500°C. The oxide was mainly rutile TiO_2 with a small amount of anatase TiO_2, while powder particles were covered with a white α-Al_2O_3. The tiny white flocculent particles of α-Al_2O_3 appeared on the surface of fine Ti_2AlC powders and increased with increasing the oxidation temperature. At 900°C, the nano needle-like protrusions of α-Al_2O_3 were present on these particles.

2:10 PM

(ICACC-S12-021-2017) Thermo-chemical surface instabilities of SiC-ZrB_2 ceramics in high enthalpy dissociated supersonic airflows

F. Monteverde*[1]; R. Savino[2]; A. Cecere[2]; 1. CNR-ISTEC, Italy; 2. University of Naples Federico II, Italy

The response of SiC-ZrB_2 ceramics at typical conditions of thermal protection systems of a re-entry spacecraft was studied. Evidences of the limits below which SiC-containing UHTC might play regarding the sharp leading edge technology for hypersonic flights were obtained. SiC-ZrB_2 lab-scale proof demonstrators, shaped like a flat button (1 cm diameter) were manufactured and tested in non-equilibrium supersonic airflows up to 21 MJ/kg of specific total enthalpy and 3.5 MW/m^2 cold wall heat flux, by using an arc-jet ground facility. The surface temperature of the demonstrator was determined coupling the recordings of an infrared thermo-camera (IR-TC) to those of a two-color pyrometer (PYR). Under these severe aero-heating conditions, the UHTC articles endured rather well, though incipient thermo-chemical surface instabilities in the form of hot-spots started taking place. A sintered SiC ceramic was also tested under the same conditions for comparison. A numerical code implemented into a commercial CFD package was used to interpret the observed phenomena. Potentials and limits of such a oxidation-resistant SiC-ZrB_2 system to withstand such extreme conditions were finally outlined.

2:30 PM

(ICACC-S12-022-2017) Development of high temperature brazing for joining of thermostructural composites

C. Madec*[1]; L. Chaffron[1]; S. Frédéric[2]; L. Salim[2]; F. Manuel[2]; C. Valérie[1]; B. Alain[3]; B. Pierre[3]; F. Marc[5]; B. Frédéric[4]; 1. CEA, France; 2. Université de Technologie de Troyes, France; 3. Université de technologie Belfort-Montbéliard, France; 4. Université de Bourgogne, France; 5. Mersen Boostec, France

The goal of the project is to find innovating process for joining ceramic and ceramic matrix composite materials in order to develop effective components in extreme environments. The work consists in developing fully dense nanostructured sputtering targets by means of two techniques: high energy ball milling and spark plasma sintering. The starting powder is a mixture of silicon and silicide. Different conditions and types of equipment (vibrating mill and attritor) were tested in order to obtain the finer and the most homogeneous powder. The as milled powders were characterized by X Ray diffraction, electron microscopy, electron microprobe and thermal analyses before sintering. Optimized sintering conditions have been investigated in order to get fully dense and homogeneous samples for different diameters while limiting grain growth. The work was funded by the French National Research Agency's "Assistense" program, no. ANR-12-RMNP-0014.

Novel Processing Methods

Room: Tomoka B

Session Chairs: Konstantina Lambrinou, SCK-CEN; Woongrak Son, Texas A&M University

3:10 PM

(ICACC-S12-023-2017) Additive Manufacturing of HfB_2 Parts

E. Feilden*[1]; D. Glymond[1]; F. Giuliani[1]; E. Saiz[1]; L. Vandeperre[1]; 1. Imperial College, United Kingdom

Additive manufacturing is reasonably well established for manufacturing conventional ceramic parts with complex geometries, but little attention has been given to less common ceramics such as UHTCs. This is in part due to the difficulties encountered in various additive manufacturing methods which often hinder full densification, and thus severely limit mechanical properties. In the present work we overcome many of these issues by using a 3D printing technique which consists of continuous extrusion of pastes, known as robocasting, to print HfB_2 in the form of complex shapes and simple bars which reach densities above 97% after pressureless sintering using B_4C as a sintering additive. First, the optimisation of the extrusion paste will be discussed with specific attention given to retaining the homogeneity of the distribution of the two phases of substantially different density while maintaining good rheological characteristics for printing. Secondly, the strength, hardness and toughness of the printed material will be compared to conventionally produced HfB_2 to demonstrate that this 3D printing technique is capable of producing reliable parts and thereby opens up the production of highly complex parts with internal structures.

3:30 PM

(ICACC-S12-024-2017) The incubation temperature effect on Sn whisker growth behavior on Ti_2SnC

P. Zhang*[1]; Z. Sun[2]; Y. Liu[1]; J. Ding[1]; C. Ling[1]; Y. Zhang[1]; 1. Southeast University, China; 2. National Institute of Advanced Industrial Science and Technology (AIST), Japan

Although the whiskering phenomenon is well documented, its growth mechanism has remained poorly understood. In this work we have investigated the Sn whisker growth behavior on Ti_2SnC-Sn substrates in argon atmosphere and the temperature effect. Compact samples were prepared by pressing the ball-milled Ti_2SnC-Sn powders under 200 MPa, and were then incubated for 72 hours in argon under 110°C, 160°C and 210°C, respectively. The 160°C incubation led to the highest number density of Sn outgrowth, while 210°C led to thicker and longer Sn whiskers than the other two. Both these two samples grew Sn polyhedrons on their surface in addition to whiskers. A catalysis-based mechanism is developed to interpret the Sn whisker growth on Ti_2SnC-Sn, with the cleavage planes of Ti_2SnC grains playing the role of heterogeneous nucleation sites. The temperature effects on the Sn whisker growth are attributed to the better wettability of Sn to Ti_2SnC, and the higher diffusion rate of Sn atoms. Tiny Sn films on the sample surfaces may melt at higher temperature and then solidify in equilibrium upon cooling to form Sn polyhedrons. The results will help uncover the general Sn whiskering phenomenon and secure the practical applications of MAX phase materials.

3:50 PM

(ICACC-S12-025-2017) Influence of Hot-Pressing Time on Phase Evolution of SHS Obtained Ti_3AlC_2 Active Precursor Powders

L. R. Chlubny*[1]; J. Lis[1]; P. Borowiak[1]; K. Chabior[1]; 1. AGH University of Science and Technology, Poland

Ti_3AlC_2 belongs to the very interesting group of ternary and quaternary nanolaminate materials called MAX phases which can be found in the Ti-Al-C-N system. Thanks to the different chemical bonding these layered structured materials have unusual set of properties locating them between metals and ceramics which lead to many potential applications as construction and functional materials. Ti_3AlC_2 active precursor powders were obtained by the Self-propagating High-temperature Synthesis (SHS) method. As a result of syntheses of substrates mixtures including elementary powders and intermetallic compounds active precursor powders containing high amount of expected 312 MAX phase were obtained. In the next step, synthesized powders were hot pressed in various sintering times and changes of phase compositions as well as changes in apparent density of obtained products were observed. Thanks to these observations, optimal hot-pressing conditions, which may help to obtain single phase or near-single phase dense products, were established.

4:10 PM

(ICACC-S12-026-2017) Formation of TaB_2-Based Ceramics for Concentrated Solar Towers as Solar Collector

K. Gurcan*[1]; E. Ayas[1]; 1. Anadolu University, Turkey

Concentrated solar power (CSP) towers are one of the most important solar energy systems. The most important factor in the operation of this system is the collector material. At the operating temperature of the tower the expected features for the materials used as a collector, is high mechanical and chemical stability, high absorption of sunlight, and low thermal diffusivity. It is well recognized that the combination of its excellent mechanical, optical and thermo-physical properties makes Tantalum diboride (TaB_2) which is a member of Ultra High temperature Ceramics (UHTC) is among the most promising candidate material for renewable solar energy systems. To improve mechanical and oxidation resistance of TaB_2, Si-based materials (SiC, $MoSi_2$, etc.) are added in different amounts as a secondary phase. On the other hand due to the high prices of starting materials for the synthesis of TaB_2 based ceramics, there is not enough literature in depth. In the present study, TaB_2 ceramic powders were produced via high-energy ball milling process and subsequently borothermal reduction of tantalum pentoxide and amorphous boron powders at relatively low temperature in vacuum. Generated TaB_2 powders were sintered with and without using a Si-based secondary phases by using Spark Plasma Sintering method. Phases, microstructural, mechanical and thermo-physical features of the samples were investigated extensively.

4:30 PM

(ICACC-S12-027-2017) Synthesis of Nano-TaC powder using modified spark plasma sintering apparatus and its densification

S. Lee*[1]; L. Feng[1]; H. Kim[1]; 1. Korea Institute of Materials Science, Republic of Korea

Nano-tantalum carbide (TaC) powder was synthesized by the carbothermal reduction of Ta_2O_5 and the nano-powder was densified into monolithic TaC ceramic with fine microstructure. Using a modified spark plasma sintering (SPS) apparatus, TaC powder was synthesized at 1450°C by the carbothermal reduction of Ta_2O_5. The synthesized TaC powder was densified at 2200°C by SPS for 30 min under 80 MPa without sintering additives. Nano-TaC powders with high purity were synthesized using a modified SPS apparatus. The synthesized powder had ultra-fine particle size (62 nm) and low oxygen content (<0.5 wt%) due to the advantages of SPS including low synthesis temperature, fast heating/cooling rate and the effects of current. The metal basis purity of the synthesized TaC powder was 99.94%. The synthesized TaC powder was densified without sintering additives at 2200°C by SPS. The average grain size of dense TaC (R.D.:98%) was about 4 μm. Nano-TaC powder with ultra-fine grains size and high purity was successfully synthesized using a modified SPS apparatus. Using the synthesized powder, the monolithic TaC ceramic with a relative density as high as 98% and average grain size of 4 μm was obtained.

S14: Crystalline Materials for Electrical, Optical and Medical Applications

Optical Material 2

Room: Tomoka C

Session Chair: Romain Gaume, University of Central Florida

1:30 PM

(ICACC-S14-017-2017) Direct imaging of light emission centers in two-dimensional crystals and their luminescence and photocatalytic properties (Invited)

S. Ida*[1]; T. Ishihara[1]; 1. Kyushu University, Japan

Light energy conversion material such as luminescent and photocatalyst play a crucial role in many types of energy-saving devices and renewable energy technology. Luminescent materials are often prepared by doping inorganic crystals to produce emission centers. However, luminescence intensity often decreases with increasing dopant concentration. The precise reason for this has still not been determined. One of the best ways to resolve this issue would be to carry out direct observations of individual photo-function centers in luminescent materials. Recently, two-dimensional oxide crystals (nanosheets) with photoluminescence characteristics have been reported. The nanosheets are prepared by exfoliation of layered compounds, and have a uniform thickness. For example, the thickness of perovskite $A_2B_3O_{10}$ (A: Ca, Sr, Ba, B: Nb, Ta) nanosheets corresponds to the length of three BO_6 octahedral units. Such nanosheets with a homogeneous ultrathin thickness are suitable host crystals for studying emission centers produced by doping. In the present study, we report direct observations of dopant atoms acting as emission centers in green-emitting Tb-doped $Ca_2Ta_3O_{10}$ nanosheet, and its unusual photoluminescence and photocatalytic properties.

2:00 PM

(ICACC-S14-018-2017) Analysis of carrier trapping centers on persistent phosphors (Invited)

J. Ueda*[1]; 1. Kyoto University, Japan

Persistent phosphors are materials showing continuous luminescence for long duration between several minutes and hours after ceasing excitation light, which is distinguished from phosphors that show luminescence with decay time between nanoseconds and milliseconds due to intrinsic transitions ions. Persistent phosphors have been used as luminous paints under indoor and outdoor environments for emergency signs, dial plate of clock and lighting in the night. The $SrAl_2O_4:Eu^{2+}-Dy^{3+}$ developed by Matsuzawa et al. shows the brightest and longest persistent luminescence among all the afterglow materials ever reported. After their discovery, many persistent phosphors doped with lanthanide ions or transition metal ions were discovered. Although many researchers have discussed the mechanisms in the persistent phosphor, the details of carrier transport path and carrier trap centers are not yet clear. In this study, the mechanism of persistent luminescence mainly in $SrAl_2O_4:Eu^{2+}-Dy^{3+}$ were investigated by photoluminescence, thermoluminescence glow curves, electron paramagnetic resonance and X-ray absorption spectroscopy.

2:30 PM

(ICACC-S14-019-2017) ZnO-Based Thin Films & Nanostructures for Energy Harvesting Applications (Invited)

D. Rogers*[1]; F. Teherani[1]; E. Sandana[1]; P. Bove[1]; 1. Nanovation, France

Zinc oxide (ZnO) is a remarkable, multifunctional semiconducting material with a direct, wide bandgap (Eg ~ 3.4 eV), intrinsically high transparency over the whole visible range, and a resistivity that can be tuned from semi-insulating right through to semi-metallic by doping. It also presents one of the highest piezoelectric responses of any semiconductor and has a relatively high thermoelectric figure of merit. Moreover, it has been judged to be biocompatible and has been approved for human consumption (in products such as vitamin pills) by the U.S. Food and Drug Administration. The figure below shows that ZnO is currently one of the hottest topics in materials science, with over 8000 publications in 2015 (based on a search of the Scopus database for the term "zinc oxide" in the abstract, title, or keywords). ZnO has become a hot topic because of its' distinctive property set plus a number of recent breakthroughs which predispose it for use in a whole range of energy harvesting applications from solar cells, through nanowire piezogenerators to thermoelectrics. In this talk, we will give an overview of these advances and present some of the wide range of ZnO-related devices and applications being researched at the moment with illustrations from the work of the French ZnO start-up, Nanovation (www.nanovation.com).

Semiconductor

Room: Tomoka C
Session Chairs: Didier Chaussende, CNRS; Gabriel Ferro, Université Claude Bernard Lyon 1

3:20 PM

(ICACC-S14-020-2017) Self-seeded growth of 3C-SiC from the vapour phase (Invited)

D. Chaussende*[1]; T. Ouisse[1]; O. Chaix-Pluchery[1]; I. Gelard[1]; E. Sarigiannidou[1]; J. Dedulle[1]; 1. CNRS, Univ. Grenoble Alpes, France

Known as the only cubic structure among the huge number of different SiC polytypes, 3C-SiC exhibits interesting physical properties that has made it extensively studied for electronic applications. Its bulk growth is still an open issue. Different attempts have already been reported from both the vapor phase and the liquid phase, but they did not give very convincing results. Indeed, all of them have encountered two main problems: i) the availability of high quality 3C seeds and ii) the availability of an adapted growth process. As a consequence, they resulted in poor quality material with low growth rate and/or small size. Here we present a high temperature vapour phase approach for the self-seeding of 3C-SiC crystals. By an appropriate control of the nucleation rate/ growth rate ratio, only a few nuclei are formed and their growth is performed under close to equilibrium conditions, at 2100°C and 0.2 mm/h. The resulting crystals, with an average diameter of 5 mm, exhibit perfectly faceted habits. Similarly to the well-known Lely crystals, they are of very high structural quality, with no measurable mosaicity and residual strain. The FWHM of the (111) and (100) X-ray diffraction rocking curves are less than 20 arcsec. Only a few defects are revealed by the KOH etching.

3:50 PM

(ICACC-S14-021-2017) The role of C vacancy on the mechanism of nitrogen incorporation into 4H-SiC during epitaxy (Invited)

G. Ferro*[1]; D. Chaussende[2]; 1. Université Claude Bernard Lyon 1, France; 2. Université Grenoble Alpes, France

Despite being a well document subject, nitrogen doping of 4H-SiC during vapor phase epitaxy is still lacking of a general model explaining the apparently contradictory trends obtained by different teams. In this presentation, the evolutions of nitrogen incorporation (on both polar Si and C faces) as a function of the main growth parameters (C/Si ratio, temperature, pressure and growth rate) will be reviewed and explained using a new model based on surface exchanges between the gas phase and the uppermost 4H-SiC atomic layers. In this model, N incorporation is driven mainly by the transient formation of C vacancies, due to H_2 etching, at the surface (on C polar face) or near the surface (on Si polar face). The C/Si in the gas phase is by far the dominant parameter for N incorporation as it directly impacts the probability of carbon vacancy formation. All the other parameters (temperature, pressure and growth rates) are mainly modifying the local C/Si at the surface. Within the proposed model, N incorporates rather on terraces than on step edges. This model can virtually be used for all types of reactors and to all impurities incorporating on C atomic site. In that sense, it is more global than the "site competition mechanism" because it addresses the nature of the incorporation sites, and how those sites can evolve with the physical parameters of the process.

4:20 PM

(ICACC-S14-022-2017) Preparation of Vanadium-doped SiC powder for growing semi-insulating SiC bulk crystal (Invited)

S. Jeong*[1]; E. Jung[1]; Y. Kwon[1]; M. Lee[1]; Y. Kim[1]; 1. Korea Institute of Ceramic Engineering and Technology (KICET), Republic of Korea

Semi-insulating SiC crystal applicable to radio frequency devices could be fabricated by purifying or compensating its n- / p- type impurities. Due to the difficulty in the reduction of impurities, however, the impurity compensation is more cost-effective fabrication method for semi-insulating SiC substrate. To compensate n- / p- type impurities, vanadium (V), an amphoteric impurity in SiC, has been used as an effective dopant. V could be doped in SiC by the co-sublimation of V powder with SiC powder. However, the precise control of vaporized V is not easy because the high vapor pressure of V, so that the uniformity of V concentration is hard to be obtained. Hence, in this study, V-doped SiC powder was applied to the bulk crystal growth of V-doped SiC. First, the V-doped SiC powder was prepared with direct carbonization method. Then SiC single crystal was grown with physical vapor transport (PVT) method using the synthesized V-doped SiC powder. The grown SiC ingot was investigated with ultra-violet fluorescence analysis, high resolution x-ray diffraction, micro-Raman spectroscopy and secondary ion mass spectrometry (SIMS) analysis. As results, we confirmed that the V concentrations in each wafers were relatively uniform, which suggested the V-doped SiC powder was effective to control the vaporization of V concentration in the grown crystals.

4:50 PM

(ICACC-S14-023-2017) Optoelectronic Properties in Ultra-small near Infrared Alloyed Nanoplatelets

Y. Zhou*[1]; M. Celikin[1]; A. Camellini[2]; G. Sirigu[2]; X. Tong[1]; L. Jin[1]; H. Zhao[1]; M. Zavelani Rossi[2]; F. Rosei[1]; 1. INRS-EMT, Canada; 2. Politecnico di Milano, Italy

Two-dimensional (2D) metal chalcogenide semiconducting nanoplatelets (NPLs) are regarded as an emerging class of innovative materials able to have an impact on photovoltaic, photoelectrochemical and optoelectronic devices. However, it remains challenging to synthesize high quantum yield NPLs with controlled ultra-small size that are suitable for optoelectronic applications. Here, we prepared ultra-small lead chalcogenide ternary NPLs ($PbSe_{1-x}S_x$) active in the near infrared (NIR) region via cation exchange starting from $CdSe_{1-x}S_x$ NPLs. The morphology, structure and optical properties of the NPLs are critically dependent on preparation parameters. The bandgap simulation of NPLs reveals that their lateral dimensions induce strong quantum confinement, reflected in size-tunable quantized absorption and photoluminescence (PL) features. To demonstrate their disruptive potential in the field of solar energy, the NPLs are applied as light absorber in a photoelectrochemical (PEC) system giving a higher saturated photocurrent density than that of standard quantum dots (QDs). The excellent optical quality together with their favorable bandgap suggests that the NPLs are good candidates for possible use in optical and electrical devices.

Poster Session A

Room: Ocean Center Arena

5:00 PM

(ICACC-FS2-P001-2017) Light Gated Zinc-Tin Oxide (ZTO) Thin Film Transistor Fabricated via Solution Process

I. Wang[1]; J. Li[1]; J. Chen*[1]; 1. National Cheng Kung University, Taiwan

Zinc tin oxide (ZTO) is a transparent semiconductor based on earth-abundant elements. In this work, ZTO films prepared by a solution route have been applied as the active semiconductor layer in a thin film transistor (TFT) with SiO_2 dielectrics and Si gate. The purpose of using solution process is owing to its advantages of low cost and feasibility for large area deposition. With thickness less than 10 nm, the ZTO film exhibits a good field-effect mobility of ~10 cm^2/Vs, small subthreshold slope of ~0.5 V/decade and high on/off current ratio of ~10^7. With light illumination of 405 nm wavelength and power density of 5 mW/cm^2, the light sensitivity is in the order of 10^6 for the I_D-off region and is around 10 for the I_D-on region. The light responsibility of ZTO TFT reaches around 2.5 and the maximum external quantum efficiency is 8. Furthermore, to see the light gating effect, the ZTO TFT is operated without applying gate voltage but under light illumination (405 nm wavelength) of various power densities. As a result, the solution-processed ZTO TFT can be gated with light illumination, in addition to the voltage gating mode. This finding will broaden the suitability of ZTO TFT in optoelectronic and optical communication applications.

(ICACC-FS2-P002-2017) Photovoltaic responses and first-principles calculation in ITO/(Bi_{1-x} Nd_x) FeO_3 ceramics/Au heterostructure

C. Tu*[1]; Y. Huang[1]; C. Lin[1]; Y. Hsieh[1]; 1. Fu Jen Catholic University, Taiwan

Photovoltaic (PV) external quantum efficiency (EQE~10%) and power conversion efficiency (PCE~0.82%) have been observed in lead-free Nd-doped $BiFeO_3$ multiferroic ceramics with indium tin oxide (ITO) and gold (Au) thin films under low-intensity blue light (wavelength λ=405 nm). Our results show promise for PV applications. The single-phase $(Bi_{1-x}Nd_x)FeO_3$ ceramics (x=0.03, 0.05, 0.07, and 0.10) were prepared by using the solid-state-reaction method. The ITO and Au thin films were deposited on ceramic surfaces by using dc sputtering as electrodes. The maximal open-circuit voltages (V_{oc}) and short-circuit current densities (J_{sc}) are in the ranges of 0.8-0.92 V and 1.0-5.2 A/m^2. The maximal PCE in the ITO/$(Bi_{0.95}Nd_{0.05})FeO_3$/Au is about 0.025% under illumination of standard solar irradiation. The origin of photovoltaic responses is mainly associated with the Schottky barrier between ITO thin film and $(Bi_{1-x}Nd_x)FeO_3$ ceramics. High-resolution transmission electron microscopy (HRTEM) and first-principles calculation have also been used to study lattice structure and electronic band structure. The calculation shows that oxygen vacancy can cause the defect states below the conduction band. The calculated direct band gaps for $BiFeO_3$ and $(Bi_{0.95}Nd_{0.05})FeO_3$ ceramics are about 2.309 and 2.274 eV, which are consistent with measured band gaps of ~2.24 and ~2.204 eV, respectively.

(ICACC-FS3-P003-2017) Developing Novel PMMA assisted Graphene Liquid Cells for In Situ Scanning/Transmission Electron Microscopy(S/TEM) Imaging via Extrusion 3D Printer

S. Shafiee*[1]; E. Firlar[1]; R. Shahbazian-Yassar[2]; T. Shokuhfar[1]; 1. UIC, USA; 2. University of Illinois at Chicago, USA

Imaging hydrated structures in electron microscopy has always been a challenge. Methods used till now include, cryo-, fluid cell- and GLCs/TEM imaging. Conventional sample preparation involves chemical fixation, staining, embedding in resins and sectioning, preventing whole cell imaging in its native state. Cryogenic approach requires freezing, hindering recognition of dynamic events. As a solution, fluid cell imaging was introduced in which sample is sealed by two layers of thick Si_3N_4 windows, making the atomic resolution imaging difficult. GLC, in which the liquid sample is encapsulated in between two thin graphene layers, resulting in obtaining the highest imaging resolution possible in liquid sample S/TEM imaging. Low mesh number TEM grids with graphene and lacey carbon supports are essential to visualize large objects, but this lacey support interferes with the sample. To overcome these, incorporation of PMMA to graphene was carried out, but the problem with this method was residual PMMA, which interferes with sample. To solve this, we have successfully printed PMMA meshes on graphene coated copper sheet via extrusion 3D printer instead of covering the entire surface of sheet, thus single layer of graphene with PMMA support can be obtained with highest uniformity and less waviness.

(ICACC-FS3-P004-2017) A Facile Method to Synthesize Highly Conductive Polymer Derived Ceramics with Arbitrary Shapes by Built-in Conductive Networks

S. Chen*[1]; L. Zhai[2]; 1. University of Central Florida, USA; 2. University of Central Florida, USA

In this paper, we proposed a prototype of synthesizing conductive polymer derived ceramics (PDCs) by using the built-in conductive networks. The conductive networks were formed by attaching the edge functionalized graphene oxide (EFGO) to the commercial polyvinal alcohol (PVA) foams. After immersing the homemade conductive networks into the precursor of polysiloxanes, the composites were cured and pyrolyzed to form networks embedded conductive PDCs. Desired shapes of the PDCs are convenient to be realized because the PVA foams are quite soft to be cut. The conductivity of the networks embedded PDCs can be up to 0.94 S/cm at room temperature, and the conductivity of that PDC at the environmental temperature of 600 celsius was increased to 1.7 S/cm. The huge electrical conductivity improvement of PDCs after pyrolysis compared to the precursor of polysiloxanes was contributed to the built-in conductive networks and the graphitic domains formed within the matrix during the pyrolysis.

(ICACC-S2-P005-2017) Development of Aerosol Deposition Apparatus with In-situ Pretreatment System of Ceramic Particles

K. Naoe*[1]; M. Nishiki[1]; 1. Hitachi, Ltd., Japan

The aerosol deposition method (ADM) is a technique to form dense and thick, typically over several micrometers, ceramic films directly on substrates even at room temperature. By impacting solid ceramic particles around 1 μm in diameter on substrates, many kinds of ceramic films, such as Al_2O_3, Pb(Zr, Ti)O_3, AlN, and MgB_2, can be obtained. To use ADM industrially, it is necessary to improve the deposition efficiency, because the amount of ceramic particles that are not deposited through the process can not be ignored to reduce the cost of the ceramic films. Therefore, we had investigated effective means to improve the deposition efficiency by clarifying the deposition mechanism. Our previous reports suggested that preventing H_2O from contaminating films was important to improve the deposition efficiency as well as the film properties. In this study, a novel apparatus of ADM with in-situ pretreatment system of ceramic particles was developed. By preheating ceramic particles in N_2 atmosphere without exposing them to the air, the H_2O content in films was reduced by 54%. In addition, this apparatus was equipped with the cyclical use system of ceramic particles, which enabled us to repeatedly use the ceramic particles that were not deposited on substrates through the process.

(ICACC-S2-P006-2017) Air plasma spray preparation of HfO_2/mullite/Mo(Si,Al)$_2$ environmental barrier coatings

H. Lan*[1]; 1. Institute of Process Engineering/CAS, China

The most heavily stressed components in future engines are likely to utilize tolerant fiber reinforced ceramic matrix composites (CMCs) with much higher temperature capability. CMC components need to be protected by environmental barrier coatings (EBCs) to prevent component recession in hydrocarbon combustion environments. In this study, a multilayered environmental barrier coatings consisting of a Mo(Si,Al)$_2$ bond coat, a mullite inter-diffusion barrier, and a HfO_2 top coat was prepared on C_f/SiC substrates by using an air plasma spray process. The phase constituent and microstructure of both the HfO_2 powders and the sprayed HfO_2/mullite/Mo(Si,Al)$_2$ coatings were studied. And the thermal shock resistance of the EBCs coatings at 1350°C was evaluated. Results revealed that the hydrothermal synthesized HfO_2 particle exhibited monoclinic structure, and its average grain size ranged from 15nm to 20nm. And the phase constituent of the sprayed HfO_2 coatings kept monoclinic structure up to 1600°C. The microcracks and holes were observed in the sprayed HfO_2 coatings. After 50 thermal cycles of air-cooling test, no degradation was found in the HfO_2 coatings. The composite oxides of Al_2O_3 and SiO_2 formed at both mullite/Mo(Si,Al)$_2$ and SiC/Mo(Si,Al)$_2$ interfaces alleviated the thermal stress and benefited the thermal-stability of the EBCs system.

(ICACC-S2-P007-2017) Fabrication and Properties of Plasma Resistant YAS(Y_2O_3-Al_2O_3-SiO_2) coated Al_2O_3 Ceramics

E. Park*[1]; H. Lee[1]; 1. Kumoh National Institute of Technology, Republic of Korea

Recently, in order to realize an ultrafine circuit wafer process in semiconductor industry, highly plasma resistant ceramic components have been increasingly required in its severe etching process. The typical plasma resistant ceramics are oxide materials such as Al_2O_3, Y_2O_3, etc. Especially, the Y_2O_3 bulk ceramics and the Y_2O_3-coated ceramic components by plasma thermal spray process have been paid great attentions due to their excellent plasma resistances. However, the Y_2O_3 bulk ceramic components are highly expensive, furthermore, surface failure of the Y_2O_3-coated ceramic components causes serious problems such as wafer contamination and low production yield thereby. This study aims to develop both a new plasma resistant ceramic materials based on YAS (Y_2O_3-Al_2O_3-SiO_2) system and a cost effective fabrication process by a simple YAS coating on the surface of dense Al_2O_3 ceramics. In this study, a number of batch compositions in YAS materials system were examined, prepared and coated on the densely sintered Al_2O_3 ceramics. And then, effects of the YAS compositions and the coating processing on the plasma resistance of YAS coated Al_2O_3 ceramics were thoroughly studied. The plasma resistance of the YAS-coated Al_2O_3 ceramics thereby showed significantly good plasma resistance, that is, 8 times higher than quartz, 2.7 times than Al_2O_3 and ~ 70% of bulk Y_2O_3 ceramics.

(ICACC-S4-P008-2017) Micro scale fracture behavior of rare earth doped magnesium aluminate spinel

F. Cui*[1]; A. Kundu[1]; R. Vinci[1]; 1. Lehigh University, USA

Adding certain rare earth dopants to polycrystalline magnesium aluminate spinel has been observed to modify grain growth behavior by altering the grain boundary chemistry and structure. These changes should also influence boundary fracture behavior but it is difficult to isolate the effects using conventional polycrystalline specimens. In-situ micro scale mechanical characterization methods have advantages over traditional experiments when focusing on effects of such nanostructure. In this study, we are developing a new mechanical testing configuration: a bowtie shaped micro beam with a chevron notch, which can achieve stable crack growth during in-situ fracture tests. By aligning the notch at a single grain boundary, the fracture plane is guided to measure the fracture behavior of a single grain boundary. Test results are compared with those from micro cantilever beams with single straight through notches that are not capable of stable crack growth. AFM is also employed to measure the grain boundary groove depth, in order to analyze the relative grain boundary energies. Hence, the grain boundary energies and the fracture toughness results provide a unified picture of the micro scale fracture behavior of the system.

(ICACC-S4-P009-2017) Raman Mapping of Amorphization Zones in Sphere-Impacted Boron Carbide

P. Cruz*[1]; G. Parsard[1]; G. Subhash[1]; 1. University of Florida, USA

Impact testing has been conducted with spherical tungsten carbide projectiles at around 350 m/s to induce amorphization, a structural weakening mechanism, in confined disks of boron carbide. The damaged zone was then infiltrated and preserved with epoxy before the specimen was sectioned perpendicular to the impacted surface at a distance from the impact site. Amorphization in boron carbide can be identified through the appearance of a broad peak near 1330 cm^{-1} in its Raman spectra, and thus area under this peak was used to map the severity of amorphization throughout the damaged region. A comparison of the characteristics of the amorphized zones beneath static indentations, dynamic indentations, and sphere-impacted regions will be performed. Implications of this research as it relates to the ballistic performance of boron carbide will be discussed.

(ICACC-S4-P010-2017) Observations of nanostructured explosion phase of boron nitride by emulsion detonation synthesis

M. Ornek*[1]; K. M. Reddy[2]; K. Xie[2]; C. Hwang[1]; A. Burgess[3]; J. Calado[4]; V. Domnich[1]; S. L. Miller[5]; K. Hemker[2]; R. A. Haber[1]; 1. Rutgers University, USA; 2. Johns Hopkins University, USA; 3. SprayWerks Technologies Inc, Canada; 4. Innovnano, Materiais Avançados, Portugal; 5. H&M Analytical Services Inc, USA

Explosion boron nitride (e-BN) is one of the metastable phase of boron nitride and it is an attractive lightweight ceramic, exhibiting excellent hardness, high compressive strength and decent thermal conductivity. However, e-BN is widely synthesized by shock explosion method and its synthesis is limited by the low yield from high-pressure and high-temperature requirements. Here we report a novel fabrication method – emulsion detonation synthesis, to produce nanostructured e-BN, using hexagonal BN (h-BN) as a starting material. Water-in-oil emulsions were mixed with starting h-BN powders and subjected to detonation. XRD, FTIR, and TEM observations showed the nanostructured e-BN was successfully synthesized. HRTEM, SAED and STEM-EDX revealed that these newly formed phases are ~10-20 nm in size and were grown from the h-BN matrix. These findings provide new avenues to mass-produce e-BN.

(ICACC-S4-P011-2017) Computer modeling of process of projectile penetration into hybrid armor panel with regular and irregular packed ceramic discrete elements

I. V. Kartuzov*[1]; O. Mikhaylov[2]; 1. IPMS NASU, Ukraine; 2. IPMS NASU, Ukraine

Effectiveness of application of hybrid structures in a construction of multilayered impact resistant panels designed to protect objects against high energy mechanical impact depends on both mechanical properties of matrix material, ceramic discrete inserts and structure of packing of crushing layer with these inserts, including their geometry. However, today, in the existing hybrid impact resistant panels (for example, under the trade mark Liba) only regular dense packing (like a crystal lattice) of ceramic cylinders with the diameter 13-18 mm in polymer matrix is employed. This effort is to present the results of comparative numerical experiment on penetration of rigid projectile into hybrid armor panel with variation of both material of ceramic inserts (including those on the base of highly boron compounds) and matrix (high strength polyethylene) as well as their geometry and structure of packing in the crushing layer of panel. Modeling is carried out at two scale levels. Deformation of every ceramic element is considered in terms of continuum approach, crushing layer is as a whole is considered as a collection of a large number of such objects with a given law of interaction.

(ICACC-S4-P012-2017) Deformation and shock response of boron carbide

M. DeVries*[1]; A. P. Awasthi[1]; G. Subhash[1]; 1. University of Florida, USA

Mechanical properties of boron carbide are investigated using molecular dynamics with the ReaxFF potential. Using rhombohedral symmetry of boron carbide crystal structure, a fully periodic structure is modeled using the LAMMPS software. The system is first equilibrated at 300K and 1 atmospheric pressure and then subjected to deformation through homogeneous strain using the NVT dynamics. Response to different levels of hydrostatic and shear strain are studied and pressure-volume relationships are obtained. At the next step, these studies are performed on different polymorphs to obtain their relative performance to deformation. The overall goal of this investigation is to elucidate the mechanisms for amorphization of boron carbide.

(ICACC-S4-P013-2017) MicroCT used to assess de-agglomeration and de-gassing methods for improvement of ISOBAM gel casting system for alumina

C. Moorehead*[2]; J. M. Sietins[1]; V. L. Blair[1]; 1. US Army Research Laboratory, USA; 2. Drexel University, USA

One of the basic requirements for fabricating high-quality bulk ceramic parts is maintaining a uniform microstructure with minimal porosity. In order to achieve that microstructure, a green body forming method must be used that minimizes both pore formation and agglomeration, which diminishes the driving force for sintering. Gel casting has been identified as an effective method for achieving such a green body, but is traditionally affected by several issues including porosity, cracking, and shrinking. Using a novel gel casting system based on ISOBAM, many of those issues are easily circumvented. While the ISOBAM system has many advantages, such as low binder content, no toxicity, and the ability to perform aqueous, room temperature casting, issues with degassing and particle dispersion must be addressed in order for the system to be useful for many applications. This work investigated the use of several de-gassing and de-agglomeration approaches, such as vacuum suction, vibration, ultra-sonication, milling, addition of steric dispersants, and micro-fluidization on ISOBAM-based gel casting of alumina green bodies. The effectiveness of each technique was visualized using high-resolution micro-computed tomography which allowed for quantification of defects. Micro-fluidization was found to mitigate agglomeration defects.

(ICACC-S4-P014-2017) Ballistic Performance of Boron Carbide Ceramics Prepared by Spark Plasma Sintering

B. Apak*[1]; M. Cengiz[1]; G. Goller[1]; O. Yucel[1]; F. Cinar Sahin[1]; 1. Istanbul Technical University, Turkey

As it is widely known boron carbide (B4C) is an advantageous armour material due to its extremely high hardness and low density. Although there are lots of studies concerning the ballistic performance of monolithic boron carbide, it is thought that there is a gap about ballistic performance investigation of the effects of metal addition to the boron carbide system in the literature. In order to fulfil this gap, 5 % (vol.) Al, Si and Ti metals are added to boron carbide and spark plasma sintered without any pre-shaping technique or binder addition. The spark plasma sintering temperatures used in this study were in the range between 1400 – 1550°C, which can be regarded as extremely low for boron carbide sintering. However, 95 % and higher relative density values are achieved. The ballistic performance of final hexagonal products is tested in accordance with NIJ 0101.06 standard by using 7.62 x 51 mm NATO Ball Full Metal Jacket (FMJ) with a lead core. The backing material is chosen as Al 5083 plate. Because of its relatively higher ductility, depth of penetration difference can be examined more clearly. The depth of penetration results of metal containing boron carbide composites are compared with the monolithic boron carbide ceramics. The hardness and fracture toughness of samples are measured by Vickers indentation technique whereas SEM is used for observing the microstructures.

(ICACC-S4-P015-2017) Growth of silver nanoparticles stimulate spectroscopic properties of Er^{3+} doped phosphate glasses: Heat treatment effect

I. s. Soltani*[1]; 1. Faculty of Tunis Manar, Tunisia

The melt quenching technique was used to prepare Erbium ion doped phosphate glasses containing silver nanoparticles (Ag NPs). The amorphous nature of the glass is confirmed by X-ray diffraction patterns. Homogeneous distribution of spherical Ag NPs was shown from the transmission electron microscopy (TEM). The EDX analysis shows the presence of Ag element. The DSC measurements indicate a relatively good thermal stability of the prepared glass samples. The characteristic surface plasmon resonance (SPR) band of Ag NPs was observed in the range of 420-570 nm in the optical absorption spectra. The SPR band stimulated by the presence of Ag NPs enhanced both the photoluminescence (PL) intensity and the fluorescence lifetime relative to the ${}^4I_{13/2} \rightarrow {}^4I_{15/2}$ transition of Erbium ion. Ideal PL enhancement was obtained after 12 hours of heat-treatment. The results indicate that there is a large influence of nucleation and growth of silver NPs on the optical and spectroscopic properties of the glass samples.

(ICACC-S4-P137-2017) The Effect of Powder Oxygen Content on the Microstructure and Mechanical Properties of Silicon Carbide

V. DeLucca*[1]; R. A. Haber[1]; 1. Rutgers University, USA

Silicon carbide (SiC) is an important material in industry and defense due to its excellent mechanical properties. Pure SiC will form a passivating oxide layer of silica on its surface in the presence of oxygen. This poses a problem when sintering as this layer can inhibit densification during solid state sintering. This work examines the effects of varied oxygen content levels in commercial silicon carbide powders on the microstructure and mechanical properties of dense SiC bodies after solid state sintering via the spark plasma sintering (SPS) method. Two SiC powders were obtained, characterized, and treated to introduce a range of different oxygen content levels. These powders were densified using boron carbide and carbon additives to produce samples with varied properties. The dense samples were characterized to determine the effect of the starting powder oxygen content on the microstructure and mechanical properties. Experimental observations show that SiC powder oxygen content affects not only the overall density of the SPS SiC bodies, but also the grain morphology and polytype composition.

(ICACC-S4-P138-2017) Hydrothermal Vapor Synthesis of $MgAl_2O_4$ powders

D. Kopp*[1]; R. Riman[1]; 1. Rutgers, The State University of New Jersey, USA

Magnesium aluminate spinel ($MgAl_2O_4$) powders are industrially used for the preparation of infrared transparent windows with exceptional ballistic protection performance. Current powder synthesis methods (i.e., solid-state, coprecipitation, and sol-gel) rely on high-temperature reactions between expensive precursors with large embodied energies. Overall, these methods have large energy requirements and therefore hinder economic success. This paper uses a low energy synthesis technique called Hydrothermal Vapor Synthesis (HVS) to crystallize $MgAl_2O_4$ powders from $Mg(OH)_2$ and α-Al_2O_3 precursors at temperatures as low as 370°C. Theoretical energy calculations reveal a 32% reduction over the solid-state method. Phase-pure $MgAl_2O_4$ powder was (1) produced by acid-washing an excess $Mg(OH)_2$ reaction and (2) hot pressed into a translucent ceramic disk.

(ICACC-S7-P016-2017) Synthesis of V-doped TiO_2 nanoparticles using the oxidant peroxide method and microwave-assisted hydrothermal treatment

A. Garcia[1]; F. A. Berutti*[1]; A. K. Alves[1]; C. P. Bergmann[1]; 1. UFRGS, Brazil

In this work, V-doped TiO_2 with good applicability for photocatalytic processes has been obtained using a low energy consumption synthesis based on oxidant peroxide method combined with microwave-assisted low temperature hydrothermal treatment. To prepare the material, titanium propoxide, hydrogen peroxide, and isopropyl alcohol were used. The influence of time and temperature during the hydrothermal step on properties such as morphology, crystallinity, phase composition, specific surface area were investigated. The photocatalytic activity of the powders determined for methyl orange degradation was investigated under a solar simulator. The results show that the V doping can restrain the crystal growth and the phase transformation from anatase to rutile. It was observed that with the increasing of the temperature during hydrothermal step, the photocatalytic properties were also improved. The nanostructured V-doped TiO_2 particles synthesized at 200°C and 30 min with this method showed photocatalytic activity comparable to commercial Aeroxide TiO_2 P25.

(ICACC-S7-P017-2017) Nature-inspired Nano-pillar Structures for Anti-microbial surface properties

S. Han[1]; S. Ji[2]; A. Abdullah[1]; D. Kim[1]; S. Yoon[3]; H. Lim[4]; D. Lee*[1]; 1. Chung-Ang University, Republic of Korea; 2. Yonsei University, Republic of Korea; 3. Chung-Ang University, Republic of Korea; 4. Korea Institute of Machinery and Materials, Republic of Korea

Multiple infections and diseases in plants, animals and humans are known to be triggered due to formation of biofilms on open surfaces. However, such overwhelming bacterial infections and emergence of disease are not able to be completely suppressed by simple chemical treatmetns. This is partly due to the formation of antibacterial medicine resistant bacterial strains, which leads to the demand for developing new approaches to prohibit the bacterial growth more effectively. In this study, we fabricate surface that mimic the natural mechanism of anti-bacterial processes using nano-pillar textured surfaces which mechanically disrupt bacterial outer cell membrane. We fabricated anti-bactericidal nano-pillars on quartz substrate via nanosphere lithography with variety of sharpness and height of pillars with fixed spacing of 0.5μm. Our results show that nano-pillars with specific height and diameter exhibited maximum bactericidal activity. Through such properties, the newly designed nano-pillar textured surfaces can be adopted to multiple areas of applications that require anti-fouling properties such as medical implant surfaces.

(ICACC-S7-P018-2017) Flexible piezoelectric energy harvesters based on the CNTs-doped 0-3 PZT/Epoxy nanocomposite

H. Kim*[1]; S. Lee[1]; 1. Electronics and Telecommunications Research Institute, Republic of Korea

Recently, polymer-based piezoelectric nanocomposites have received a great deal of attention for sensors, actuators and transducers. Typically, 0-3 piezoelectric nanocomposite consisting of a particulate ceramic randomly dispersed within a polymer matrix has many advantages of better flexibility, easier manufacturing and lower cost, compared to other types. However, it still has critical drawbacks of poorer piezoelectric properties since the ceramic particles within a polymer matrix are hard to be fully polarized. In this paper, the CNTs-doped PZT/Epoxy nanocomposites with PZT ceramic fillers of 81 wt.% and various amounts of carbon nanotubes (CNTs) additives are studied to achieve high performance flexible piezoelectric energy harvesters. To prepare the CNTs-doped PZT/Epoxy nanocomposite, various volume fractions of CNTs up to 0.4 wt.% were added to the nanocomposites. The electrical conductivity and dielectric constant results show that increase of the CNTs content can improve the electrical conductivities and, as a result, they can increase the poling efficiency of the composites. Based on the highest piezoelectric performance of the CNTs-doped nanocomposite (CNTs contents fo 0.08 wt.%), it was shown that the flexible piezoelectric energy harvester fabricated with the CNTs-doped nanocomposite film can generate an open-circuit voltage of 3.8 V and a short-circuit current of 120 uA.

(ICACC-S7-P019-2017) Fabrication and Characterization of WO_3 nanofibers synthesized via hydrothermal methods

S. Park[1]; C. Nam*[2]; 1. Hannam University, Republic of Korea; 2. Hannam University, Republic of Korea

Tungsten oxide (WO_3) is one of the interesting materials for wide applications in electrochromic devices, photo-catalysts, and gas sensors. For those applications, several attempts have been made in the synthesis of WO_3, such as structural modification, doping of various elements and morphology changes. Nanostructures of WO_3 have superior properties compared with its bulk structures. In this presentation, the growth mechanism of hexagonal WO_3 nanofibers have been studied by using scanning electron microscopy (SEM), X-ray diffraction, BET techniques, and UV-vis spectroscopy for morphology, structure, surface area, and band-gap measurements, respectively. The hydrothermal methods were used to fabricate nanofiber structures by adding oxalic acid in the process of the preparation of solution. The tungsten precursor of $Na_2WO_4 \times 2H_2O$ were dissolved in double distilled water. The control of pH was adjusted by adding diluted HCl solution. After adding the direct agent (oxalic acid), the resulting mixture was transferred to a Teflon-lined stainless hydrothermal reaction autoclave. The duration time and heating temperature during the hydrothermal synthesis are 1 h and 120°C, respectively. The obtained WO_3 powder showed single phase hexagonal structure, 20-40 nm diameter and 6-8 mm long nanofiber bundles.

(ICACC-S7-P020-2017) Photovoltaic effects in atmosphere controlling neodymium doped $BiFeO_3$ multiferroic ceramics

C. Lin*[1]; Y. Huang[1]; C. Tu[1]; 1. Fu Jen Catholic University, Taiwan

This work is to study the photovoltaic responses and effects of oxygen vacancies in $(Bi_{1-x}Nd_x)FeO_3$ (BFO100xNd) polycrystalline ceramics prepared by the solid state reaction with controlling N_2/O_2 gas-flow ratio during the calcining and sintering procedures. Grain morphologies, structures, nanodomains, and phonons have been investigated as functions of N_2/O_2 ratio by SEM/EDS, high-resolution synchrotron X-ray diffraction, high-resolution TEM and back-scattering Raman spectroscopy. Synchrotron soft X-ray absorption spectra(SXAS) of the O K-edge and Fe L-edge show that the iron valence state shift to lower energy and the hybridization between oxygen 2p and Fe 3d orbitals. The heterostructures of indium tin oxide (ITO) thin film /BFO7Nd ceramics/Au thin film were prepared by dc sputtering deposition to make p-n junction and the photovoltaic(PV) responses were carried out as functions of light intensity under near-ultraviolet illumination of λ=405 nm. Current-voltage(I-V) characteristic curves, photovolatic short-circuit current density, and open-circuit voltage exhibit strongly dependences on the oxygen percentage. The Schottky barrier between ITO film and $(Bi_{1-x}Nd_x)FeO_3$ ceramics. Is responsible for the photovoltaic responses. The maximum of the PV power conversion efficiency and external quantum efficiency can respectively reach 0.25% and 5% in ITO/BFO7Nd/Au for gas-flow ratio of N_2:O_2=1:1.

(ICACC-S7-P021-2017) Direct evidence of local ferroelectric ordering in $Ba_{0.9}Ca_{0.1}Ti_{0.9}Zr_{0.1}O_3$ thin films

D. Dorantes[1]; O. Solis-Canto[1]; G. M. Herrera*[4]; J. Holguin-Momaca[2]; S. Olive-Mendez[1]; D. Morales[1]; G. Tapia-Padilla[3]; 1. CIMAV, Mexico; 2. CIMAV, Mexico; 3. CONACyT CIMAV, Mexico; 4. CONACyT CIMAV, Mexico

Switching spectroscopy by piezo-response force microscopy (SS-PFM) in dual AC resonance tracking (DART) mode is an effective tool to elucidate the superficial and local switching behavior and piezo-response (PR) of ferroelectric thin films (TF). The PR is measured locally within a single domain and thus the response is independent of domain size. This work focus on the characterization by SS-PFM of $Ba_{0.9}Ca_{0.1}Ti_{0.9}Zr_{0.1}O_3$ (BCZT) TF. A bulk target of BCZT was prepared by the modify Pechini method and sintered at 1250°C. X-ray diffraction (XRD) pattern of this bulk suggests a perovskite-type tetragonal structure with P4mm space group. The TF were prepared on Pt substrate by using a radio frequency magnetron sputerring system. XRD results for the TF suggest a (211) preferred orientation. The piezoelectric response d_{33} was determined by analysing the local amplitud "off-state" measurements (electrostatic effects have been minimized) through the following equation $(V - V_1)\ d_{33} = D - D_1$. The TF shows a d_{33} of 34 pmV^{-1} at the maximum voltaje of 15 V with a coercive voltaje of 2.8 V. A DC voltaje was applied as a nanolitography tool to fabricate a nanostrucred pattern. Understanding the switching behavior in ferroelectric BCZT-TF in the nanometer range is relevant to the development and optimization of applications for example as a high-density data storage.

(ICACC-S7-P022-2017) Structural and magnetic characterization of Zr-substituted magnetite (ZrxFe3-xO4,0≤x≤1)

A. Gangwar*[1]; 1. IIT BHU Varanasi, India

We report the preparation of monophasic Zr-substituted magnetite (ZrxFe3-xO4; 0≤x≤1) nanoparticles by wet chemical route. Such a report is lacking in the literature. Rietveld refinement of the X-ray diffraction (XRD) data suggests continuous decrease in the lattice parameter with increased Zr-substitutions. From TEM analysis, the size of the as synthesized particles was found to be in the range of 5-20 nm. The differential scanning calorimetry (DSC) curves for the samples with different amount of substitutions displayed considerable differences in their behaviors. The room temperature specific magnetization at 2 T has been found to decrease from 50 (x=0.2) to 41 A m2/kg (x=1.0) with increased Zr-substitution. From XRD and magnetic behavior analysis we could suggest that due to occupation of Zr4+ ions at tetrahedral and octahedral voids of Fe3O4, the amount of Fe2+ was more at octahedral site for x≤ 0.4 and at tetrahedral site for x>0.4 respectively. The ferrofluids prepared from these magnetic nanoparticles possessed good heating ability and Specific Absorption Rate (SAR) values suitable for bioapplications.

(ICACC-S7-P023-2017) The low-temperature chemical synthesis of an LTO based material for Li-ion batteries

D. De Sloovere[1]; W. Marchal[1]; F. Ulu[1]; M. Van Bael*[1]; A. Hardy[1]; 1. Hasselt University, Belgium

Combustion synthesis can be applied to lower both synthesis temperature and processing time of ceramic materials. Heating a precursor solution containing metal ions, fuel and oxidizer to a low process temperature results in an exothermic reaction which provides the energy for complete sample conversion. This approach is dependent on a large number of parameters. The synthesis of LTO, an anode material for Li-ion batteries, typically comes at a high energy cost: the solid-state synthesis method requires temperatures up to 900°C to be kept for several hours. By applying the combustion synthesis method to the synthesis of LTO, the synthesis temperature of this material was reduced to 300°C and the processing time was reduced to the order of minutes. The thermal decomposition of the precursor was studied using TGA/DSC, while the phase composition and particle size of the product were determined with XRD, TEM and DLS. Regarding its electrochemical characteristics, the product consisted of LTO as main electrochemically active phase, with the presence of a small amount of impurities. While the discharge capacity of this material was 165 mAh/g at 0.1C for the first cycle, the 40th cycle at this rate showed a capacity of 158 mAh/g. At 1C, the discharge capacity was 140 mAh/g. These characteristics are explained by the small size (200-300nm) of the product, though there were also agglomerates.

(ICACC-S7-P024-2017) 3D structured indium tin oxide electrodes via ultrasonic spray deposition on high-aspect ratios

E. van den Ham[1]; G. Bonneux[1]; K. Elen[1]; G. Maino[1]; M. Van Bael*[1]; A. Hardy[1]; 1. Hasselt University, Belgium

In a world seeking a sustainable future, smart materials and structures are opted, among which 3D all-solid-state Li-ion batteries. Although deposition of functional materials in 3D forms a challenge, implementation of 3D all-solid-state batteries leads to devices with high power and capacity with excellent cycling capabilities. The deposition challenges are not limited to battery materials, as a reliable 3D structured electrode is essential for current collection. Currently, the choice of 3D electrodes is limited to ALD TiN and Pt. In view of up-scaling new materials are sought, including indium tin oxide (ITO). Typically, ITO is deposited by sputtering – which is of no use for non-planer geometries. We present ultrasonic spray deposition as a viable alternative, using the process developed by Gielis et al. Complete coating of the high aspect ratio micro-cylinders was achieved, at the optimal deposition temperature. With an acceptable resistivity of 9 mΩ●cm, successful current collection was shown in combination with a WO_3 anode during Li (de)intercalation. Therefore, the low-cost and high upscaling potential of this deposition method for 3D electrodes yields a promising route to realize 3D all-solid-state Li-ion batteries.

(ICACC-S7-P025-2017) The effect of polymer agent and porosity on photocatalytic properties and surface wetability of TiO2 nanocoating fabricated by sol-gel

E. Mehmandoustesfahani*[1]; M. Ghorbani[1]; 1. Sharif University of Technology, Islamic Republic of Iran

This approach relies on synthesis and characterization of high porous TiO2 based thin films and the effect of polymer agents on wettability and photocatalytic properties of surface. Thin layer films of TiO2 includes different weight percent of Polyethylene glycol (PEG) (20,30,40 gr/l) through sol-gel process aided with dip coating have been prepared over the quartz glasses. The crystal structure of titania powder was characterized by X-ray diffraction (XRD)and related surface area achieved by Brunauer-emmett-teller (BET). The photocatalytic activity of the TiO2 films evaluated by the decomposition of methyl orange. XRD shows stability of anatase phase in all powders at 500°C but the rutile diffraction pecks was appeared gradually by adding more polymer. The decomposition of PEG during high temperature treating is considered to be responsible for the generation of porous structure in the films and resulted in high specific surface area of (165 m^2 /g) for TiO2/40PEG films. Highest photocatalytic activity provided by decomposition of methyl orange through TiO2/40PEG film annealed at 500°C. Addition of PEG induced a marked increase in hydrophilic properties of all surfaces and a superhydrophilic surface with low contact angel(<10°) was provided for TiO2/40PEG after 1 hour UV- light exposure.

(ICACC-S7-P140-2017) Photocatalytic Activity of Nanocrystalline SnO_2-doped TiO_2

J. Miagava*[1]; D. Gouvêa[2]; 1. Insper, Brazil; 2. University of Sao Paulo, Brazil

TiO_2 is well known as an efficient photocatalyst and for its low cost and high stability. However, in order to optimize its performance, ions have been incorporated as additives to stabilize the surface or modify the band gap. In this work, $Ti_{1-x}Sn_xO_2$ nanoparticles were synthesized by the polymeric precursor method and UV photocatalytic activities were evaluated based on methylene blue degradation experiments. The doped samples showed enhanced efficiency, which is associated with their higher surface area. Photodegradation experiments performed using the same total surface area of catalyst revealed that, even though the sample containing 1% of SnO_2 presents an anatase-rutile mixture, this sample exhibited lower photocatalytic activity than pure anatase. In contrast, the TiO_2 sample synthesized in the presence of chlorine species, which is 12% rutile, showed better performance than 100% anatase, suggesting the existence of an appropriate rutile/anatase ratio to obtain optimal photocatalytic activity. X-ray fluorescence analysis showed that all chlorine volatilizes during synthesis and rutile phase stabilization occurs only during initial crystallization.

(ICACC-S8-P026-2017) Fracture of gamma-alon – h-BN anisotropic composites

M. M. Bucko*[1]; 1. AGH University of Science and Technology, Poland

Composites in the gamma-alon – h-BN system can be used as components of some metallurgical installations that have direct contact with molten or hot metals and a high thermal conductivity is necessary. Anisotropy of thermal conductivity makes additional opportunity to receive a heat sink in a direction perpendicular to a plane of contact with a hot metal which can reduce an insulating layer. SHS technique was used to obtain complex powders with both phases synthesized in situ. Mixtures of aluminium, aluminium oxide and different amount of boron were combusted in nitrogen and then the powders were hot-pressed. The h-BN grains in the composites showed plate-like shapes and crystallographic orientation; the (0002) plane of the h-BN structure was preferentially oriented on the surface perpendicular to the pressing force. The composite samples were subjected for compressive strength test while pressing force was applied in the perpendicular and parallel directions in relation to h-BN grains. The tests revealed anisotropy of the compressive strength of the composites in relation to boron nitride content. Vickers indentation was applied to cause cracks in the different directions and it was shown that paths of crack propagation were strictly connected with anisotropic microstructure.

(ICACC-S8-P028-2017) Hermetic crystal oscillator substrate use a direct bonded copper method

K. Chuang*[1]; K. Chiu[1]; 1. Industrial Technology Research Institute, Taiwan

Direct Bonded Copper Ceramic substrate (DBC) was been applied to large electrical components, such IGBT power modules, for harsh environment application. The minimum width/thickness of DBC copper layer cannot less than 0.5 mm/150 μm because the manufacture process limit. This study constructed a type 2016 (LxW:2.0x1.6 mm) DBC crystal oscillator substrate for hermetic packaging to replace traditional high-cost sliver brazing crystal oscillator substrate. Use a gas-eutectic method to complete the bonding of copper ring and HTCC Al_2O_3 substrate; it can be implemented copper width/thickness 150 μm /150 μm. The copper bonding shear strength up to 230-140 MPa, The bonding process time can be completed in 30-16 min. The results of frequency of packaged oscillator show it have similar hermetic reliability like traditional sliver brazing crystal oscillator substrate.

(ICACC-S8-P029-2017) High-temperature heat treatment giving enhanced visible light photocatalytic activity to TiO_2 with high concentration Nb doping

T. Yonezawa*[1]; Y. Tsujimoto[2]; C. Zhang[2]; T. Uchikoshi[2]; T. Ishigaki[1]; 1. Hosei Univesity, Japan; 2. National Institute for Materials Science (NIMS), Japan

High-concentration Nb-doped TiO_2 nano powders were synthesized by RF thermal plasma processing method. The doping concentration of Nb was up to 25 at%, which was much higher than that attained by usual wet-chemical methods. The powders were composed of major anatase phase and minor rutile one. The post heat treatment was performed at various higher temperatures than 700°C in air. The transformation from anatase to rutile proceeded including the ejection of Nb as $TiNb_2O_7$ from the TiO_2 host. Photocatalytic activity was examined for isopropyl alcohol (IPA) decomposition under visible light irradiation. Remarkably high photocatalytic activity was recognized by confirming the complete decomposition of IPA to CO_2.

(ICACC-S8-P030-2017) Boron Carbide powders: Specialized development for advanced sintering applications

T. Schmidt*[1]; S. E. Vogel[2]; 1. H.C. Starck GmbH, Germany; 2. H.C. Starck NA Trading LLC, USA

The unique combination of low specific gravity, excellent elastic modulus and high hardness makes boron carbide an attractive ceramic for personal armor. But B_4C is difficult to form and to sinter, so hot pressing technology remains the dominating manufacturing method; however it is limited in the geometries that can be produced. New requirements and increasing demand for more complex shapes became a challenge and led to more intensive development efforts regarding alternative production processes (e.g. pressureless sintering or slip casting) allowing more freedom for final shapes. Thus, finer and purer starting materials are needed; and in particular higher sintering activity is now a key property. Today's powder manufacturers face a multitude of challenges to satisfy their customers' requirements for such higher quality grades. H.C. Starck – Germany, a renowned producer of high class non-oxide ceramic powders, has taken that opportunity by introducing a set of micron and sub-micron class B_4C powders. The distinct grades vary by their fineness and purity due to a combination of various milling and purification techniques available through our versatile technology platform. We will discuss the results of chemical and physical powder characterization, with emphasis on the improvements achieved and their potential benefits on the use of these powders.

(ICACC-S8-P031-2017) Fabrication of AlN fibers via combustion synthesis with Mg additive

A. Hiranaka*[1]; J. Lin[2]; S. Lin[2]; T. Akiyama[1]; 1. Hokkaido University, Japan; 2. Lamar University, USA

Aluminum nitride (AlN) fiber is promising filler for heat transfer due to their high thermal conductivity, electrical resistance, and a thermal expansion coefficient close to Si. However, traditional methods of producing AlN fibers are time consuming and uneconomical. Combustion synthesis (CS), a process utilizing highly exothermic reactions, has been successfully used to produce high purity AlN. In comparison to conventional methods, this process is energy saving and can be operated at room temperature. In previous works, additives were effective in controlling the AlN product's morphology and reaction mechanism, in which the growth rate of AlN fibers was promoted by the addition of a volatile material. In our study, we investigated the effect of Mg addition on the formation of AlN fiber in a pressurized N_2 atmosphere using CS method. Mg and Al powders were mixed and electrically ignited through carbon foil with the igniter of Al:AlN. TG/DSC was used to study the reaction mechanism. The phase purity and morphology of the products after CS was analyzed by XRD and SEM respectively. The resultant products appeared to be white, needle-like fibers composed of mainly AlN with small amounts of Mg-Al alloy. The SEM images and EDS analysis showed that the obtained fibers were approximately 5 μm in diameter and 0.5 cm in length. The results suggest the possibility of innovative AlN fiber production through CS using Mg.

(ICACC-S8-P032-2017) Improvement of transparency in textured Alumina by slip casting in a magnetic field and SPS

T. Ashikaga*[1]; H. Kiyono[1]; T. S. Suzuki[2]; 1. Shibaura Institute of Technology, Japan; 2. National Institute for Materials Science (NIMS), Japan

Alumina has excellent optical properties and mechanical properties. It was reported that crystalline orientation even in diamagnetic ceramics could be controlled by a strong magnetic field and the c-axis of alumina was aligned parallel to the magnetic field. The axis of easy magnetization of crystal rotates to the direction of the magnetic field by a magnetic torque. In this research, we tried to improve the transparency in alumina by control of the microstructure with the crystalline orientation and densification using a slip casting in a magnetic field and SPS. The suspensions were consolidated by slip casting in a strong magnetic field of 12 T. The green compacts were densified by two-step sintering using SPS at 1273K for 1h followed by 1423K for 10 min. For comparison, powder was sintered by SPS without preparing the green compact through colloidal processing. The preparation of the dense and textured alumina polycrystal was achieved. The in-line transmission of alumina prepared from powder and green compacts without a magnetic field were 17.7% and 38.4%, respectively at a wavelength of 640nm. As a result, the transmittance was improved by colloidal processing. Furthermore, the in-line transmission of the c-axis oriented alumina was 48.5%. Improvement of the transparency of alumina with fine microstructure could be achieved by the c-axis orientation.

(ICACC-S8-P033-2017) Low magnetic field orientation of Si_3N_4 ceramics using multilayered-graphene coated composite particles

M. Sado*[1]; T. Takuma[2]; J. Tatami[1]; M. Iijima[1]; 1. Yokohama National University, Japan; 2. Kanagawa Academy of Science and Technology, Japan

Si_3N_4 ceramics exhibit intrinsic high thermal conductivity in the c-axis direction owing to its crystal anisotropy. The c-axis oriented Si_3N_4 ceramics has been prepared with a high and rotating magnetic field, which exhibited high thermal conductivity along the c-axis oriented direction. However, the space in the super conducting magnet is too small to mass-produce them. In this work, high thermal conductive c-axis oriented β-Si_3N_4 ceramics were fabricated by molding in a low and static magnetic field using multilayered-graphene coated β-Si_3N_4 particles as seeds for microstructural development. The synthesized β-Si_3N_4 and commercial multilayered-graphene particles were mechanically treated. An aqueous slurry consists of multilayered-graphene coated β-Si_3N_4 was molded in a static magnetic field 0.5T. As a result of firing at a high temperature, c-axis orientation of the sample prepared in the magnetic field was confirmed by XRD analysis. SEM observation showed that elongated β-Si_3N_4 grains grew parallel to the applied magnetic field. Thermal conductivity obtained with the parallel direction is 96 W/m K, which is higher than that by the normal direction (64 W/m K). Therefore, it found to be useful for preparing high thermal conductive Si_3N_4 ceramics to mold in the low and static magnetic field using multilayered-graphene coated β-Si_3N_4 particles.

(ICACC-S8-P034-2017) Ceramics Grain Cluster Boundary Fractal Micro-capacitors

V. Mitic*[1]; L. Kocic[2]; V. Paunovic[3]; Z. Vosika[3]; 1. Serbian Academy of Sciences, Serbia; 2. Faculty of Electronic Engineering, Serbia; 3. Faculty of Electronic Engineering, Serbia

The broad application of ceramics capacitors based on barium–titanate offers a challenge of constant researching of its structure. Intergranular structure and dielectric properties of ceramics depend on grain cluster boundary configuration. Porousity of powder material imposes two ingradients of cluster fractality: the negative space - collection of holes and pores, and the positive space made by collection of grains. Connection of these two spaces can be described by the concept of Minkowski hull: The belt zone between two objects. By using the classic, sphere-generating hull, it is possible to have six intergranular connection types. Here, the generalized Minkowski hull is used by replacing sphere by any bounded convex body. For the proposed concept of the generalized Minkowski contents, with the corrected degree diameter (scale depend diameter), exists new ways to contact of the ceramic grains in the cluster, between maximums and minimums of the grain boundaries. This approach directly affects description of the surface area energy reduction and concept of working temperature of BaTiO3-ceramics, i.e. its dielectric and conductive properties. Since the REE additives may increase fractality, a systematic study of REE-fractal dimension relationship may open the gate to ceramic supercapacitors.

(ICACC-S8-P035-2017) Additive manufacturing of complex-shape graded TiC-steel composites

A. Levy*[1]; A. Miriyev[2]; A. Elliott[3]; S. Babu[4]; N. Frage[1]; 1. Ben-Gurion University of the Negev, Israel; 2. Columbia University, USA; 3. Manufacturing Demonstration Facility, USA; 4. University of Tennessee, USA

Functionally graded ceramic-metal composites (cermets) exhibit wide range of properties, controllably altered throughout a single material piece. One of the long standing challenges has been the inablility to fabricate cermet parts with complex geometries. The present work demonstrates a novel integrated approach for fabrication of fully dense complex-shape graded TiC/steel composites by 3D-printing of TiC preforms and subsequent free infiltration with molten carbon steel. Thermodynamic analysis allowed to predict the final composition of the phases and design composites with tailored properties. Gradient in hardness of the composites was achieved by using titanium carbide with different stoichiometries (TiC_x) which provides different carbon contents in the steel across the printed part during an infiltration, and hence- different properties of the steel after solidification and heat treatments. The microstructural characteristics of the graded cermets and the effect of the composition on hardness gradient after the heat treatment are discussed in details. The suggested approach opens the door for a wide range of advanced cermet applications in tooling, wear, aerospace and ballistic fields.

(ICACC-S8-P036-2017) Electrical, Mechanical, and Thermal Properties of Silicon Carbide-Boron Nitride Composites

Y. Seo[1]; Y. Kim*[1]; 1. University of Seoul, Republic of Korea

Silicon carbide-boron nitride composites were fabricated by conventional hot-pressing from β-SiC and h-BN powders with 2 vol% Y_2O_3 as a sintering additive. Electrical, mechanical, and thermal properties of SiC-BN composites were investigated as a function of the BN content in the starting materials. The addition of BN suppressed the grain growth of the SiC and triggered the $\beta \rightarrow \alpha$ phase transformation of the SiC in SiC-BN composites. A minimum electrical resistivity of 3.7×10^{-2} Ωcm was obtained for the SiC-4 vol% BN composite (SBN4). This low electrical resistivity was ascribed to N-doping in the SiC lattice, which acts as a donor for supplying electrons to the conduction band. Typical values for the electrical conductivity, fracture toughness, flexural strength, and thermal conductivity of the SBN4 at room temperature were 27 $\Omega^{-1}cm^{-1}$, 4.1 $MPam^{1/2}$, 566 MPa, and 87 W/m/K, respectively. The SiC-4 vol% BN composite can be electrical discharge machined to form complex shapes.

(ICACC-S8-P037-2017) Effect of heat treatment in α/β two-phase field on the structure and mechanical properties of Ti-7.5Mo alloy

J. Chern Lin*[1]; Y. Peng[1]; C. Ju[1]; 1. National Cheng-Kung University, Taiwan

A biocompatible, low modulus alpha-double-prime (α")-type Ti-7.5Mo alloy has been developed in the present authors' laboratory. The present study investigates the effect of heat treatment in the α/β two-phase field (roughly between 700 and 800°C at 7.5 wt% Mo in Ti-Mo binary phase diagram) on the structure and mechanical properties of the alloy. The Ti-7.5Mo alloy was prepared in-house using a commercial arc-melting vacuum-pressure type casting system operated in an argon atmosphere. The ingots were acid-cleaned and re-melted three times to improve chemical homogeneity of the alloy prior to be cast into a graphite mold at room temperature. The cast alloy was then heat-treated to the α/β two-phase field, followed by water quench. The experimental results indicate that the fast-cooled, as-cast Ti-7.5Mo alloy has an acicular morphology comprising fine, α'' platelets/needles uniformly distributed throughout the alloy. After being heat-treated into the α/β two-phase field, the XRD patterns indicate the β/α intensity ratio to increase with increasing heat treatment temperature. The yield strength and tensile strength of the heat-treated alloy are found to increase, while the elongation decreases, with heat treatment temperature. The research is supported by the Ministry of Science and Technology of Taiwan, ROC under the research grant MOST 104-2221-E-006-142.

(ICACC-S8-P038-2017) Anorthite ($CaAl_2Si_2O_8$)-Aluminum Interface: Kinetics of High-Temperature Interactions

E. Adabifiroozjaei*[1]; H. Ma[1]; P. Koshy[1]; C. C. Sorrell[1]; 1. The University of New South Wales, Australia

The kinetics of the anorthite-Al system at 850°-1150°C for 0.5-250 h have been examined. High-density anorthite was exposed to liquid aluminum and the ceramic-metal interfaces were investigated by electron microscopy (SEM, EDS, EPMA, and TEM). The results showed that Si^{4+}-Al^{3+} interdiffusion and associated oxygen vacancies plus Ca^{2+}-Al^{3+} interdiffusion and associated calcium vacancies drove the anorthite → CA2 and the CA2 → A (alumina) transformations, respectively, at 850° and 950°C. At 1050° and 1150°C, increased solubilities of silicon and oxygen in the liquid Al resulted in significant formation of CA2, which, when in contact with anorthite, reacted to form gehlenite. Alternatively, when CA2 was in contact with liquid Al, Ca^{2+}-Al^{3+} interdiffusion drove the CA2 → CA6 → A transformation path. When gehlenite was in contact with liquid Al, Si^{4+}-Al^{3+} and Ca^{2+}-Al^{3+} interdiffusion resulted in the formation of CA, the latter interdiffusion of which subsequently drove the CA → CA2 → CA6 → A transformation path. Si^{4+}-Al^{3+} interdiffusion was identified as the controlling process of the anorthite-Al interactions and so it has been quantified in terms of the activation energy of Q = 112 kJ/mol and the diffusion coefficient pre-factor of $D_0 = 4 \times 10^{-8}$ m^2/s.

(ICACC-S9-P039-2017) Highly active Ce-doped mesoporous alumina catalyst

I. Hussainova*[1]; M. Aghayan[1]; M. A. Rodríguez[2]; R. Mnatsakanyan[3]; B. Maaten[1]; F. Rubio-Marcos[2]; 1. Tallinn University of Technology, Estonia; 2. Instituto de Cerámica y Vidrio. CSIC, Spain; 3. Institute of Chemical Physics, Armenia

Ce-doped mesoporous gamma-alumina with a unique fibrous structure was prepared with the help of a novel wet-combustion synthesis method using a network of highly aligned alumina nanofibers with an average single fiber diameter of 10 nm. The effect of a process conditions on the morphology of the prepared catalysts and their performance in reducing and oxidizing environment were thoroughly studied by XRD, FESEM, TEM-EDS and DSC, DTA/TG analysis. Due to a high temperature achieved in a short time during reaction and a large amount of gases evolved, the wet-combustion synthesis yielded homogeneously precipitated nanoparticles of CeO2 on alumina nanofibers possessing an intrinsic catalytic activity. The fuel type used in the wet-combustion synthesis strongly influences the crystallite size of CeO2 particles ranging from 5 to 12 nm when glycine or urea is used, respectively. The homogeneity of distribution of CeO2 nanoparticles over the alumina mesoporous template depends on parameters of the synthesis as well as on the fuel type. Based on the results of the study, glycine is shown to serve as a fuel providing the homogeneous deposition of CeO2 with a narrow nano-particle size distribution. Due to the unique morphology, CeO2-Al2O3 nanostructure turns to CeAlO3 in hydrogen at 300°C and self-oxidizes back to CeO2-Al2O3 in air at room temperature. The catalyst shows high structural stability after several redox cycles.

(ICACC-S9-P040-2017) Platelets-induced stiffening and strengthening of ice-templated highly porous alumina scaffolds

H. Kang*[1]; M. Banda[1]; N. Dhavale[1]; D. Ghosh[1]; 1. Old Dominion University, USA

This presentation will describe the effects of the grain-level anisotropy on the microstructure and uniaxial compressive response of the ice-templated porous alumina ceramic scaffolds. Highly porous (~80 vol.%) scaffolds were fabricated from alumina powders of equiaxed morphology as well as from powder mixtures containing equiaxed and small amount of platelet particles. Presence of the platelets (diameter 8 μm and thickness 400 nm) led to the formation of the interlamellae bridges, and significantly enhanced the stiffness and compressive strength of the scaffolds. Measured improvement of the mechanical response is rationalized based on the stiffening and strengthening effects exhibited by the intralamella and interlamella platelets. Overall, results presented here are expected to provide significant insights to design bio-inspired hierarchical materials for structural, biomedical and energy storage applications.

(ICACC-S9-P041-2017) A comparison of microstructure and uniaxial compressive response of ice-templated alumina scaffolds fabricated from two different particle sizes

M. Banda*[1]; N. Dhavale[1]; H. Kang[1]; D. Ghosh[1]; 1. Old Dominion University, USA

This study investigates the effects of two different particle sizes (0.3 μm vs. 0.9 μm) on the microstructure and uniaxial compressive response of ice-templated sintered alumina scaffolds as a function of the solids loading of the ceramic suspensions and freezing front velocity (FFV). For a comparable solids loading and FFV, variation of the particle size is observed to have a significant effect on the microstructure of the fabricated scaffolds. Moreover, transition of the pore morphology with the increasing solids loading and FFV is observed to be more drastic for the scaffolds processed from the 0.9 μm size powder particles compared to the scaffolds processed from the 0.3 μm size powder particles. Similarly, particle size variation also influenced significantly the relative density and porosity of the scaffolds. Interestingly, in spite of the observed significant differences of the microstructure and relative density, uniaxial compressive stress-strain measurements revealed marginal effects of the particle size variation on the compressive strength. The measured comparable uniaxial compressive response of the sintered alumina scaffolds fabricated from two different particle sizes are rationalized based on the relative density, pore aspect ratio, and interlamellae bridge density.

(ICACC-S9-P042-2017) Cellulose Particles as Pore Creator for Ceramics

A. Czarnetzke[1]; S. Galic*[1]; C. Brandes[2]; S. Kroll[2]; K. Rezwan[2]; 1. J. Rettenmaier & Sohne GmbH & Co KG, Germany; 2. Universität Bremen, Germany

Organic fibers touch our lives each day; from the food we eat, to the products used in our homes – from medicines and vitamins, to innovative ceramics. J. Rettenmaier & Söhne is committed to the development and production of fibers from natural, renewable raw materials for industrial applications. One of our latest innovations is the usage of our cellulose particles in the wide field of ceramic production. This poster will show the function of our particles as natural pore creators. We will present the results of our joint project with Advanced Ceramics department of Bremen university. In this study we analyzed the differences of the cellulose particles regarding size, structure and raw material. We used cellulose particles in the size range of 15 to 300 μm. Structures: fibrous, granular, cubic, spherical. Base materials are cellulose pulp and chemically-modified microcrystalline cellulose. By changing these parameters we researched the influence on pore structure, pore size and pore volume. We used an alginate-modified gel casting process developed by Advanced Ceramics. Target of the study was to identify the advantages of cellulose particles in the production of ceramic catalysts and filter elements.

(ICACC-S10-P043-2017) The model of amorphisation in ceramic nanostructured electrolytes

V. Pervov[2]; A. Petrov*[1]; 1. Frumkin Institute of Physacal Chemistry and Electrochemistry RAS, Russian Federation; 2. Kurnakov Institute of General and Inorganic Chemistry RAS, Russian Federation

It is known that forming of transition structures of crystal intermediate between defective and amorphous states can enhance ionic conductance of ceramic compositional electrolytes by several orders of magnitude. It is also know that amorphization near interfaces in such structures results from incommensurability of substructures. This is observed in nanostructured materials and depends on synthesis conditions. On the other hand, nanoionics states that the reduction of grain size of electrochemically active structures must increase the ionic conductance because ion transfer distance decreases. We propose a theoretical model to be able to estimate amorphisation in nanostructured composite electrolytes and the extent to which it depends on unit cell volume and concentration of substructures. The model is based on two approaches. The first one is that the two-dimensional model is used, describing a binary system as jammed packing of incommensurate hard disks. The other approach is that the amorphization is to some degree the equivalent of molar excess volume, which is common in physics of metals. As the model is two-dimensional, the excess volume will be equal to excess area. Simulations were performed by means of molecular dynamics. Obtained results allows to conclude that amorphization depend on incommensurability and can reach the maximum value at equimolar concentration and incommensurability approximately 30%.

(ICACC-S10-P044-2017) First-principles calculations of high-efficiency abrasive grain for GaN chemical mechanical polishing

T. Igarashi*[1]; K. Kawaguchi[1]; Y. Ootani[1]; T. Nishimatsu[1]; Y. Higuchi[1]; N. Ozawa[1]; M. Kubo[1]; 1. Institute for Materials Reserch, Tohoku University, Japan

In order to propose a high-efficiency abrasive grain for GaN chemical mechanical polishing (CMP), we investigate the GaN CMP process via density functional theory method. Firstly, in order to reveal the effect of oxidation of a GaN substrate on an efficiency of the CMP process, we analyze the structural change in a bond between the SiO_2 abrasive grain and normal and oxidized substrates in the process of pulling up the abrasive grain. In the normal substrate, the bond between the substrate and the abrasive grain is dissociated and the surface Ga atom is not abstracted. In contrast, in the oxidized substrate, the Ga-N bond in the substrate is dissociated. These results suggest that the oxidation of the GaN surface enhances the CMP. Secondly, in order to reveal the effect of abrasive grain materials on the efficiency of the CMP process, we analyze the change of binding energy before and after the dissociation of Ga-N bond in the substrate with different abrasive grains. We investigate OH-terminated nano-diamond (ND) in addition to SiO_2. We find that the OH-terminated ND has larger negative value of the binding energy between the substrate and the abrasive grain after the dissociation of the Ga-N bond. This indicates that the OH-terminated ND induces the abstraction of the Ga atom. We conclude that the OH-terminated ND is more effective abrasive grain for GaN CMP than SiO_2.

(ICACC-S10-P045-2017) Tribochemical Reaction Dynamics of Carbon Nitride Thin Films by Tight-Binding Quantum Chemical Molecular Dynamics Simulations

M. Nakamura*[1]; S. Sato[2]; Y. Ootani[1]; Y. Higuchi[1]; N. Ozawa[1]; K. Adachi[2]; M. Kubo[1]; 1. Tohoku University, Japan; 2. Tohoku University, Japan

Recently, ceramic lubricants have gained much attention for realizing super-low friction in automotive engines, aerospace instruments, etc. One of the authors, Adachi, experimentally discovered that carbon nitride (CNx) gives super-low friction. However, the super-low mechanism of CNx has not been clarified experimentally. Especially the role of nitrogen seems to be very important, however its mechanism has not been elucidated. Therefore, we developed tribology simulator based on our tight-binding quantum chemical molecular dynamics method. We simulated the friction dynamics of H-terminated CNx by our simulator. The simulation result shows low friction coefficient of 0.05 under 1 GPa pressure. We noticed that hydrogen-hydrogen repulsion is source of the super-low friction. However, this result does not clarify the role of nitrogen. Then, we simulated the friction coefficient of H-terminated CNx under water vapor environments. It shows the generation and evaporation of NH3 molecules by the chemical reaction of CNx and water. We suggest that the generation and evaporation of NH3 molecules give low density diamond-like carbon thin films. Therefore, we propose that low density diamond-like carbon generated by the chemical reactions of CNx and water gives super-low friction.

(ICACC-S10-P047-2017) Unraveling Composition-Structure-Ionic Conductivity Relationships in Garnet-Type Solid Electrolytes Using Cheminformatics Approaches

N. Kireeva*[1]; V. Pervov[2]; 1. Frumkin Institute of Physical Chemistry and Electrochemistry RAS, Russian Federation; 2. Kurnakov institute of general and inorganic chemistry RAS, Russian Federation

The organic electrolytes of today's commercial rechargeable Li-ion batteries (LiBs) are flammable, toxic and have limited electrochemical energy window. All-solid-state batteries technology suggests improved safety, cycling performance, electrochemical stability, possibility to device miniaturization and enables a number of breakthrough technologies towards development of new high power and energy density all-solid-state microbatteries for electronics with low processing cost, solid oxide fuel cells, electrochromic devices and many others. Rational materials design experienced a boost that have resulted in a strong demand of methodology able to accelerate design of materials with tailored properties and cheminformatics can be used as the efficient tools in this respect. In this study, we have focused on the methodology: (i) to identify the parameters responsible for high Li-ion conductivity of the compounds related to garnet structured oxides and to transfer the knowledge to other structural types in order to develop "universal" descriptors; (ii) to develop quantitative models unraveling composition–structure–ionic conductivity relationships taking into account the details of sample preparation; (iii) to circumscribe the materials space of solid electrolytes attractive for virtual screening. This work has been supported by RFBR (project 14-29-04084)

(ICACC-S11-P046-2017) Effect of addition of friction modifiers on tribological behavior of semi-carbonized Cu/phenolic-derived semi-metallic friction material

C. Ju*[1]; H. Huang[1]; M. Hung[1]; C. Su[1]; H. Lin[1]; K. Lee[2]; J. Chern Lin[1]; 1. National Cheng-Kung University, Taiwan; 2. I-Shou University, Taiwan

The present presentation reports some effects of addition of friction modifiers (graphite, mica, and vermiculite) on the tribological behavior of a series of semi-carbonized copper/phenolic resin-derived semi-metallic friction materials. The materials used for the study were prepared by dry-mixing appropriate amounts of phenolic resin powder, pure copper fiber, pure copper powder, along with appropriate additions of graphite, mica, and/or vermiculite powders, followed by hot-pressing, post-curing and semi-carbonization. The tribological performance of the materials was evaluated using a disk-on-disk sliding wear tester at ambient temperature. Experimental results showed that graphite addition effectively lowered wear and noise level but at the same time reduced COF. Mica addition appeared less effective in lubrication than graphite in this study. The addition of vermiculite alone increased COF but also increased wear. The combination of graphite and vermiculite with an appropriate ratio demonstrated the best performance, which is better in friction, wear and especially noise level than a commercial high temperature-sintered metallic system for bike brake application. The research is supported by the Ministry of Science and Technology of Taiwan, ROC under the research grants MOST 102-2221-E-006-059 and MOST 105-2221-E-006-046.

(ICACC-S11-P048-2017) Reactive Texturing of Y-TZP and Ce-TZP in Very High Magnetic Field

O. Van der Biest*[1]; D. Vriami[1]; E. Beaugnon[2]; 1. K U Leuven, Belgium; 2. Laboratoire National des Champs Magnétiques Intenses, France

Tetragonal zirconia powder cannot be aligned by applying a strong magnetic field during green forming of a powder compact. In prior work we have shown that a strong texture can be achieved in 3Y-TZP by using a reactive texturing technique. One uses a suspension that contains monoclinic zirconia that can be oriented in a strong field during green forming for instance by slip casting. The monoclinic (100) plane is oriented perpendicular to the magnetic field direction. When a mixture of pure monoclinic zirconia powder and 8 mol% yttria co-precipitated zirconia is used, a strong texture of single phase 3Y-TZP is obtained with the (001) plane of the tetragonal phase perpendicular to the magnetic field direction. In the strong magnetic field of 17.4 T used in the present work, the texture of the cast green ceramic, can be measured (Lotgering factor f = 0.15) whereas at lower fields texture in the green body is barely discernible. After sintering a texture index of 32.5 is obtained. We attempted to texture Ce-TZP by a similar strategy but with less success. The suspension consisted of a mixture of monoclinic zirconia and nanometric ceria powder. After reactive sintering a texture index of about 3 was measured. It appears that for successful reactive texturing of zirconia the differences in composition between the monoclinic phase and the phase containing the stabilizing element should be as small as possible.

(ICACC-S11-P049-2017) The influence of process temperature on the nitriding behavior of austenitic stainless steel

H. Park*[1]; K. Moon[1]; 1. KITECH, Republic of Korea

Austenitic stainless steels are most popular materials in the stainless steel family used in various applications due to their excellent corrosion resistance and good forming characteristics.. Nevertheless, this type of materials has low hardness as well as poor wear resistance due to the inherent austenitic structure. To overcome this problem, efforts have been made to improve their surface hardness and wear resistance such as gas nitriding, salt bath nitriding and ion nitriding. Among them, it is well known that plasma nitriding process is the highest efficient method than others. In this method, especially, nitrogen supersaturated(S-phase) layer without reducing its corrosion resistance was performed at process temperature below the 723K. Otherwise, CrN layer was formed on the its surface during nitriding temperature over the 723K resulting in deterioration of corrosion resistance. Therefore, It has disadvantage the process time is too long in order to form thick S-phase layer. So, many researchers are studying to form thick S-phase layer during short process time.

(ICACC-S11-P050-2017) Mechanical Properties of Duplex plasma nitriding/DLC film on AISI4140 steel

H. Park*[1]; M. Kim[1]; K. Moon[1]; 1. KITECH, Republic of Korea

For engineering applications, diamond-like carbon films (DLC) are the most suitable coatings if high wear resistance and low friction are needed. However, one problem is their very poor adhesion on steel substrates when no intermediate layer is supplied. In this study, duplex plasma nitriding/diamond like carbon (DLC) coating have been deposited on AISI 4140 steel by a PECVD equipment with a unipolar bias plasma generator. In particular, the effects of the existence of compound layer as the buffer layer of DLC coatings have been evaluated. Especially, plasma nitriding and PECVD process for DLC coatings have been deposited subsequently in a same chamber. So, the parameters for DLC coatings are very similar to those of the well-known and industrialized plasma-nitriding process. But the temperature of DLC coating have been changed ranges from 0 to 450°C. The effect of an intermediate SiC layer prepared with tetramethylsilane (TMS) as a precursor has also been investigated for the adhesion of DLC coatings. The results of the mechanical property test showed that nitriding of the AISI 4140 steels improves the adhesion and the wear resistance of DLC coating as compared to as-hardened substrate.

(ICACC-S11-P051-2017) Effects of Si Powder Characteristics on Sintered Reaction-Bonded Silicon Nitride (SRBSN) Ceramics with High Thermal Conductivity

H. Oh*[1]; H. Lee[1]; 1. Kumoh National Intitute of Technology, Republic of Korea

Recently, Si_3N_4 ceramics have fascinated many researchers as a promising substrate materials for power device due to its high thermal conductivity, mechanical and chemical stability. The highly thermal conductive Si_3N_4 ceramics are, in general, fabricated via reaction-bonding of Si and post-sintering (SRBSN). It is well-known that characteristics of the starting Si powder, i.e., purity as well as the particle size and its distribution, play an important role in processing and characteristics of SRBSN ceramics. In this study, Si scrap waste from semiconductor industry was employed as a starting material for economical consideration. After careful attrition millings of the coarse Si scrap waste, the fine Si powders were prepared, which showed average particle sizes of ~ 1micron with different size distributions according to the milling parameters. After hydraulic forming and nitridation, SRBSN ceramics were prepared by post-sintering at 1900 degrees celsius for 6 h under a nitrogen pressure of 1 MPa. Thermal conductivity of SRBSN ceramics were affected by particle size distributions of the milled Si powder. Characterization of the milled Si powders, RBSN and SRBSN ceramics were conducted through particle size analysis, scanning electron microscopy, x-ray diffraction, ICP, O analyzer, and thermal conductivity measurement.

(ICACC-S12-P052-2017) Oxidation behavior of a $Mo(Si,Al)_2$ composite at 1700°C in low aO_2 environment

K. M. Hellström[1]; E. O. Strom*[2]; 1. Swerea IVF, Sweden; 2. Sandvik Heating Technology, Sweden

$MoSi_2$-based materials are commonly used as furnace heating elements because of their high temperature oxidation resistance, low density, high melting point, and electrical and thermal conductivity. Although $MoSi_2$ heating elements form a protective silica scale during heat treatment at high temperatures, the material cannot be used in reducing environments due to the formation of volatile SiO. The problems associated with SiO evaporation can be overcome by substituting parts of the Si with Al, yielding a $Mo(Si,Al)_2$ composite. It is known that this material forms a stable and adherent alumina scale and can be used in both oxidizing and reducing atmospheres up to approximately 1600°C. In this study it was examined if 1600°C really is the temperature limit for these materials. A commercial heating element Kanthal Super ER ($Mo(Si,Al)_2$-based composite) was exposed at 1700°C for 1 h up to two weeks in reducing atmosphere (95% N_2 + 5% H_2). The aim was to investigate the oxide formation and stability at low oxygen partial pressures as well as the impact of potential nitride formation on oxidation behaviour of $Mo(Si,Al)_2$-based materials. After exposure, the samples were investigated with XRD, SEM and EDX. It was found that AlN is formed during the initial oxidation stages, this layer is transformed to alumina after longer exposure times.

(ICACC-S12-P053-2017) Synthesis and evaluation of oxygen-free precursor for SiC/ZrC composite ceramics

Y. Tian*[2]; M. Ge[1]; S. Yu[1]; X. Lv[2]; W. Zhang[1]; 1. Institute of Process Engineering/CAS, China; 2. University of Chinese Academy of Sciences, China

A novel polymeric precursor for SiC-ZrC composite ceramics was synthesized by dimethyldichlorosilane, Zirconocene dichloride and sodium in one-pot. The pyrolysis behavior and structural evolution of the precursor, the microstructure and composition of the synthesized ceramics were fully investigated. The results showed that the precursor can completely be transformed into SiC and ZrC after heat treatment in argon at temperatures above 1100°C, with ceramic yields about 67%. The obtained ceramic consisted mainly of amorphous matrix phase SiC, in which ZrC nanocrystallites were distributed uniformly with 10-20 nm in size. This new precursor is suitable for production of ultra-high temperature ceramic fiber and composite matrix.

(ICACC-S12-P054-2017) Improved Conductive Coatings for High Resolution Quantitative Analysis of UHTCs

K. Shugart*[1]; K. Crispin[2]; P. Gopon[3]; 1. UES, Inc., USA; 2. The Pennsylvania State University, USA; 3. University of Wisconsin-Madison, USA

Scanning electron microscopy, energy dispersive spectroscopy, and wavelength dispersive spectroscopy are all essential techniques for characterization of Ultrahigh Temperature Ceramics (UHTCs.) However, due to the insulating nature of many ceramics and their oxides, these techniques can be challenging to effectively perform. Many use carbon, gold, or gold-palladium coatings to increase the conductivity of their samples. This study, which includes iridium as an alternative source, sought to optimize the thickness of coatings on insulating samples (e.g. UHTCs). The goal is to improve spatial resolution of imaging, while decreasing the impact on quantitative microanalysis. Coatings applied using sputter deposition were evaluated via a combination of scanning electron microscopy, atomic force microscopy, transmission electron microscopy, and electron probe microanalysis. Iridium has been found to have two major advantages: (i) providing reasonable conductivity at lower thicknesses than gold or gold-palladium and (ii) allowing for quantitative analysis of carbon contained within a ceramic sample.

(ICACC-S12-P055-2017) Ceramic Coatings on the Zr Cladding and Their Corrosion Behavior in Steam at High Temperature

J. Zhou*[1]; 1. Ningbo Institute of Industrial Technology, Chinese Academy of Science, China

MAX phase coating on the Zircaloy cladding has been regarded as a promising approach to enhance the accident tolerant capability of nuclear fuel assembly after the Fukushima nuclear accident in 2011. Herein dense Ti_2AlN and Ti_2AlC coatings with good adhesion properties were obtained through reactive cathodic arc deposition process accompanied by subsequent vacuum annealing treatment. Moreover, an additional layer of dense AlTiSiN hard coating is proposed to reduce the pre-existing defects within the coatings. Finally, the high temperature steam corrosion behavior of Zr-2.5Nb substrates with and without coating was investigated up to 1000°C. It is shown that Ti_2AlC-coated Zr-2.5Nb display slower oxidation rate in the 700-1000°C temperature range, but pre-existing defects acted as short-circuit diffusion path and local interface oxidation occurred at 900-1000°C. Bilayer Ti_2AlC-AlTiSiN coatings exhibited much better oxidation resistance, and the formation of a protective oxide film can effectively prevent further oxidation of the bare Zr-2.5Nb, whereas, the ZrO_2 layer of about 130 micron was observed on the Zr-2.5Nb surface after oxidized at 1000°C for 1800s in steam. This result demonstrates the effectiveness of MAX phase ceramic coatings with fine controlled microstructure prepared by PVD method as a corrosion barrier layer to resist the high-temperature steam oxidation of Zircaloy.

(ICACC-S14-P056-2017) Fabrication of nitride phosphor particle dispersed transparent α-SiAlON ceramic composites

I. Kokubun*[1]; J. Tatami[1]; M. Iijima[1]; T. Takuma[2]; M. Yokouchi[3]; 1. Yokohama National University, Japan; 2. Kanagawa Academy of Science and Technology, Japan; 3. Kanagawa Industrial Technology Center, Japan

Phosphor particles dispersed in a resin were used in conventional white LEDs. The resin is easily degraded by heating during operation, resulting in the decrease in the lifetime of the white LEDs. Using transparent and fluorescent bulk ceramics instead of the particle / resin composite are one of the ideas to solve the problem. Because a-SiAlON is essentially transparent under visible light, transparent a-SiAlON ceramics can be obtained by removing the source of absorption and scattering of light. In this study, nitride phosphor particles were dispersed in Y- and Ca-α SiAlON ceramics. The dispersed phosphor used in this study was β SiAlON: Eu^{2+} and Ca-α SiAlON:Eu^{2+}. As a result of XRD analysis, a-SiAlON and trace β SiAlON were detected. Use of homogeneous green body and limited grain growth by low temperature sintering caused the high transparency. Moreover, they showed green or yellow color emission resulted from nitride phosphor particles under visible light. The luminescence property resulted from inhibition of the reaction between the raw materials and the nitride phosphor by low firing temperature for densification.

(ICACC-S14-P057-2017) Insight into orange persistent luminescence mechanism in $Ca_3Si_2O_7:Eu^{2+}-Ln^{3+}$ (Ln=Sm, Tm) based on vacuum referred binding energy diagram

R. Maki[1]; J. Ueda*[1]; S. Tanabe[1]; 1. Kyoto University, Japan

Eu^{2+}-doped persistent phosphors such as $SrAl_2O_4:Eu^{2+}-Dy^{3+}$ (green) and $CaAl_2O_4:Eu^{2+}-Nd^{3+}$ (blue) have been successfully commercialized for luminous painting. However, there is still lack of warm-color persistent phosphors with bright and long persistent luminescence (PersL) compared to the commercial green and blue persistent phosphors. In this paper, based on the 4f energy levels of lanthanide ions in the vacuum referred binding energy (VRBE) diagram of the $Ca_3Si_2O_7$ (CSO) host, two potential candidate ions (Sm^{3+} and Tm^{3+}) were selected as electron traps to induce orange PersL at room temperature in the CSO:Eu^{2+} phosphor. Both of CSO:Eu-Sm and CSO:Eu-Tm show orange PersL peaked around 600nm due to Eu^{2+}:4f-5d at room temperature. In the PersL decay curves monitoring orange emission, CSO:Eu-Tm has strong initial intensity just after ceasing excitation light than CSO:Eu-Sm by approximately 50 times and PersL decay rate for CSO:Eu-Tm is faster than CSO:Eu-Sm. From the thermoluminescence (TL) glow curves of CSO:Eu-Sm and CSO:Eu-Tm, TL glow peak temperatures for CSO:Eu-Tm and CSO:Eu-Sm are 215 K and 443 K, and the trap depths are estimated to be respectively 0.54 eV and 1.14 eV. This result is in good agreement with the values from the VRBE diagram (0.68 eV for CSO:Eu-Tm and 1.15 eV for CSO:Eu-Sm).

(ICACC-S14-P058-2017) Near Infrared Emitting Scintillators for Remote and Online Measurements based on Optical Fibre Coupled Configuration

G. Okada*[1]; N. Kawaguchi[1]; T. Yanagida[1]; 1. Nara Institute of Science and Technology, Japan

There has been increasing demands of online large-dose measurements particularly in the fields of radiation therapies and nuclear power plants. During such radiation therapies, patients are exposed to large-dose radiations, which must be accurately measured on-line and remotely. Also, such measurements are needed in, or close to, a nuclear reactor, or even during a demolition work after nuclear power plant accidents. In this paper, we propose an approach for remote and online measurements using near-infrared (NIR) emitting scintillators. In this approach, a scintillator is coupled with an optical fibre, which guides the scintillation light to photo-detector over a long distance such that the measurement operators do not experience radiation exposure and it does not require any power source to run the measurement system at the measurement site.

Here, we use a scintillator emitting NIR light so that Cherenkov radiation, which appears in the UV-blue region, can be easily distinguished and the propagated scintillation light is not absorbed by radiation-induced colour centres, which appears in the same region. For preliminary testing of principles, rare-earth doped garnet crystals are used.

(ICACC-S14-P059-2017) Synthesis and processing of CaF_2 and ZnS nanocomposites for IR transparent windows

N. Ku*[1]; V. L. Blair[1]; 1. U.S. Army Research Laboratory, USA

Zinc sulfide is currently a common material for use in IR transparent missile domes. While exhibiting high transmission, the material has a relatively low strength, which is disadvantageous for missile dome windows. Past work has shown that by creating a nanocomposite ceramic system, the strength of bulk material can be increased while minimally affecting the optical properties. This work will investigate the synthesis and processing of a CaF_2 and ZnS nanocomposites. The synthesis method will consist of titrating ZnS onto dispersed CaF_2 particles to create an intimately mixed, nanoscale starting powder for processing into a bulk ceramic. The ratio of CaF_2 to ZnS will be varied to optimize the properties of the final, sintered nanocomposite.

(ICACC-S14-P060-2017) Degradation mechanism of piezoelectric materials

H. Sabarou*[1]; Y. Zhong[1]; 1. Florida International University, USA

The disappearance of polarization hysteresis characteristics occurs for piezoelectric (PZT) materials so that the performance of the materials is being degraded. To avoid this problem, finding the origin of this issue is necessary. This research approaches to the problem from a new aspect for the first time and mainly focuses on perovskite structure of PZT materials. The several tests have been designed to scrutinize the structural evolution of perovskite under various oxygen partial pressures and temperatures and to simulate working conditions of PZT materials. PZT pellets have been annealed under different temperatures and oxygen partial pressures. The evolution of crystal structure has been studied via XRD. Thermal analysis tests also exhibit changes in perovskite structure and truly justify the degradation in polarization characteristics of PZT materials. The mechanism of the structural evolution is discussed and promoted to real PZT applications.

(ICACC-S14-P061-2017) Thermodynamic study on PMN-PT single crystals

H. Sabarou*[1]; Y. Zhong[1]; 1. Florida International University, USA

A new thermodynamic approach on the structural evolution of $Pb(Mg_{1/3}Nb_{2/3})O_3$-$xPbTiO_3$ (PMN-PT) single crystal has been presented, and it is used to justify the inconsistency of the ferroelectric properties of single-crystal perovskite. A series of experimental tests under different temperatures and oxygen partial pressures has been applied to simulate the required working conditions of these materials. The phase stability and structural changes have been scrutinized via X-ray diffraction and thermogravimetric methods. The involved mechanisms for crystal symmetry changes and stabilizing perovskite structure clearly explain the degradation of the piezoelectric properties of the single crystals and competently match with their electric polarization measurements. The thermodynamic approach discusses the origin of the polarization hysteresis degradation and provides conditions to save ferroelectric properties.

(ICACC-S14-P062-2017) Development of glass-cladded single-crystalline LiNbO3 optical fiber

J. Wang*[1]; 1. National Sun Yat-Sen University, Taiwan

In contrast to widely popular adaption of planar LiNbO3 waveguide for electro-optic (EO) applications, this study is to aiming to development glass-cladded single-crystalline electro-optic (EO) LiNbO3 fiber with electrodes embedded between core and cladding. One-step process by CO2 laser heated pedestal growth (CO2-LHPG) technique is the ultimate goal. This means fiber waveguide formation (core-cladding) and electrodes can be made in one drawing step without any other post processing. To archive this, quite a few important material and process issues have to address. A half-wave voltage (Vπ), effective electro-optic coefficient, and transmission loss as function of cladding glass composition, electrode, CO2-LHPG processing parameters (heating zone temperature, drawing speed) few-mode versus single mode LiNbO3 fiber will be discussed. Here, cladding glass compositions to be studied include SiO2-based glasses since softening temperature and viscosity limitations imposed from core crystal LiNbO3 and electrode selections include ITO and ZnO. A technical performance comparison of this new type of LiNbO3 EO fiber against conventional LiNbO3 planar waveguide and its new potential applications will be also briefly presented.

(ICACC-S14-P063-2017) Optical and conducting properties of MnF_2 containing oxyfluoride borosilicate glasses

S. S. Danewalia*[1]; N. Gupta[1]; S. Aggarwal[1]; G. Sharma[1]; K. Singh[1]; 1. Thapar University, Patiala, India

Borosilicate oxyfluoride glasses with variable amount of CaO and MnF_2 are prepared by melt-quench technique. The optical parameters of the glasses are obtained using UV-visible spectroscopy. The optical band gap, Urbach energy, refractive index, average molar reflection etc. are calculated from the UV-visible spectroscopic data. Optical band gap of the glasses lies in the insulator range, which increases with the MnF2 content. Replacement of CaO by MnF2 increases the microhardness of the glasses. The conductivity of these glasses is observed using impedance analysis. Mixed bonding might create localization effect which prevents the moments of the ions.

(ICACC-S14-P064-2017) Processing of Doped Hafnia Ceramics for Fundamental Structure Studies

M. Kasper*[1]; B. Johnson[1]; S. Jones[1]; C. Chung[1]; J. Jones[1]; 1. North Carolina State University, USA

HfO2 thin films have been shown in prior work to exhibit ferroelectric behavior when doped with certain elements (e.g., Si, Y, Zr, and Gd). In our group's prior work, we studied Si-doped HfO_2 powders and bulk ceramics in order to understand details about the structure and phase stability of these systems. The use of bulk materials for these structure investigations enables better understanding than currently available through thin film structure characterization. In the present work, we aim to understand fundamentals about the structure and phase stability of other doped systems including La (0.5-7%), Y (1-50%), and Gd (1-9%). The methods employed include solid-state calcination, sintering, density measurements, and X-ray diffraction (XRD) characterization. Different calcination temperatures were tested in order to examine the influence of calcination temperature on the developed phases. To further explore the phase evolution, the materials were also measured as a function of temperature in real time using in situ XRD. Y- and Gd-doped samples showed both cubic and monoclinic phases, whereas La-doped samples only show a monoclinic phase. These results are consistent with their phase diagrams. The samples, part of two undergraduate research projects, will be further used for electrical characterization and structure studies (for example, Rietveld refinement).

(ICACC-S14-P065-2017) A IGZO based p-type metal oxide semiconductor

T. Chou*[1]; S. Chiou[1]; T. Huang[1]; K. Chiu[1]; 1. Industrial Technology Research Institute, Taiwan

In this research, p-type IGZO:Ca films are successfully synthesized. First, Vienna Ab-initio Simulation Package is involved in the calculation of the variation of density of states to energy in the IGZO-based metal oxide semiconductor materials doped with different dopants. Through the simulation and calculation procedures, it is known that the Fermi level of the Ca-doped IGZO-based metal oxide semiconductor material shifts to the valence band, therefore the IGZO:Ca metal oxide semiconductor material is a p-type material. Second, the pure crystalline sputtering target of $In_{1-x}Ga_{1-y}Ca_{x+y}ZnO_{4+m}$ with 0<x+y%0.1, 0%m%3, and 0<x, 0%y, or 0%x, 0<y is fabricated using the powder synthesized by the thermal decomposition of a metal–nitrate–tartrate gel complex at 600°C. The complex is prepared by dissolving nitrates of indium, gallium, calcium and zinc in an aqueous solution with nitric and tartaric acids. The resulting compacts, which can be densified at 1350°C, exhibit a decreasing trend of electrical resistivity with increasing Ca content. The hole carrier concentration of the crystalline material is in a range of $1x10^{15}$ ~ $6x10^{19}$ cm^{-3}. P-type IGZO:Ca films have been grown by magnetron RF sputtering method. It shows that the proposed p-type film has the stable electrical characteristics: hole-density of $5x10^{12}$ cm^{-3}, hall mobility of 10 cm^2/Vs, and the resistivity of 1 x $10^4\Omega$-cm when RF power is 70 W, and Ar/O2% is 0.001025%.

(ICACC-S14-P066-2017) $MHfO_3$ (M = Ca, Sr) as Novel Host Lattices for Eu^{2+} Ions

S. Krüger*[1]; C. Wickleder[1]; 1. University Siegen, Germany

The luminescence of Eu^{2+} ions is extremely efficient due to their parity-allowed 5d-4f transitions and is therefore well investigated and reported in many different crystalline and nanostructured host lattices. Another benefit of Eu^{2+} is the possibility of tuning the emission colour by variation of the host composition because of the huge impact of the local surrounding on the position of the 5d orbitals. For applications as LED phosphors, there is, however, a lack of red emitting materials with high luminescent efficiency and long-time stability of the materials. In this work, we introduce alkaline earth hafnates doped with Eu^{2+} ions for the first time. They were prepared by solid state synthesis from the binary oxides HfO_2, SrO and EuO in tantalum containers. Due to the air stability of the materials and their emission bands located at low energies such as 631 nm ($SrHfO_3:Eu^{2+}$) and 660 nm ($CaHfO_3:Eu^{2+}$), they are very well suited for future LED phosphors. The red shift of the emission band of $CaHfO_3:Eu^{2+}$ compared to that of the respective Sr compound can be explained by the larger crystal field splitting of the 5d states. Also the position of the excitation bands of these materials are interesting for developing new LEDs. The red emission can be excited by blue light which is the main excitation energy for phosphors in many common two phosphor converted LEDs. This is an essential point for future energy saving lamps.

(ICACC-S15-P067-2017) On the design of novel polymer matrix composites (PMCs) by Streolithography (SL)

D. Blue[1]; E. Kramer[1]; L. Yutrzenka[1]; R. Larson*[1]; R. Dunnigan[1]; S. Gupta[1]; 1. University of North Dakota, USA

This poster reports the synthesis and characterization of novel particulate reinforced polymer matrix composites by 3D printing (3DP). Novel Particulate-Photopolymer Resin composites were designed by adding different particulates in the Resin matrix. Microstructure studies by SEM studies were performed to understand the dispersion of these particulates in the resin matrix. Tensile testings and tribological behavior study by pin-on-disk method were performed to understand the mechanical and tribological behavior of these solids.

(ICACC-S15-P068-2017) Rheological Modifications of Sanitaryware Slurries for Additive Manufacturing

E. I. Tanisan*[1]; S. Tarhan[2]; F. Kara[1]; 1. Anadolu University, Turkey; 2. Kaleseramik, Turkey

Complex and large sanitaryware articles have been produced by slip casting method which is a cost effective method for volume production of such shapes. However, introduction of new designs of sanitarware articles is a costly and time consuming process. On the other hand, additive manufacturing of ceramic articles have been gaining increasing importance. This method presents making almost any shapes without moulds with unlimited design possibilities. It can, not only avoid costly and time consuming design process, but also may be a viable production process for customized production of the sanitarware articles. In this study, clay based sanitaryware slurries were modified by various additives as a way to get inks for additive manfacturing of sanitaryware articles, such as washbasin. Effect of some flocculating agents and organic molecules on the rheological properties of the ink and their deposition behaviour will be reported.

Wednesday, January 25, 2017

3rd Pacific Rim Engineering Ceramics Summit

Energy and Environmental Issues II

Room: Coquina Salon C
Session Chairs: Ziqi Sun, Queensland University of Technology; Zoltan Lences, Institute of Inorganoc Chemistry, Slovak Academy of Sciences

8:30 AM

(ICACC-PACRIM-021-2017) Ultrathin 2D metal oxide nanosheets for sustainable applications (Invited)

Z. Sun*[1]; 1. Queensland University of Technology, Australia

Two-dimensional (2D) transition metal oxide systems present exotic electronic properties and high specific surface areas, and also demonstrate promising applications ranging from electronics to energy storage. Yet, in contrast to other types of nanostructures, the question as to whether we could assemble 2D nanomaterials with an atomic thickness from molecules in a general way, which would give them some amazing properties such as those of graphene, still remains unresolved. In this presentation, we will report a generalized and fundamental approach to molecular self-assembly synthesis of ultrathin 2D nanosheets of transition metal oxides by rationally employing lamellar reverse micelles. It is worth emphasizing that the synthesized crystallized ultrathin transition metal oxide nanosheets possess confined thickness, high specific surface area, and chemically reactive facets, so that they presented promising applications in nanostructured electronics, photonics, sensors, and energy conversion and storage devices.

9:00 AM

(ICACC-PACRIM-022-2017) Towards Solid Oxide Fuel Cells (SOFCs) with Proton Conducting Electrolytes Operating at Low Temperature: Is the Scale-up Feasible? (Invited)

E. Traversa*[1]; 1. Xi'an Jiaotong University, China

Solid oxide fuel cells (SOFCs) are an efficient and environmental-friendly alternative to combustion engines for supplying power. There is at present a keen interest on SOFCs in China with the government supporting a number of commercialization initiatives with large grants. However, their high operating temperatures and cost hampers their broad use and causes long-term stability problems. A step forward towards reducing the SOFC working temperature at 600°C or below can be the use of high temperature proton conductor (HTPC) oxides as electrolytes, due to their lower activation energy for proton conduction (0.3-0.6 eV), with respect to oxygen-ion conducting electrolytes. We have recently made significant progresses in the development of chemically-stable HTPC electrolytes and cathode materials associated, by using Y-doped barium zirconate (BZY). In this talk, after an introduction reporting an overview of the state-of-the-art of SOFC research in China, the recent work performed in our group toward the prospective commercialization of SOFC based on chemically stable proton conducting electrolytes operating at 600°C will be presented.

9:30 AM

(ICACC-PACRIM-023-2017) Computational Design of High Performance Solid Oxide Fuel Cell Materials (Invited)

H. Kwon[1]; J. Han*[1]; 1. University of Seoul, Republic of Korea

Solid oxide fuel cells (SOFCs) operated at high temperature have received much interests as prospective device due to their high efficiency and fuel flexibility. There are, however, several critical issues for enhancing the SOFC performance. First, the rate of reduction, oxidation or transport of oxygen drastically deteriorates at reduced temperature. Second, severe degradation observed in electrode affects long-term stability in negative ways. To resolve these problems, it is essential to rationally develop the materials of SOFC components. Unfortunately, it is not easy to completely achieve it by depending only on traditional experimental methods. Here, we introduce computational approaches to design SOFC materials mainly based on density functional theory (DFT) calculations. Our results provide mechanistic information required to understand surface reactions and discover the promising potential materials. We believe that our computational study will play an important role in improving SOFC performance by guiding or complementing the relevant experiments.

10:20 AM

(ICACC-PACRIM-024-2017) Experimental and Theoretical Approach to Lanthanide-Doped $LaSi_3N_5$ Phosphors (Invited)

Z. Lences*[1]; I. Ibrahim[1]; P. Sajgalik[1]; L. Benco[2]; 1. Institute of Inorganoc Chemistry, Slovak Academy of Sciences, Slovakia; 2. University of Vienna, Austria

Lanthanide (Ln) doped $LaSi_3N_5$ based phosphors were prepared experimentally and their electronic structure and band gaps were calculated using the screened Coulomb hybrid HSE06 functional. The synthesized $LaSi_3N_5$:Ce phosphor emits violet-blue light, $LaSi_3N_5$:Eu – green-yellow and $LaSi_3N_5$:Sm red light. The calculations showed that with increasing atomic number the band gap of Ln^{3+}-doped $LaSi_3N_5$ smoothly decreases from 4.6 eV (Ce) to 0.5 eV (Eu). The band gap of Ln^{2+}-doped $LaSi_3N_5$ increases from 0.80 eV (Ce, Pr) to 0.95 eV (Nd, Pm), 1.43 eV (Sm), and to 3.28 eV (Eu). The electronic transition in Ln^{2+}-doped LaSi3N5 has 4f-5d character. Because the Ln dopants were experimentally added mainly in a form of oxides, also the influence of N/O substitution on the band gap of $LaSi_3N_5$ was calculated. The results showed that with increasing oxygen content in the cell of $LaSi_3N_5$ the band gaps decreased by 0.2 – 0.6 eV. Some of the lanthanides were added to $LaSi_3N_5$ also in the form of fluorides and their photoluminescent properties were compared with Ln_2O_3-doped phosphors. The energy level locations were constructed from the ab initio calculated electronic structures and compared with the empirical energy level diagram. There is a good agreement between calculated and experimental data and the method can be used for phosphor material design.

10:50 AM

(ICACC-PACRIM-025-2017) Recent development and properties of white silicon oxycarbides derived from polymer precursors in a hydrogen atmosphere (Invited)

M. Narisawa*[1]; K. Sasakawa[1]; S. Takeuchi[1]; H. Inoue[1]; 1. Osaka Prefecture University, Japan

Silicon oxycarbide (SiOC) synthesized by polymer precursor method has been called “black glass”. Due to the incorporation of excess carbon in amorphous network, its black appearance was thought to be intrinsic nature of the synthesized material. Recently, however, some SiOC ceramics with transparency was successfully synthesized and began to attract wide attentions. Our group in Osaka Prefecture University used hydrogen atmosphere to pyrolyze precursors, and found that some of the precursors accepted decarbonization effectively. These obtained Si-O-C(-H) ceramics exhibited not only the transparency but also unique photoluminescence under UV irradiation. The concentration of dangling bonds in the white ceramics was also diminished as compared with the black one which was derived from the same precursor in an Ar gas flow. In the talk, effects of high temperatures on the properties and structures of Si-O-C(-H) ceramics will be described from viewpoints of experiments and thermodynamic calculations. Studies on synthesizing various forms of Si-O-C(-H), like fibers and hot-pressed dense monolith, will be also described.

11:20 AM

(ICACC-PACRIM-044-2017) Updated Composite Materials Handbook-17 (CMH-17) Volume 5 — Ceramic Matrix Composites

R. Andrulonis*[1]; 1. Wichita State University, USA

A wide range of issues must be addressed during the process of certifying CMC (ceramic matrix composite) components for use in commercial aircraft. The Composite Materials Handbook-17, Volume 5, Revision A on ceramic matrix composites has just been revised to help support FAA certification of CMCs for elevated temperature applications. The handbook supports the development and use of CMCs through publishing and maintaining proven, reliable engineering information and standards that have been thoroughly reviewed. Volume 5 contains detailed sections describing CMC materials/processing, design/analysis guidelines, testing procedures, and data analysis and acceptance. A review of the content of this latest revision will be presented along with a description of how CMH-17, Volume 5 could be used by the FAA (Federal Aviation Administration) and others in the future.

6th Global Young Investigator Forum

Special topic: Professional Development for the Early-career Ceramic Engineer

Room: Coquina Salon F
Session Chair: Kathleen Shugart, UES, Inc.

8:30 AM

(ICACC-GYIF-028-2017) Critical Business Knowledge for the Early-career Ceramic Engineer (Invited)

K. Brosnan*[1]; 1. GE Global Research, USA

not available

Special topic: Networking for Early-Career Ceramic Engineers

Room: Coquina Salon F
Session Chair: Jesse Angle, Exponent

10:20 AM

(ICACC-GYIF-029-2017) Professional Networks: Who? What? Why?

K. Shugart*[1]; 1. UES, Inc., USA

One piece of advice commonly given to students and recent graduates is to network, network, network. But why? And with whom? While in school, professional societies are easy to find and cheap to join. Many societies have established networking groups specifically designed for students and for young professionals and these can be very helpful. As an example, the American Ceramic Society has the President’s Council of Student Advisors (PCSA), the Global Graduate Research Network (GGRN), and the Young Professionals Network (YPN). Joining groups like these can aid in making connections and staying involved with the overall society. They also provide opportunities for professional development training and create a technical network for members. This talk will aim to generate discussion on the advantages of professional society membership and professional networks and how to get the most benefit from membership. It will also compare and contrast the various ceramics-oriented societies open to students and young professionals.

10:40 AM

(ICACC-GYIF-030-2017) Dreaming of a career in science: What comes first - network or mobility?

E. Hemmer*[1]; 1. University of Ottawa, Canada

Mobility and networks are two aspects that are considered as essential for the development of a scientific career in our days. But what does this mean? Do we have to be mobile in order to build up our network? Or do we need a strong network in order to become mobile? This presentation will provide a snapshot on the journey of a materials scientist from Germany moving via Japan to Canada. In the luggage: multifunctional lanthanides in molecules and nanomaterials. Some experience from the time being a PhD student, from the period discovering new countries and research areas as a postdoctoral fellow, and the most recent ongoing adventure of starting an independent research group will be shared with the audience. While the search for the ultimate recipe for the straight forward achievement of a scientific career is still going on, some of the basics that may be required for a scientific journey by any young researcher or scientist will be discussed. These basics clearly include curiosity, self-motivation and an open mind, not to forget about endurance as well frustration tolerance, while taking pleasure in what we do must always be kept in sight. (Disclaimer: Unfortunately, the author won't be able to provide an all-inclusive-package for success in science. Yet, discussion of innovative approaches will be most welcome.)

FS2: Advanced Ceramic Materials and Processing for Photonics and Energy

Photonics, Electronics and Sensing I

Room: Halifax A/B

Session Chairs: Riad Nechache, École de technologie supérieure (ETS); Peter Mascher, McMaster University

8:30 AM

(ICACC-FS2- 019- 2017) Processing and Optical Properties of Semiconductor Core Optical Fibers

M. Ordu[1]; J. Guo[1]; B. Tai[1]; S. Erramilli[1]; S. Ramachandran[1]; S. Basu*[1]; 1. Boston University, USA

Semiconductor core optical fibers have many applications such as chemical sensing and biomedical surgery. Borosilicate glass has a good coefficient of thermal expansion (CTE) match with Ge, which itself has good mid-IR transmission properties. In this study, mid-IR fibers were drawn by the 'rod in tube' method at 1000°C using a laboratory-made mini draw tower. Scanning/transmission electron microscopy (S/TEM) based energy dispersive x-ray (EDX) spectroscopy analysis showed minimal diffusion of oxygen and silicon from cladding to the core. Infrared transmission through the fiber was characterized by a Quantum Cascade Laser (QCL), showing transmission losses in the 3-8 dB/cm range in the spectral range of 5.75-6.3 μm. To the best of our knowledge, these are the first drawn mid-IR transmitting Ge-core fibers with borosilicate glass cladding.

8:50 AM

(ICACC-FS2- 020- 2017) Comparison of optical properties of CsPbBr3 and CsPb2Br5 (Invited)

J. Bao*[1]; 1. University of Houston, USA

Inspired by organic-inorganic hybrid perovskites, Cs-based perovskites have also been synthesized and have attarcted a lot of attention because of their high fluorescence efficiency. CsPbBr3 and CsPb2Br5 have different crystal structures, but both show a similar bandgap at visible wavelength, and have been used to demonstrate bright light emitting diodes. In this talk, I will present a detailed study of their chemical stability, structural, electronic and optical properties.

9:20 AM

(ICACC-FS2- 021- 2017) Rare Earth Doped Silicon-based Nanostructures and Their Role in Silicon Photonics (Invited)

P. Mascher*[1]; Z. Khatami[1]; J. Miller[1]; J. Wojcik[1]; 1. McMaster University, Canada

The possible application of luminescent Si-based materials for solid-state lighting (SSL) has emerged as an interesting area of research as it would offer substantial advantages in terms of cost and manufacturability. In order for Si-based materials to be used in SSL schemes it is necessary to have precise control of the emission from these materials. This can be accomplished through the use of rare earth dopants such as Ce, Tb, and Eu (or Pr) to obtain precise blue, green, and red emissions, respectively. After a brief review of the latest developments in the field, this talk will focus on the luminescence of rare earth (Ce, Tb, Eu) doped silicon oxides, nitrides, and carbides. We have demonstrated very high, optically active concentrations of the rare earths by using in-situ doping processes, using plasma enhanced chemical vapour deposition as a low thermal budget process for film deposition. Details of the luminescence mechanisms in these materials, however, remain a matter of debate, particularly in cases where the composition of the host matrix is varied and/or where nanoclusters/nanocrystals form during the anneal process. Finally, I will discuss some of the challenges in developing electrically driven lighting cells suitable for SSL and in particular, the development of white light emitters from rare earth doped Si-based materials.

10:10 AM

(ICACC-FS2- 022- 2017) Photonic and plasmonic confinement in self-assembled nanostructures for control and manipulation of light emission, sensing, and harvesting (Invited)

J. A. Zapien*[1]; 1. City University of Hong Kong, Hong Kong

Self-assembled strategies facilitate the growth of high quality nanostructures enabling a wealth of applications hardly available with conventional epitaxial techniques that require lattice-matching substrates. For example, efficient wave guiding with low optical losses can be achieved in low defect density and high refractive index II-VI nanowires and nanoribbons. Coupled with high optical gain, from strong near-band gap emission and large exciton binding energy, self-assembled optically pumped nanolasers with pre-selected emission wavelength are readily available from tunable-composition II-VI ternary and quaternary self-assembled nanostructures. The photonic emission and confinement in these materials can be further tailored using the efficient coupling of electromagnetic (EM) energy with plasmonic effects that can occur at metal-dielectric interfaces. One prominent example is the large EM enhancement in Surface Enhanced Raman Scattering (SERS) that has been shown to enable single-molecule chemical detection. Clearly then, hybrid photonic-plasmonic nanostructures present multiple opportunities to manipulate light at the nanoscale. In this presentation I will discuss recent advances and opportunities in the use of strategies for the development of improved light emission, sensing, and harvesting schemes.

10:40 AM

(ICACC-FS2- 023- 2017) Carbon/ perovskite hybrid materials based photodetectors (Invited)

R. Nechache*[1]; 1. École de technologie supérieure (ETS), Canada

Nanostructured carbon (NC) are very attractive nanomaterials for numerous applications because of their unique mechanical and optoelectronic properties. So far, few groups have explored the potential of NC when combined with perovskites materials. In this combination, the perovskites is the charge generator while the NC is expected to act as both charge collector and transporter. In this work, we fabricate NC/perovskites hybrid based photodetectors by using a simple method to control amount of NC followed by spin coating the perovskites. Here, we will present the structural, optical and unprecedented photoconductive properties of the obtained hybrid materials and their dependence on the NC ratio.

11:10 AM

(ICACC-FS2- 024- 2017) Synthesis of small Al/Al_2O_3 nanoparticles for plasmonics (Invited)

J. Plain*[1]; 1. Université de technologie de Troyes, France

UV Localized Surface Plasmon Resonances (LSPRs) are very attracting because their energy matches with most of the electronic transition energies of molecules or solids. In this scope, the development of efficient and low-cost techniques for the synthesis of reproducible Al nanostructures with very good crystalline quality and optical properties has to be investigated. In this presentation, we describe various methods for the growth of crystalline Al-NPs. The nanoparticles are made using very reproducible synthesis routes. The first approach is based on the reduction of aluminum ions. The second approach relies on the use of sono-chemistry of aluminum foils. Particles as small as 2nm have been synthesized and characterized with a transmission electron microscope, extinction spectroscopy and other methods. By playing on various the medium of synthesis and the temperature of reaction, it appears to be possible to tune under control the size of the nanoparticles. We completed the characterizations by investigating the optical properties of the synthesized Al NPs. To summarize, we described in this presentation various chemical method for the growth of aluminum nanoparticle. AL-NPs present a very good homogeneity and reproducibility. They exhibit sharp localized surface plasmon resonances (LSPRs) in the UV region as it has been showed by extinction spectroscopy characterization.

S1: Mechanical Behavior and Performance of Ceramics & Composites

Environmental Effects and Thermo-mechanical Performance

Room: Coquina Salon D

Session Chairs: Marina Ruggles-Wrenn, Air Force Institute of Technology; Randall Hay, Air Force Research Laboratory

8:30 AM

(ICACC-S1-033-2017) Creep of HI-NICALON™ S Fiber Tows at 700°C in Air and in Silicic Acid-saturated Steam (Invited)

M. Piper[1]; M. Ruggles-Wrenn*[1]; 1. Air Force Institute of Technology, USA

Advanced SiC/SiC ceramic matrix composites (CMCs) are being considered for demanding aerospace applications, which require exposure to high temperature combustion environments. Steam is a major component of these environments. Steam entering composite through cracks in SiC matrix becomes saturated with $Si(OH)_4$. Investigating stressed oxidation of advanced SiC fibers at elevated temperature in $Si(OH)_4$ saturated steam is critical to assessing long-term durability of SiC/SiC CMCs. To study these phenomena, creep tests of Hi-Nicalon™ S SiC fibers were performed at 700°C in air and in $Si(OH)_4$ saturated steam. The fibers were tested in bundles consisting of approximately 500 filaments with an average diameter of 12 mm, as supplied by the manufacturer. Creep stresses ranged from 2.96 to 1256 MPa in air and from 2.96 to 798 MPa in $Si(OH)_4$ saturated steam. Primary and secondary creep regimes were observed in all tests. Creep run-out defined as 100 h at creep stress was achieved at 1023 MPa in air, but only at 750 MPa in $Si(OH)_4$ saturated steam. Steady-state creep rates in $Si(OH)_4$ saturated steam were approximately an order of magnitude higher than those in air. Post-test SEM examination revealed passive oxidation of fibers tested in air or in steam, but showed no evidence of active oxidation.

9:00 AM

(ICACC-S1-034-2017) Modeling of Different Fiber Type and Content SiC/SiC Minicomposite Creep Behavior

A. S. Almansour*[1]; G. N. Morscher[2]; 1. NASA Glenn Research Center, USA; 2. University of Akron, USA

Silicon Carbide based Ceramic Matrix Composites (CMCs) are attractive materials for use in high-temperature applications in the aerospace and nuclear industries. However, creep damage mechanism in CMCs is the most dominant mechanism at elevated temperatures. Consequently, the tensile creep behavior of Hi-Nicalon, Hi-Nicalon Type S SiC fibers and Chemical vapor infiltrated Silicon Carbide matrix (CVI-SiC) were characterized and creep parameters were extracted from creep experiments. Some fiber creep tests were performed in inert environment at 1200°C on individual fibers. Creep behavior of different fiber content pristine and precracked Hi-Nicalon and Hi-Nicalon Type S reinforced minicomposites with BN interphases and CVI-SiC matrix were then modelled using the creep data found in this study and the literature and compared with creep experiments results for the pristine and precracked Hi-Nicalon and Hi-Nicalon Type S minicomposites. Finally, the effects of load-sharing and matrix cracking on CMC creep behavior will be discussed.

9:20 AM

(ICACC-S1-035-2017) Fiber Strength, Oxidation and Scale Crystallization Kinetics, and Scale Dewetting after $Si(OH)_4$ Saturated Steam Exposure of SiC Fibers at 500 to 1600C

R. Hay*[1]; 1. Air Force Research Laboratory, USA

Most applications for SiC-SiC CMCs involve exposure to high temperature steam as a combustion environment component. The effects of such exposure on fiber mechanical properties is consequently of great interest to lifetime prognosis for these materials. Hi-Nicalon^{TM}-S SiC fibers were exposed to steam at 500 to 1600°C for up to 100 hours. The steam was pre-saturated with $Si(OH)_4$ to promote passive oxidation. Scale thickness, morphology, and crystallization extent were characterized by TEM. Fiber strength was measured using 30 single-filament tensile tests. Oxidation kinetics were determined for glass and crystalline scales. Crystallization kinetics did not obey simple Avrami kinetics, and extensive scale dewetting with cristobalite bead formation on fiber surfaces occurred during steam exposure at 700 to 900°C. Scale dewetting is accompanied by more rapid oxidation because of the exposure of SiC surface unpassivated by a silica scale. Dewetting mechanisms are discussed. Fiber strength degradation after steam exposure was observed at all temperatures except 500°C. Mechanisms and models for fiber strength degradation are discussed and compared with those developed for dry air oxidation.

9:40 AM

(ICACC-S1-036-2017) A new temperature dependent first matrix cracking stress model for the unidirectional fiber reinforced ceramic matrix composites

W. Li*[1]; Y. Deng[1]; R. Wang[2]; 1. Chongqing University, China; 2. Chongqing University of Science and Technology, China

In this paper, based on a temperature dependent matrix fracture surface energy model and the classical ACK theory, a new temperature dependent first matrix cracking stress model is developed for the unidirectional fiber reinforced ceramic matrix composites. The combined effects of temperature, matrix fracture surface energy, interfacial shear strength, Young's modulus and residual thermal stress on the first matrix cracking stress of the composites are included in the new model. According to the model, the temperature dependent first matrix cracking stress of materials can be easily predicted using some basic material parameters. The model is verified by comparison with experimental data of SiC fiber reinforced reaction-bonded Si_3N_4 composites at different thermal environment. Good agreement is obtained between the predicted and the experimental data of first matrix cracking stress of fiber reinforced composites. The dependency of first matrix cracking stress on several parameters is systematically discussed.

10:20 AM

(ICACC-S1-037-2017) Creep-Rupture Behavior of Notched Oxide/Oxide Ceramic Matrix Composite in a Combustion Environment

A. K. Singh*[1]; V. Sabelkin[1]; S. Mall[1]; 1. Air Force Institute of Technology, USA

Creep-rupture tests were performed in a simultaneous combustion environment on double-edge notch and center hole oxide/oxide ceramic matrix composite specimens. The composite, designated as N720/A, constituted woven 0°/90° Nextel^{TM}720 fibers in alumina matrix. The specimens recorded a maximum temperature of 1250 ± 50°C in the notch region where the combustion flame directly impinged. Specimens were loaded to the desired load levels at a rate of 25 N/s, following which the load was sustained till either the specimen ruptured or a run-out time of 25 hours (90,000s) was achieved. Optical and scanning electron microscopes were used to characterize damage in the failed specimens. The test results were compared to earlier published results in 1200°C (isothermal) laboratory air environment. For a certain creep life, the applied creep stress for both the notch types was generally lower in the combustion environment than the laboratory air environment. The difference in the applied stress between the two environments however kept shrinking with increasing creep life such that for a life 90,000s, the applied creep stress in the two environments was almost the same. Finite element simulations attributed lower applied creep stress in the combustion environment to the presence of thermal gradient stresses, which were not present in the isothermal laboratory air environment.

10:40 AM

(ICACC-S1-038-2017) Estimation of micro-crack behaviour in particulate ceramic composite under conditions of subcritical crack growth

K. Stegnerova*[1]; L. Nahlik[1]; Z. Majer[1]; P. Hutar[1]; 1. Institute of Physics of Materials, Academy of Sciences of the Czech Republic, Czech Republic

Particulate ceramic composites are nowadays widely used in many engineering applications. The contribution is focused on crack behaviour during so-called subcritical crack growth (SCCG) in glass-ceramic particulate composites. During conditions of SCCG the existing cracks can propagate under an applied stress with stress intensity factor value lower than the fracture toughness. Finite element method was used and 2D numerical model of ceramic particulate composite with alumina particles embedded in glass matrix was developed. The crack propagation through the composite under SCCG conditions (static load) was simulated. An existence of residual stresses developed during composite manufacturing process was taken into account in simulations. An approach based on the strain energy release rate was used for description of the crack behaviour. The residual lifetime of the ceramic composite is estimated by analytical approach. The influence of presence of particles and residual stresses on crack growth rate is determined. The paper contributes to a better understanding of micro-crack propagation in particulate ceramic composites in the field of residual stresses.

11:00 AM

(ICACC-S1-039-2017) Diesel Particulate Filters: Understanding Thermal Chemical Conditions Lead to Early Failures

K. Yang*[1]; 1. Lehigh University, USA

The effects of Na, K, and Fe with porous cordierite were investigated at elevated temperatures. The samples were heated to 300°C, 500°C, 900°C, and 1100°C – and the effects of the ash components were appraised by XRD, SEM and WDX. The temperatures selected were identified to represent various DPF thermal regimes experienced during operation, consisting of; typical operation (300°C), passive regeneration (500°C), active regeneration (900°C), and active regeneration with exothermic soot combustion (1100°C). At 900°C and 1100°C, $FeSO_4$ reacted with cordierite through Mg substitution with Fe as detected by XRD. Both Na_2CO_3 and K_3PO_4 possess similar corrosive mechanism as they substitute for the Mg or Al in cordierite to form sodium alumisilicate, spinel, nepheline, kaliophilite or potassium alumisilicate. The corrosive effects of Na and K begin at temperatures around 300°C. As observed by WDX, the corrosive pathway of Na and K begins by attaching to the cordierite surface. With increased temperature, both sodium and potassium diffuse throughout the entire porous substrate. SEM results suggest that Na_2CO_3 produced the most cracks, when compared to $FeSO_4$ and K_3PO_4 exposure. When considering XRD results, the cordierite substrate became more amorphous for Fe, Na and K exposed DPF substrate – with respect to increased temperature and increased exposure time.

11:20 AM

(ICACC-S1-040-2017) Microcrack trapping process of discontinuous carbon fiber-SiC/C minicomposite phase in discontinuous carbon fiber-reinforced SiC matrix composites

Y. Atsumi*[1]; K. Kajihara[1]; Y. Kagawa[1]; 1. The university of Tokyo, Japan

Tough behavior of discontinuous carbon fiber-dispersed SiC matrix composite originates from crack trapping mechanism in heterogeneous microstructure of composite. The composite consists of four major phases: SiC, Si, carbon fiber-SiC, and carbon fiber-C minicomposite phases. Major crack trapping mechanism exists in discontinuous carbon fiber-carbon matrix minicopmposite phase. Understanding of the trapping mechanism needs interaction between crack formed in Si or SiC phase and minicomposite phases. The minicomposite phases have large anisotropy and the anisotropic factors play important role on the interaction behavior. In the present study, effects of anisotropic properties of minicomposite phase on crack-minicomposite interaction process and crack arrest behavior are analyzed using FEM. The results are discussed in terms of crack trapping mechanism and optimum fiber distribution in composite.

11:40 AM

(ICACC-S1-041-2017) Effect of test parameters on the mechanical properties of single tow SiC mini-composite

K. Kawanishi*[1]; 1. IHI Corporation, Japan

Single tow SiC-CVI matrix minicomposite is useful and effective to evaluate the fracture mechanism of SiC fiber and SiC-CVI matrix, but the test or evaluation method has not been established. In this paper, the effect of several test parameters including specimen length and testing rate on the minicomposite strength behavior has been investigated. Also, as the parameter of manufacturing, the CVI operation time for minicomposite manufacturing has been changed and the influence of the CVI thickness variation has been investigated. The fracture and creep rupture tests was performed under the fixed condition, and the influence of coating interface on the strength behavior has been found.

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

CMAS-related TBC/EBC Degradation and Mitigation Strategies II

Room: St. John
Session Chairs: Valerie Wiesner, NASA Glenn Research Center; Peter Mechnich, German Aerospace Center (DLR)

8:30 AM

(ICACC-S2-030-2017) Thermochemistry of CaO-MgO-Al_2O_3-SiO_2 (CMAS) and Advanced Thermal and Environmental Barrier Coating Systems

G. Costa*[1]; D. Zhu[1]; 1. NASA Glenn Research Center, USA

CaO-MgO-Al_2O_3-SiO_2 (CMAS) oxides are constituents in a broad number of materials and minerals which have recently inferred to discussions in materials science, planetary science, geochemistry and cosmochemistry communities. In materials science, there is increasing interest in the degradation studies of thermal (TBC) and environmental (EBC) barrier coatings of gas turbines by molten CMAS. CMAS minerals usually are carried by the intake air into gas turbines, e.g. in aircraft engines, and their deposits react at high temperatures (>1000°C) with the coating materials. This causes degradation and accelerated failure of the static and rotating components of the turbine engines. We discuss some preliminary results of the reactions between CMAS and Rare-Earth (RE – Y, Yb, Dy, Gd, Nd and Sm) oxide stabilized ZrO_2 or HfO_2 systems, and the stability of the resulting oxides and silicates. Plasma sprayed hollow tube samples (~ Ø = 2.2 mm and 26 mm height) were half filled with CMAS powder, wrapped and sealed with platinum foil, and heat treated at 1310°C for 5h. Samples were characterized by differential scanning calorimetry, X-ray diffraction and cross section electron microscopy analysis.

8:50 AM

(ICACC-S2-031-2017) Comparison of CMAS resistance of YSZ, YAG and Gadolinium Zirconate TBCs

R. Kumar*[1]; E. H. Jordan[2]; M. Gell[1]; 1. University of Connecticut, USA; 2. University of Connecticut, USA

It is well known that calcium-magnesium-alumino-silicate (CMAS) that is ingested into gas turbines degrades thermal barrier coatings (TBCs) especially for the most widely used material; Yttria Stabilized Zirconia (YSZ). Gadolinium Zirconate (GZO) coatings vigorously react with CMAS to form blocking phases. In the present work we primarily examine the behavior of Yttrium Aluminum Garnet (YAG) and also other Yttrium-Aluminum compositions (YAP and YAM), with respect to CMAS resistance. We have previously shown that YAG with a specific microstructure (stress relieving vertical cracks) has good cyclic furnace test performance, erosion performance, competitive thermal conductivity, low density and high temperature stability. CMAS interaction studies have been done by applying CMAS paste on the aforementioned ceramic TBCs which resulted in almost immediate failure during thermal cycling. To prevent such copious amounts of CMAS exposure, Spritz test was done on the TBCs which comprised of spraying a diluted aqueous solution of CMAS during subsequent thermal cycles till TBCs failure. In both Paste and Spritz tests the durability of YAG in CMAS environment was greater than YSZ and GZO. Microstructure difference must be considered in interpreting these results.

9:10 AM

(ICACC-S2-032-2017) Calcium-magnesium-aluminosilicate (CMAS) interactions with advanced environmental barrier coating candidate material

V. L. Wiesner*[1]; N. S. Johnson[2]; D. Scales[3]; B. J. Harder[1]; N. Bansal[1];
1. NASA Glenn Research Center, USA; 2. Colorado School of Mines, USA;
3. University of Washington, USA

Air-breathing turbine engines regularly ingest particulates, such as sand and volcanic ash, during operation. At temperatures >1200°C, particulates melt, yielding calcium-magnesium-aluminosilicate (CMAS) glass deposits. These deposits can significantly reduce the durability of present and future engine component materials, including silicon-based ceramic matrix composite (CMC) materials with environmental barrier coatings (EBCs). In this study, a CMAS glass prepared by melting a synthetic sand mixture was used to evaluate the high-temperature interactions with ytterbium disilicate ($Yb_2Si_2O_7$), a candidate environmental barrier coating (EBC) material. CMAS glass was applied to the surface of hot-pressed EBC substrates, which were heated in air at temperatures from 1200°C to 1500°C. Samples were characterized using scanning electron microscopy, X-ray energy-dispersive spectroscopy, X-ray diffraction and electron microprobe analysis to evaluate the resulting microstructure, phases and compositions at the CMAS/ $Yb_2Si_2O_7$ interface.

9:30 AM

(ICACC-S2-033-2017) On the role of process induced microstructural variants on CMAS interactions in plasma sprayed single and multilayer coatings

V. Viswanathan[1]; E. Gildersleeve*[1]; G. Dwivedi[1]; S. Sampath[1]; 1. Stony Brook University, USA

The continued need for increments in gas turbine operating temperatures has necessitated developments in new thermal barrier materials and their processing. Of particular interest in recent years is the potential for Gadolinium Zirconate as candidate TBCs to replace yttria stabilized zirconia. $Gd_2Zr_2O_7$ in particular provides lower conductivity and is resistant to environmental damage from ingested sand particles (categorized as CMAS for calcium magnesium alumino-silicate). Zirconates however, have several challenges including low fracture toughness, incompatibility with the thermally grown alumina and low erosion resistance. Multilayer concepts based on combinations of zirconia and zirconate along with microstructural variants (porous, dense, dense vertically cracked) have been developed to address the multifunctional requirements. Initial results suggest that these microstructural differences impact the CMAS interactions and associated failure mechanisms. This presentation will outline the multilayer strategies along with observations of CMAS interactions.

10:10 AM

(ICACC-S2-034-2017) Ytrria-Rich Zirconia Thermal Barrier Coatings: An Approach to Enhance Calcium-Magnesium-Aluminum-Silicate (CMAS) Resistance

R. Naraparaju*[1]; J. Gomez[2]; U. Schulz[1]; C. V. Ramana[2]; 1. DLR - German Aerospace Center, Germany; 2. The University of Texas, El Paso, USA

Yttria rich-zirconia thermal barrier coatings (TBCs) with a nominal composition of 65 wt % Y_2O_3 balanced ZrO_2 deposited by electron-beam physical vapor deposition (EB-PVD) were proposed and tested for calcium-magnesium-aluminum-silicate (CMAS) infiltration resistance. The infiltration studies were performed with a set of one synthetized CMAS composition and two real volcanic ashes from the Eyjafjallajökull volcano located in Iceland and the Sakurajima volcano located in southern Japan. The coatings were tested at 1250 C for short term (5 min) and long term (intervals from 1 to 20 h). The results indicate significantly different reaction processes for the synthesized CMAS compared with the natural volcanic ashes. The yttria-rich zirconia coatings demonstrate promising results against infiltration by vigorously reacting against the molten glass inducing its crystallization by forming oxyapatite and garnet phases. The formed reaction products effectively sealed the columnar gaps of the TBC and generated a uniform reaction layer that prevented further infiltration.

10:30 AM

(ICACC-S2-035-2017) Spreading dynamic of viscous volcanic ash droplets onto EB-PVD and APS thermal barrier coatings

W. Song*[1]; F. B. Wadsworth[1]; T. I. Yilmaz[1]; U. Schulz[2]; D. B. Dingwell[1]; 1. LMU Munich, Germany; 2. DLR - German Aerospace Center, Germany

Volcanic ash is the most voluminous and widespread product of explosive volcanic eruptions, which may present a critical risk to aviation safety. The melting temperature of most volcanic ash is about 1100°C, which is below the operating temperature of jet engines (1200–2000°C). When ingested, volcanic ash particles are exposed to high temperatures for short timescales which may cause partial or complete melting, and subsequent sticking upon collision with hot parts. Thus, the spreading dynamics of molten volcanic ash particle is fundamental to investigate the impingement phenomena of ash droplets on surfaces during jet engine operation and mitigate the subsequent deterioration of jet engine performance. Here, we study both the natural volcanic ash and a standard glass (DGG). We produce homogeneous droplets with various diameters (1-3 mm) onto a graphite substrate. Subsequently, we experimentally and theoretically investigate the evolution of spreading dynamics of molten silicate droplets onto 200-μm-thick thermal barrier coatings (TBC) consisting of tetragonal, 7 wt% Y_2O_3 partially stabilized ZrO_2 (7-YSZ) deposited by electron-beam physical vapor deposition (EB-PVD) and air plasma spray (APS), respectively, at 1200°C using an improved sessile-drop method. Finally, we analyze infiltration and degradation by silicate melts and their effects on spreading dynamic of silicate melts.

10:50 AM

(ICACC-S2-036-2017) Effect of CMAS Corrosion on Thermal Conductivity and Mechanical Stresses in Lanthanide Zirconate Pyrochlore Coating Materials

J. Fergus*[1]; A. Bakal[1]; W. Deng[1]; H. Wang[1]; K. Roebbecke[1]; 1. Auburn University, USA

Lanthanide zirconate pyrochlore materials have been investigated for use in thermal barrier coating systems due to their low thermal conductivity and high temperature phase stability. Reaction of lanthanide zirconates with CMAS has been shown to result in the formation of a two-phase mixture of a lanthanide-containing silicate and a cubic fluorite phase. This reaction product can provide a barrier to limit the reaction but also affects the thermal conductivity and mechanical stresses, which will be the focus of this presentation.

11:10 AM

(ICACC-S2-037-2017) Investigation of Rare Earth (RE) titanates as potential Environmental Barrier Coating (EBC) constituents for mitigation of CMAS attack

R. Webster*[1]; E. Opila[1]; 1. University of Virginia, USA

Calcium magnesium alumino-silicate (CMAS) attack is a pressing issue in the development of EBCs for Ceramic Matrix Composites (CMCs). At temperatures greater than approximately 1200°C, CMAS becomes molten and can penetrate EBC materials, causing premature coating failure. Inducing crystallization of these glassy deposits has been suggested in the literature as a mitigation strategy. In this study, the crystallization behavior of CMAS in contact with novel, possible candidate coating materials RE_2TiO_5 and $RE_2Ti_2O_7$ (RE=Y, Yb), was investigated. Powder mixtures of the RE titanates and CMAS (70:30 wt %, respectively) were heat-treated at either 1300 or 1500°C in a stagnant air box furnace. Samples were held for 10 min and 4, 24, or 96 h then cooled to RT at 10°C/min. Resulting phases were characterized by X-Ray diffraction (XRD); RE_2TiO_5 showed formation of a silicate oxyapatite and $RE_2Ti_2O_7$ after short exposure times (≤ 10 min) while $RE_2Ti_2O_7$ dissolved quickly (≤ 10 min) in the glass with no new crystalline phase formation. Preliminary studies on RE titanate "pellets" have also been conducted to elucidate the morphology and kinetics of reaction.

11:30 AM

(ICACC-S2-038-2017) Mechanical and Chemical Consequences of CMAS Interaction with Y2Si2O7 Environmental Barrier Coating

W. D. Summers*[1]; 1. UCSB, USA

The introduction of SiC/SiC ceramic matrix composites as high temperature components in turbine engines is in part predicated on the development of durable Environmental Barrier Coatings (EBC). The EBC protects the SiC/SiC composite from water-vapor induced oxidation at high-temperatures but suffers at the hands of molten-silicate debris (known as CMAS) that can be ingested by the engine. Molten CMAS can react with EBC materials, in this case Yttrium Di-silicate, forming a new phase known as Apatite, which has vastly different thermo-mechanical properties to the EBC and leads to the formation of vertical cracks which can penetrate through the EBC to the SiC/SiC substrate. In this work we couple experimental studies of the reaction kinetics and thermodynamics with mechanical modelling of the cracking phenomena to understand the degradation mechanisms that are active which may lead to failure of the coating. We present concepts to mitigate these degradation mechanisms for future EBC applications.

S3: 14th International Symposium on Solid Oxide Fuel Cells (SOFC): Materials, Science and Technology

Fuel Electrode

Room: Crystal
Session Chairs: Scott Barnett, Northwestern Univ; Tatsumi Ishihara, Kyushu University

8:30 AM

(ICACC-S3-027-2017) $LaFeO_3$ based oxide anode for reversible solid oxide fuel cells and applied for co-electrolysis of CO_2/H_2O (Invited)

T. Ishihara*[1]; K. Wu[1]; K. Hosoi[1]; S. Ida[1]; 1. Kyushu University, Japan

Reversible operation of high temperature electrolysis and solid oxide fuel cell mode is also important from energy storage. In this study, we investigated $La_{0.6}Sr_{0.4}FeO_3$ (LSF) based oxide for anode of reversible operation of SOFC. Although power density of the cell is little lower than that of Ni-Fe conventional metallic anode. It was found that much higher stability was observed after few 10 cycles. This suggests that LSF is highly stable against exposure to oxidation and reduction atmosphere. In this study, catalytic activity of Mn doped LSF for cathode of high temperature electrolysis were also studied. In case of the conventional Ni-Fe cathode, H_2 was always dominated in product from CO_2/H_2O co-electrolysis because of high activity to water gas shift reaction ($CO+H_2O=H_2+CO_2$). Therefore, control of CO/H_2 ratio in product is rather difficult on the conventional metallic cathode. In contrast, it was found that CO/H_2 ratio is almost the same with CO_2/H_2O in reactant when LSFM is used for cathode of electrolyzer. Therefore, this study shows the superior oxide anode and cathode performance of $LaFeO_3$ perovskite oxide for SORC using $LaGaO_3$ electrolyte.

9:00 AM

(ICACC-S3-028-2017) Solid Oxide Fuel Cells on Hydrocarbon Fuels at Low-Temperatures

M. Abdul Jabbar*[1]; C. Gore[2]; K. Pan[2]; B. Blackburn[2]; E. Wachsman[2]; 1. University of Maryland, USA; 2. Redox Power Systems, LLC, USA

Solid oxide fuel cells (SOFCs) are highly efficient electrochemical devices that can directly convert chemical energy stored in hydrogen or non-hydrogen fuels (e.g., natural gas) into electrical energy. SOFCs have a unique feature of internally reforming the hydrocarbon fuels. For large-scale stationary applications, SOFCs can efficiently operate in the high temperature ranges between 800-1000°C by integrating with gas turbines. Lowering the operating temperature down to 400°C unfolds smaller scale applications such as range extender for plug-in-vehicles. This work focuses on the need and perceived issues of low-temperature SOFCs operable on hydrocarbon fuels. In particular, developments of fuel electrode materials suitable for low-temperature SOFCs with an emphasis on direct utilization of hydrocarbon fuels are discussed. At temperatures < 600°C, modified Ni-ceria based anodes are widely-used materials to directly utilize the hydrocarbon based fuels. Alternatively, ceramic oxides are emerging as potential anode materials for high performing, robust, low temperature operating SOFCs.

9:20 AM

(ICACC-S3-029-2017) Redox Stability and Electrochemical Performance of Co Doped $Sr_2Fe_{1+x}Mo_{1-x}O_{6-\delta}$ for Solid-Oxide Fuel Cells

H. Qi*[1]; T. Thomas[1]; E. Sabolsky[1]; X. Liu[1]; J. Zondlo[1]; R. Hart[2]; E. Jezek[2]; 1. West Virginia University, USA; 2. GE, USA

Based on $Sr_2Fe_{1.5}Mo_{0.5}O_{6-\delta}$, a potential electrode material with high powder density both as cathode and anode, perovskite materials $Sr_2Fe_{1+x}Mo_{1-x}O_{6-\delta}$ (x=0.4~0.6) with Co doping on B site have been successfully synthesized by sol-gel method. Conductivity, electrochemical impedance spectroscopy (EIS) and coefficient of thermal expansion (CTE) of $Sr_2Fe_{1+x}Mo_{1-x}O_{6-\delta}$ family will be measured at 800°C in forming gas and air. Full cells will operate in wet H_2 (3% H_2O) below 800°C. After analyzed in single atmosphere, thermal cycles test and redox cycles test will be followed, which focuses on anode material $Sr_2Fe_{1+x}Mo_{1-x}O_{6-\delta}$ family's stability and fuel cell's endurance in redox atmosphere. Influence of iron concentration on crystal structure stability in redox atmosphere and Co doping amount on electrochemical performance improvement will be analyzed and discussed in the end.

9:40 AM

(ICACC-S3-030-2017) Development of a thermal spray, redox stable, ceramic anode for metal supported SOFC (Invited)

V. K. Srivastava*[1]; R. Hart[1]; R. Northey[1]; E. Jezek[1]; L. Rosenweig[1]; P. Thomas[1]; M. Alinger[3]; L. Leblanc[3]; E. Sabolsky[2]; T. Thomas[2]; X. Liu[2]; J. Zondlo[2]; H. Qi[2]; S. Bancheri[1]; 1. GE Global Research, USA; 2. Department of Mechanical and Aerospace Engineering, USA; 3. GE Fuel Cell, USA

GE Global Research in partnership with West Virginia University (WVU) and GE-Fuel Cells LLC, and in collaborative agreement with US-DOE-NETL are developing thermal-spray, redox stable, ceramic SOFC anodes that will enable robust, large-scale, metal supported solid oxide fuel cell (SOFC) technology. GE's metal supported cells and thermal spray manufacturing approach deliver a low-cost, scalable product. Integration of new anode materials will drive improved reliability and stack robustness to deliver a lower operating cost for GE's SOFC customers. Stack testing results on 100cm^2 metal supported cells with lanthanum strontium titanate and gadolinium doped ceria thermal spray deposited electrodes demonstrates improved mechanical performance during redox cycling. Formulation and microstructure improvement efforts will also be described.

10:30 AM

(ICACC-S3-031-2017) Infiltration of SOFC Anodes for Improved Performance at High Fuel Utilization

Y. Lu[1]; P. Gasper[1]; U. Pal[1]; S. Gopalan[1]; S. Basu*[1]; 1. Boston University, USA

High fuel utilization conditions result in reduced electrochemical performance in SOFCs due to an increase in anodic polarization. One approach to ameliorate this problem is to increase the anodic TPB length by deposition of sub-micron nickel particles in standard cells with Ni/YSZ cermet anodes. This paper reports the results of nickel infiltration on the performance of anode-supported SOFC over a wide range of hydrogen and water vapor partial pressures at intermediate temperatures. The microstructure and stability of the nano-sized infiltrated Ni particles will be reported. I-V and EIS results will also be presented and the performance of the cells will be connected to the anode microstructure.

10:50 AM

(ICACC-S3-032-2017) Low-Temperature Solid Oxide Cells With Thin (La,Sr)(Ga,Mg)O_3 (LSGM) Electrolytes: Effect of Infiltrated Anode

Z. Gao[1]; C. Ng*[1]; M. Y. Lu[1]; S. Barnett[1]; 1. Northwestern Univ, USA

This paper describes SOFCs with thin LSGM electrolytes with LSGM anode functional layers (AFLs) and (Sr,La)TiO_3 supports infiltrated with different species. Cells infiltrated with Ni yield optimal performance, e.g., > 1.0 W cm^{-2} at 600°C, for 50-micron-thick AFLs with 32 % porosity and a Ni loading of 12.3 vol%. There is also interest in Ni-free cells that can potentially work with alternative carbon-containing and impurity-laden fuels. Results are presented on cells that have been infiltrated with Gd-doped Ceria (GDC) and GDC with Ru. The anode performance of these cells is not sufficient for low-temperature operation, but is reasonable at 700 – 800°C. The GDC-infiltrated cells yield a maximum power density of 0.7 W cm^{-2} at 800°C, compared to 0.95 W cm^{-2} at 800°C with GDC-Ru infiltration. The beneficial effect of Ru is even greater at lower temperatures – at 700°C, for example, the maximum power density is increased from 0.25 for GDC alone to 0.55 W cm^{-2} for GDC-Ru. The GDC-Ru-infiltrated cells also worked reasonably well with humidified CH_4 fuel, e.g., yielding a maximum power density of nearly 0.4 W cm^{-2} at 700°C. Cells without Ru yielded very poor performance in methane, 0.07 Wcm^{-2} at 700°C. Results on life testing in both hydrogen and methane will be presented, along with impedance spectroscopy data and electron microscopy imaging of the anodes.

11:10 AM

(ICACC-S3-033-2017) Understanding Poisoning of Proton Conducting SOFC Anode by Hydrogen Sulfide and Carbon Dioxide

S. Sun*[1]; Z. Cheng[1]; 1. Florida International University, USA

Proton conducting solid oxide fuel cells (SOFCs) attract great interest due to potential for higher performance at intermediate temperature (~400-600°C). Here we report our study aimed at revealing the poisoning behavior caused by hydrogen sulfide (H_2S) and carbon dioxide (CO_2) as fuel contaminants for proton-conducting SOFCs. Anode-supported proton-conducting SOFCs with $BaZe_{0.1}Ce_{0.7}Y_{0.1}Yb_{0.1}O_3$ (BZCYYb) electrolyte and Ni-BZCYYb anode were subject to hydrogen fuel mixture containing ppm-level H_2S or percentage-level CO_2. Results suggest that, contrary to conventional SOFCs that show "sulfur poisoning" and "CO_2 tolerance", such Ni-BZCYYb anode/BZCYYb electrolyte anode-supported SOFC button cells show tolerance to H_2S up to ~5 ppm while obvious poisoning by 5% CO_2. On the other hand, test on anode symmetrical cells of Ni-BZCYYb/BZCYYb/Ni-BZCYYb shows poisoning by both 3-10 ppm H_2S and 5% CO_2 in H_2 and the change in impedance at 750°C due to such poisoning was very similar. Experimental results aimed at understanding such electrochemical behaviors for proton-conducting SOFCs versus conventional oxide-ion conducting SOFCs will be presented and their implications on the fundamental mechanism for anode hydrogen electrochemical oxidation reaction in proton conducting SOFCs, especially the potential catalytic roles played by BZCYYb proton conducting electrolyte, will be discussed.

11:30 AM

(ICACC-S3-034-2017) Ni-Cu Spinel Catalysts for Reforming of Hydrocarbons on Solid Oxide Fuel Cell Anodes

S. Misture*[1]; P. Sowinski[1]; 1. Alfred University, USA

$(Ni_{0.375}Cu_{0.375}Mg_{0.25})Al_2O_4$ spinel catalyst has been synthesized by solid state reaction and tested for use as an anode internal reforming layer for solid oxide fuel cells (SOFCs). High temperature X-ray powder diffraction (XRD/HTXRD) was used to monitor selective reduction of the oxide to form well-dispersed and catalytically active Ni-Cu alloy nanoparticles on the surface of the spinel support. Anodes were fabricated as typical Ni/YSZ composites with and without an added surface layer of the spinel reforming catalysts. Button cell testing using H_2 fuel demonstrated that the spinel reforming catalyst has no effect on the power density when using H2 fuel at 850, 750, and 650[endif]-->C. For a non-coking methane fuel (2 CO_2: 1 CH_4) the peak power density increased by 30-35% at 850 and 750[endif]-->C when using the spinel reforming catalyst. While operating in dry 1 CO_2: 1 CH_4 at 850[endif]-->C, the addition of 0.015g of spinel catalyst resulted in a power density of 117 $mWcm^{-2}$, again an increase of roughly 32%. Furthermore, the spinel catalyst has been successfully operated for 96 hours under dry 1:1 CH_4:CO_2 fuel mixture at 850°C without significant power degradation.

S4: Armor Ceramics

Advances in Synthesis and Processing I

Room: Coquina Salon E
Session Chair: Kristopher Behler, U.S. Army Reseach Laboratory; Hywel Jones, Sheffield Hallam University

8:30 AM

(ICACC-S4-028-2017) B_6O, Synthesis and Powder Properties

C. M. Weiss*[1]; 1. US Army Research Laboratory, USA

The evolving threat for the Warfighter continues to press the need for the development of new materials and mechanisms for the advancement of soldier protection. Boron suboxide (B_6O), with high hardness and low density, has been considered as a potential enabler for improving ballistic efficiency beyond the current state of the art ceramics such as boron carbide (B_4C) and silicon carbide (SiC). There are no large scale commercial suppliers of this powder so various synthesis methods are being explored internally. There are several methods for producing B_6O powder in the literature, including directly reacting boron with B_2O_3, and processes involving oxidation and reduction of boron based materials with metals, such as magnesium. These methods however, have specific drawbacks, including the need for post-synthesis milling steps and residual impurities. The U.S. Army Research Laboratory (ARL) has developed a synthesis route for B_6O involving a multi-step oxidation process, in which amorphous boron is converted to B_6O through solid/gas reactions. This method has the advantage of retaining a powder morphology throughout the reaction sequence, allows for a better control of the synthesis process, and eliminates the need for powder milling. Powders produced using the ARL method will be characterized and compared against the other available sources to assess the effect of powder morphology and chemistry on the resultant densification behavior.

8:50 AM

(ICACC-S4-029-2017) Boron carbide room temperature injection molding and pressureless sintering

A. Diaz Cano*[1]; R. Trice[1]; J. Youngblood[1]; 1. Purdue University, USA

The properties of boron carbide (B4C) make it suitable for multiple applications. However, traditional forming processes are limited to simple geometries (e.g. plates, disks or bars). Additionally, near-net shape forming approaches employed large amounts of binders or cross-linking reactions are required to develop significant green body strength. Present study proposes a colloidal processing approach, mixing up to 56 vol.% B4C, water and a dispersant to produce yield pseudo-plastic flow behavior. Polyethylenimine (PEI) was selected as the dispersant (and binder) based on its zeta potential with the B4C powder. Yield point of the suspension was tailored by adjusting PEI content. Suspensions in this study can be used via injection molding, casting, and additive manufacturing. All forming process carried out at room temperature. Finally, sintering aids were added to attrition milled boron carbide. Pellets were formed under 35 MPa for 20 seconds and sintered at 2000oC in argon. Density and hardness were measured for each sample and compared to a reference composition, i.e. attrition milled boron carbide with no sintering aid added. Microstructures were analyzed via SEM and effect of sintering aids were studied. Y2O3 was found to be the most effective sintering aid at a 10 wt % concentration.

9:10 AM

(ICACC-S4-030-2017) Preparation of Boron Carbide with SiC Intergranular Phase

C. Hwang*[1]; S. DiPietro[2]; K. Xie[3]; Q. Yang[1]; A. Khan[1]; V. Domnich[1]; K. Hemker[3]; R. A. Haber[1]; 1. Rutgers University, USA; 2. Exothermics, Inc, USA; 3. Johns Hopkins University, USA

Boron carbide has found applications as lightweight impact-resistant ceramics, such as for body armor and hypersonic vehicles, due to its high hardness, low density, and high melting point. However, further development and application have been restricted by its low fracture toughness, catastrophic failure under high-velocity impacts, and poor sinterability. To overcome these disadvantages various approaches have been applied. Here we report the results of a new approach that is enhance crack-propagation resistance of boron carbide by incorporating a non-oxide intergranular phase of SiC, which is also a hard and lightweight material with relatively high toughness. First we synthesize boron carbide powders with nanoscale surface coating of SiB_6 or Si through DC magnetron sputtering process and then densify the powders through hot press or SPS process while controlling microstructure and phases developing during densification process. Microstructure and phase developments will be discussed based on characterization results of XRD, Raman spectroscopy, FE-SEM, TEM, and EDS. Hardness and crack-propagation resistance evaluated by micro-/nano-indentation methods will be presented.

9:30 AM

(ICACC-S4-031-2017) The Effect of Silica and Alumina-based Additives on the Densification and Microstructure of Hot-Pressed Boron Carbide

K. D. Behler*[1]; J. LaSalvia[2]; C. A. Voigt[3]; S. D. Walck[4]; 1. U.S. Army Research Lab (SURVICE Engineering), USA; 2. U.S. Army Research Lab, USA; 3. U.S. Army Research Lab (CQL), USA; 4. U.S. Army Research Lab (SURVICE Engineering), USA

The effects of silica and alumina-based additives on the densification and microstructure of hot-pressed boron carbide were investigated. Samples were densified between 1800 and 2000°C. Isothermal holds were employed between 1900 and 2000°C to investigate the densification kinetics and determine the activation energies for densification. Ram displacement during hot-pressing was monitored by a linear variable differential transducer (LVDT). Densities of >99% of the theoretical density were obtained in samples containing alumina-based additives as measured by Archimedes' method. The resulting microstructures were characterized using scanning electron microscopy (SEM). Rietveld refinement of x-ray diffraction (XRD) was used to identify phases. X-ray dispersive spectroscopy (XEDS) and Raman spectroscopy were used to identify and map the Al- and Si-based additives within the microstructures. TEM was used to further characterize microstructures as well as material at the grain boundaries. The densification kinetics and microstructure were found to be influenced by the addition of these additives. Experimental procedures, analyses, and results are presented.

Advances in Synthesis and Processing II

Room: Coquina Salon E
Session Chair: Jerry LaSalvia, U.S. Army Research Laboratory; Hywel Jones, Sheffield Hallam University

10:20 AM

(ICACC-S4-032-2017) Progress in Developing Time-Temperature-Transformation (TTT) Diagrams for Grain Boundary Complexion Transitions (Invited)

M. P. Harmer*[1]; 1. Lehigh University, USA

Grain boundaries play a decisive role in determining the processing, microstructure and properties of engineering materials. One important aspect of grain boundaries that has not been fully appreciated until more recently is that they can undergo rapid and discontinuous changes in structure and chemistry (and hence properties) at certain values of thermodynamic and interfacial parameters. These phase-like changes in the state of grain boundaries are referred to as grain boundary complexion transitions, to differentiate them from bulk phase changes. The kinetics of such complexion transitions has been largely unexplored until recently[1,2]. Just as with bulk phase transformations, complexion transition kinetics can be represented on time-temperature-transformation (TTT) diagrams. This talk will address the progress that has been made in developing grain boundary TTT diagrams and discuss some of the future challenges and opportunities.

10:50 AM

(ICACC-S4-033-2017) Effect of Additives on the Densification Kinetics and Microstructure of Hot-Pressed Boron Suboxide

K. D. Behler*[1]; J. LaSalvia[2]; E. R. Shanholtz[3]; C. A. Voigt[4]; L. R. Vargas-Gonzalez[2]; S. D. Walck[5]; 1. U.S. Army Research Lab (SURVICE Engineering), USA; 2. U.S. Army Research Lab, USA; 3. U.S. Army Research Lab (ORISE), USA; 4. U.S. Army Research Lab (CQL), USA; 5. U.S. Army Research Lab (SURVICE Engineering), USA

The effects of silica- and alumina-based additives (1, 2 and 5 vol.%) on the densification kinetics and microstructure of hot-pressed boron suboxide (B_6O) powders has been studied. Isothermal-hold experiments have been conducted within the 1700 – 1850°C temperature range to investigate densification kinetics and calculate activation energies. The densification kinetics and resulting microstructures were found to be significantly influenced by the additives, lowering the onset of rapid densification and activation energy. Raman spectroscopy was used to identify and map the silica- and alumina-based additives within the microstructures. The starting powder morphology and composition as well as the microstructure and phases present within consolidated samples were characterized using scanning electron microscopy (SEM), X-ray energy dispersive spectroscopy (XEDS) and x-ray diffraction (XRD). The starting B_6O powder was found to be oxygen deficient, and contained both boron and magnesium borate phases. For the hot-pressed specimens, the presence of Al_2O_3 and SiO_2 additives resulted in the formation of aluminum borate and possibly borosilicate, respectively. Experimental procedures, analyses, and results are presented.

11:10 AM

(ICACC-S4-034-2017) High Temperature Coupling of Boron Carbide and Silicon Hexaboride for Diffusion Mapping and Mechanical Testing

A. M. Etzold*[1]; R. A. Haber[1]; V. Domnich[1]; A. Khan[1]; 1. Rutgers University, USA

Boron carbide is known to suffer from a loss in compressive strength in high pressure events due to amorphization. DFT simulations indicate that the introduction of silicon into the boron carbide lattice could mitigate this amorphization. Spark Plasma Sintering is used to produce diffusion couples of minimal free carbon boron carbide with a boron rich silicide. The prepared couples are analyzed through x-ray diffraction, Raman spectroscopy, Energy Dispersive X-ray Spectroscopy, and TEM in order to determine their phases, stoichiometries, and map the diffusion of silicon. The results demonstrate the feasibility of incorporating silicon atoms into the boron carbide lattice, which could potentially lead to synthesis of precisely Si-doped boron carbide with enhanced ballistic properties.

11:30 AM

(ICACC-S4-035-2017) Flash sintering of armor ceramics

V. L. Blair*[1]; J. H. Yu[1]; N. Ku[1]; B. McWilliams[1]; J. Campbell[1]; R. E. Brennan[1]; 1. US Army Research Laboratory, USA

High electric current has been shown to enhance sintering kinetics in ceramics by using spark plasma sintering, which can yield fully dense parts in a matter of minutes rather than hours by traditional hot pressing. Recent work has shown that boron suboxide can be flash sintered in air at relatively low temperatures, ~900°C. However, there is a process engineering challenge at hand that results in the outside of the part being fully dense with a green, unsintered core. Additionally, there is the basic scientific question of what electrical properties dictate "flash sinterability." This presentation will attempt to fulfill these challenges and seeks to develop a fully dense ceramic part by flash sintering with high voltages.

11:50 AM

(ICACC-S4-036-2017) Carbon Distribution in Transparent $MgAl_2O_4$ Spinel Synthesized by Spark-Plasma-Sintering (SPS) Processing

K. Morita*[1]; B. Kim[1]; H. Yoshida[1]; Y. Sakka[1]; 1. National Institute for Materials Science (NIMS), Japan

Spark-plasma-sintering (SPS) technique is a powerful sintering tool and has widely been utilized to consolidate various types of materials. The benefit of the SPS technique is heating rates higher than 50°C/min, and hence, can save the total processing time. It is reported, however, that the SPS processed oxides such as Al_2O_3, ZrO_2 and spinel exhibit discoloration. Such a discoloration is known to be remarkable than the HIP/HP processed oxides and affect the optical properties. The discoloration in spinel can be explained by the combination of carbon contaminations and lattice defects, which are introduced in the spinel matrix depending on the SPS conditions. Although the carbon contamination and defects formations can be reduced with decreasing the heating rate to some extent, those still remain in the oxide matrix. The lattice defects can be removed by post-annealing in air. However, since the carbon phases transform to CO/CO_2 babbles within the materials by the post-annealing in air due to the reacting with oxygen, it is very difficult to remove the carbon from the sintered bulk materials. Therefore, the carbon contamination would cause serious problems for the SPS processed materials, especially for oxide ceramics. In the presentation, we will discuss the distribution of carbon contaminant caused depending on the SPS parameters.

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

Thermal Energy Conversion and Energy Storage VII

Room: Tomoka A
Session Chair: Xiangxin Guo, Shanghai Institute of Ceramics, Chinese Academy of Sciences

8:30 AM

(ICACC-S6-023-2017) Stabilization of Battery Electrode-Electrolyte Interfaces Employing Nanocrystals with Passivating Epitaxial Shells (Invited)

J. Cabana*[1]; 1. University of Illinois at Chicago, USA

Chemical degradation at electrode-electrolyte interfaces in high energy storage devices such as Li-ion batteries imposes durability challenges that affect their life and cost. In positive electrodes, degradation is linked to the presence of redox active transition metals at the surface. In recent years, we refined a strategy toward the stabilization of interfaces using core-epitaxial shell nanocrystals. The core of the nanocrystal is composed of an electroactive oxide, which is passivated by an ultrathin epitaxial oxide shell enriched in a redox inactive ion. This approach imparts interfacial stability while preserving the high storage capability and fast carrier transport of the material compared to unmodified versions. The validity of the concept is proved with nanocrystals of either layered-type $LiMO_2$ or spinel LiM_2O_4 with a 1-2 nm Al-rich shell, which showed reduced sensitivity to harsh environments compared to bare counterparts. In this presentation, we will overview the synthetic methodology and the insight gained from this approach on the mechanisms that affect electrode-electrolyte interactions.

9:00 AM

(ICACC-S6-024-2017) Thermochemical investigation of layered $LiNi_xMn_xCo_{1-2x}O_2$ ($0 \leq x \leq 0.5$) compounds as promising lithium ion battery cathode materials

H. J. Seifert*[1]; M. Masoumi[1]; T. L. Reichmann[1]; D. M. Cupid[1]; 1. Karlsruhe Institute of Technology, Germany

Compounds from the $LiNi_xMn_xCo_{1-2x}O_2$ series ($0 \leq x \leq 0.5$) including NMC111 and NMC442 (of commercial interest) are considered as important layered cathode materials for lithium ion batteries. The enthalpies of formation from oxides and oxygen and from elements of samples in this series with x = 0, 1/6, 1/3, 0.4, and 0.5, respectively, were determined using high temperature oxide melt drop solution calorimetry. Sodium molybdate was used as the solvent for calorimetric measurements at 701°C. The studied samples were synthesized by a sol-gel method using metal acetates and adipic acid. The effect of substitution of Co^{3+} for equal amounts of Mn^{4+} and Ni^{2+} on the lattice constants as well as on the relative thermochemical stabilities of the layered structure in the studied series is discussed. The results of our high temperature oxide melt solution calorimetry measurements show that the standard enthalpies of formation of the $LiNi_xMn_xCo_{1-2x}O_2$ compounds from the elements become more exothermic with increasing x. Therefore we show that the investigated NMC compounds are energetically more stable than $LiCoO_2$. This is in agreement with better cycling performance of Ni/Mn-rich compounds at higher voltages.

9:20 AM

(ICACC-S6-025-2017) Structure and Electrochemical Properties of a New Lithium Iron Vanadate

Y. Benabed*[1]; L. Castro[2]; V. Razafindramananan[2]; N. Penin[2]; J. Darriet[2]; M. Dolle[1]; 1. University of Montreal, Canada; 2. Institut de Chimie de la Matière Condensée de Bordeaux, France

Iron vanadates have been widely explored as possible electrode material for Li batteries. Up to now, the ternary Li_2O-Fe_2O_3-V_2O_5 phase diagram only displays two lithium iron vanadium oxides with a similar spinel structure. Our recent investigations allow us to identify two new phases in this system. One of these compounds will be discussed in this presentation. Using the results obtained from DSC-TGA analyses, the synthesis protocol was improved to obtain a pure phase. Then, single cristals were grown and the crystal structure was solved by refinement of the collected XRD data on a KappaCCD diffractometer at 293.15K. The compound was then mixed with carbon black and PVdF to prepare an electrode, which was tested against Li metal. The electrochemical properties were tested at different cycling rate and using variable cut-off voltages. The choice of the good electrochemical window allows to obtain a reversible capacity of 100 mAh/g.

9:40 AM

(ICACC-S6-026-2017) Lithium Recovery from Used Li-ion Batteries using an Innovative Electrodialysis Method with a Lithium Ionic Superconductor

T. Hoshino*[1]; 1. National Institutes for Quantum and Radiological Science and Technology (QST), Japan

Lithium (Li) is rapidly becoming a valuable commodity. In recent years, the industrial importance of Li has increased due to its use in Li-ion batteries. For example, large-sized Li-ion batteries are used as power supplies for electric vehicles. Thus, I have developed new method for the recovery of Li from a solution of used Li-ion batteries using a Li ionic superconductor functioning as a Li-ion separation membrane (LISM). Only Li ions were successfully recovered from a solution of used Li-ion batteries through the LISM; other ions did not permeate the membrane. In this study, $Li_{0.29}La_{0.57}TiO_3$ was selected as the LISM because it exhibits high durability against water. The area and thickness of the LISM are 25 cm^2 (5.0 cm × 5.0 cm) and 0.5 mm, respectively. The positive side of the dialysis cell was filled with used Li-ion battery solution. Then the negative side was filled with distilled water. The applied dialysis voltage was 5 V, and the duration of dialysis was 72 h. The electrodes were Ti–Ir alloy on the positive electrode side and SUS316 on the negative electrode side, and electrode area was 16 cm^2. The Li recovery ratio increased with electrodialysis time, reaching approximately 10% at 72 h of dialysis. This new method for recycling Li-ion batteries shows good energy efficiency and is easily scalable.

Thermal Energy Conversion and Energy Storage VIII

Room: Tomoka A
Session Chair: Yongsheng Hu, Institute of Physics, Chinese Academy of Sciences

10:20 AM

(ICACC-S6-027-2017) Regulating Electrochemical and Chemical Reactions in Metal-oxygen and Li-ion Batteries (Invited)

J. Liu*[1]; 1. Shanghai Institute of Ceramics, CAS, China

There are very complicated electrochemical and chemical reactions occurred in charge and discharge processes in metal-oxygen and Li-ion batteries. Regulation of these reactions is an important strategy to optimize electrochemical performance by changing discharge products and charge reaction mechanisms. First-principles thermodynamics calculations combining with experimental verifications were performed to unravel regulating mechanisms of electrochemical and chemical reactions in metal-oxygen and Li-ion batteries. In this talk, several examples such as Li-O$_2$, Na-O$_2$, Li-ion cathode, and solid-state electrolyte materials were taken to present possible regulating mechanism in order to improve electrochemical performances of reducing overpotential and improving cycling stability. Our calculations reveal surface-acidity-dependent catalytic activity in reducing overpotentials of Li-O$_2$ batteries, electrolyte-controlled discharge product distribution in Na-O$_2$ batteries, and discharge-voltage-dependent structural evolution in Li-ion cathode materials. Our calculated predictions were further confirmed by our comparative experiments.

10:50 AM

(ICACC-S6-028-2017) Redox processes and materials design for lithium-sulfur and lithium-oxygen batteries (Invited)

Y. Lu*[1]; Q. Zou[1]; Z. Liang[1]; Z. Li[1]; 1. The Chinese University of Hong Kong, Hong Kong

Lithium-sulfur (Li-S) and lithium-oxygen (Li-O$_2$) batteries have received extraordinary attention owing to their high theoretical gravimetric energy densities. However, practical applications of these battery systems have been hindered by poor cycle life and low round-trip efficiency. In this presentation, we will discuss fundamental redox processes and design strategies for Li-S and Li-O$_2$ batteries. We exploit various spectroscopic techniques coupled with single-cell electrochemical characterizations to probe the Li-S and Li-O$_2$ reactions. We will examine solvent-dependent Li-S reaction pathways and the effect of host materials on sulfur-immobilization by operando UV-vis spectroscopy. The enhancement mechanism and the nature of the bonding between the host materials and polysulfides will be discussed. In addition, we will discuss how the redox mediator influence the reaction stability of Li-O$_2$ reactions. The effect of redox mediator on charging and cycling instabilities in Li-O$_2$ batteries will be discussed.

11:20 AM

(ICACC-S6-029-2017) Amorphous Li$_2$O$_2$: Chemical Synthesis and Electrochemical 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$_2$O$_2$. The morphology of Li$_2$O$_2$ 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$_2$O$_2$ 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$_2$O$_2$ because of the rarity of the compounds with high purity. Here, amorphous Li$_2$O$_2$ 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$_2$O$_2$ demonstrates enhanced charge transport properties and increased electro-oxidation kinetics, manifesting itself a desirable discharge phase for high-performance Li-O$_2$ batteries.

11:50 AM

(ICACC-S6-030-2017) The Interactions Between Lithium Polysulfides and N-doped Graphene: A Density Functional Theory Study

L. Yin*[1]; J. Liang[1]; F. Li[1]; H. Cheng[1]; 1. Insitute of Metal Research, Chinese Academy of Sciences, China

Nitrogen-doped graphene (N-G) was widely reported be able to suppress the shuttling of lithium polysulfides (LiPSs), thus improving the cycling stability of a Li-S battery. However, considering the many possible doping configurations that may exist in N-G and the difficulty of controlling the synthesis of N-G with specific configurations, it would be difficult to validate experimentally which doping configuration in N-G works as an effective immobilizer for LiPSs in a Li-S battery. Thus, it is important to theoretically study the interactions between N-G with different doping configurations and LiPSs to obtain the optimal design of N-doped carbons for Li-S batteries. By using DFT calculation, we found that, only N-G with clustered pyridinic N-dopants (CPD-N) strongly binds to LiPSs with larger binding energies than those between LiPSs and electrolyte solvent molecules. Therefore, it is highly desirable to introduce as many of CPD-N as possible into the N-G or directly choose the materials with a large number of CPD-N, such as polymeric carbon nitride (p-C$_3$N$_4$), as electrode materials for Li-S batteries. In fact, the calculated E$_b$ between different LiPSs and p-C$_3$N$_4$ is about 1.5 times of those between LiPSs and electrolyte solvents. As predicted, we found that the Li-S system with a hybridized electrode consisting of p-C$_3$N$_4$ and graphene shows an outstanding cycling performance.

S7: 11th International Symposium on Nanostructured Materials: Functional Nanomaterials and Thin Films for Sustainable Energy Harvesting, Environmental and Health Applications

Solar Fuels III

Room: Coquina Salon A
Session Chair: Saim Emin, University of Nova Gorica

8:30 AM

(ICACC-S7-020-2017) Interfacial architecture of perovskite solar cells-MAPbX$_3$, CsPbX$_3$, and MASnPbX$_3$- (Invited)

S. Moriya[1]; K. Hamada[1]; D. Hirotani[1]; Y. Ogomi[1]; T. Ripolles[1]; Q. Shen[2]; T. Toyoda[2]; K. Yoshino[3]; T. Minemoto[4]; S. Pandey[1]; T. Ma[1]; S. Hayase*[1]; 1. Kyushu Institute of Technology, Japan; 2. The University of Electro-communications, Japan; 3. University of Miyazaki, Japan; 4. Ritsumeikan University, Japan

Hybrid perovskite solar cell (PVK solar cell) have attracted interest because of the high efficiency. In this presentation, the relationship between photovoltaic performances and interface structures on MAPbI$_3$, CsPbI$_3$, and MAPbI$_3$ are reported. From the results using quartz crystal microbalance sensor (QCM) and XPS, we concluded that the Ti-OH on the surface of TiO$_2$ reacts with I-Pb-I to form Ti-O-Pb-I at the interface between TiO$_2$ and perovskite layer. It was found that Jsc also increased with an increase in the Ti-O-Pb density. We concluded that the interface between TiO$_2$ and perovskite layer has passivation structure consisting of Ti-O-Pb-I which decreases the trap density of the interfaces and suppresses charge recombination (2, 3, 4). It was found that adsorption density of PbCl$_2$ on TiO$_2$ surface was higher than that of PbI$_2$. This may explain partially the high efficiency when the perovskite layer was fabricated by one step process consisting of MAI and PbCl$_2$ solution. The interface of hole transport layer/perovskite layer was passivated with organic amines in the same way (5). The passivation was also effective for increasing Voc and Jsc (Efficiency: 17.5%). This was explained by the results of transient absorption spectroscopy. In addition, enhancement of mixed metal perovskite (MAPbSnI3) showing photoconversion in IR region are reported from the view point of interface architecture (6).

9:00 AM

(ICACC-S7-021-2017) Composite Nanostructures for High-Efficiency Excitonic Solar Cells (Invited)

A. Vomiero*[1]; 1. Lulea University of Technology, Sweden

The typical photoanode in dye- and quantum dot- sensitized solar cells is composed of a wide band gap semiconductor, which acts as electron transporter for the photoelectrochemical system. Anatase TiO$_2$ nanoparticles are one of the most used oxides and are able to deliver the highest photoconversion efficiency in this kind of solar cells, but intense research in the last years was also addressed to ZnO and other composite systems. Modulation of the composition and shape of nanostructured photoanodes is key element to tailor the physical chemical processes regulating charge dynamics and, ultimately, to boost the efficiency of the end user device, by favoring charge transport and collection, while reducing charge recombination. We investigated several systems: (i) TiO_2 nanoparticles / ZnO nanowires; (ii) Multiwall carbon nanotubes (MWCNTs) / TiO_2 nanoparticles; (iii) TiO_2 nanotubes; (iv) Hierarchically self-assembled ZnO sub-microstructures. Both dye molecules and semiconducting quantum dots were applied as light harvesters. Possible tailoring of structure and morphology of the photoanodes and of the quantum dots, and their implication in improving the functional properties of these kinds of excitonic solar cells will be discussed in detail.

9:30 AM

(ICACC-S7-022-2017) Design, Synthesis, and Structure-Function Relationship of Amphiphilic Organic Ligands for Stable Nanoparticle Dispersions in various solvents (Invited)

H. Kamiya*[1]; 1. Tokyo University of Agriculture and Technology, Japan

Inorganic nanoparticles such as metals, semiconductors, and oxides have recently been recognized as promising functional materials which have physicochemical properties, including electrical, optical and mechanical properties. In order to take advantage of the unique property of nanoparticles, it is necessary to obtain stable dispersion of nanoparticles in several solvents and polymer thin film, hopefully in high concentration. In most cases, the colloidal stability is achieved by modifying nanoparticles with a monolayer of organic ligands. However, the choice of suitable organic ligands for each solvent and particle generally requires trial-and-error approaches and can be problematic. We designed the amphiphilic organic ligands with different lengths of alkyl and ethylene glycol chains and a phosphoric acid group for TiO_2 and NH_2 group for silver nanoparticles as an anchoring moiety. A series of the ligands were synthesized in 3 steps from commercially available alkyl bromides. The ligands structures and purities were characterized by NMR analysis. TiO_2 and Ag nanoparticle aqueous suspension were modified with the synthesized ligands by mechanical stirring. The modified nanoparticles were aggregated in the aqueous solution, which were then collected by centrifugation and washed with water to remove free ligands and dried in vacuum.

10:00 AM

(ICACC-S7-023-2017) Luminescent silica nanoparticles to increase the efficiency of silicon solar cells (Invited)

F. Enrichi*[1]; 1. Museo Storico della Fisica e Centro Studi e Ricerche Enrico Fe, Italy

Sub-wavelength nanoparticles deposited on the surface of solar cells may increase the efficiency of the cell by increasing the light trapping into the active layer. Moreover, energy harvesting may be improved by spectral conversion layers, to convert poorly absorbed frequencies of the solar spectrum into the region of maximum absorption of the cell. With the idea of combining these two approaches, we report the preparation and application of efficient downshifting silica nanoparticles. Amino-functionalized silica nanoparticles were obtained by a modified Stober synthesis, while the luminescence was induced by a subsequent thermal annealing. Nanoparticle size and spectral properties can be widely and finely tuned as a function of the synthesis parameters. Their application to increase the efficiency of PV solar cells is discussed.

Solar Fuels IV

Room: Coquina Salon A
Session Chair: Alberto Vomiero, Lulea University of Technology

10:30 AM

(ICACC-S7-024-2017) Multifunctional materials for electronics and photonics (Invited)

F. Rosei*[1]; 1. INRS, Canada

The bottom–up approach is considered a potential alternative for low cost manufacturing and patterning of nanostructured materials. It is based on the concept of self–organization of nanostructures on a substrate, and is emerging as an unconventional paradigm for traditional top down fabrication used in the semiconductor industry. We demonstrate various strategies to control nanostructure assembly at the nanoscale. Our overarching objective is to design and synthesize new materials that will help address the energy challenge. We study, in particular, multifunctional materials, namely materials that exhibit more than one functionality, and structure/property relationships in such systems, including for example: (i) control of size and luminescence properties of semiconductor nanostructures, synthesized by reactive laser ablation ; (ii) we devised new strategies for synthesizing multifunctional nanoscale materials to be used for applications electronics and photovoltaics.

11:00 AM

(ICACC-S7-025-2017) Sustainable solar fuels using earth abundant materials (Invited)

T. Andreu*[1]; 1. IREC, Spain

An important approach towards an efficient and sustainable economy is storing solar energy into chemical fuels through photoelectrochemical (PEC) water splitting and CO_2 reduction. Cheap and earth abundant materials, optimal band gap and electrolyte adaptability is mandatory for large scale industrialization and deployment of PEC technology. In this talk, it will be presented the intrinsic limitations of several semiconductors and the strategies to enhance solar-to-fuel efficiencies, considering its light absorption, charge separation and charge transfer. To upscale the technology, it will be needed the optimization of the individual key parameters such as the electronic transference of the (photo)anode and (photo)cathode, their overpotentials to oxygen evolution reaction (OER), hydrogen evolution reaction (HER) or carbon dioxide electroreduction (CO_2R) as well as the device design.

11:30 AM

(ICACC-S7-026-2017) Photoelectrochemical Water Splitting Studies with Nanostructured n- and p-Type Semiconductor Electrodes (Invited)

S. Emin*[1]; M. Valant[1]; 1. University of Nova Gorica, Slovenia

Photoelectrochemical water splitting has been demonstrated as a promising way to efficiently split water. Currently, solar-to-hydrogen conversion efficiency using state-of-the-art material combinations in PEC system is in the order of 7%. Fabrication of nanostructured materials with unique morphologies and compositions is an important factor to fully utilize the possibilities in this field. We will present different strategies for the preparation of nanostructured metal oxide thin films by using electrodeposition and wet-chemistry techniques. Focus will be given on the preparation of ZnO and CuO thin films where intermediate phases like $Zn(OH)_8Cl_2.H_2O$ and CuX (X=Br, Cl) were electrodeposited. Wet-chemistry synthesis techniques will be also explored for the preparation of nanostructured WO_3 and a-Fe_2O_3 thin films. Especially, the hot-pyrolysis technique for the preparation of colloidal W and Fe/Fe-oxide nanoparticles will be shown. Spin-coating of W and Fe/Fe-oxide NPs onto optically conductive substrates and subsequent heat treatment of the obtained films was found to be a convenient way for the preparation of nanostructured WO_3 and a-Fe_2O_3 thin films. By using the present approach, we prepared textured WO_3 and thin films with different thicknesses ranging from 200 to 900 nm. The obtained textured ZnO, CuO, WO_3 and a-Fe_2O_3 thin films were used in PEC water splitting studies

11:50 AM

(ICACC-S7-027-2017) Mo/Cu(InGa)Se_2 interface control for enhanced solar cell performance (Invited)

W. Kim*[1]; S. Kwon[1]; J. Koo[1]; 1. Yeungnam University, Republic of Korea

The chalcopyrite Cu(InGa)Se2 (CIGS) photovoltaic absorber was fabricated traditionally by three-stage co-evaporation or selenization of metal-precursors. The CIGS thin films deposited by three-stage co-evaporation were reported to have a (220) plane preferred crystal orientation, whereas the CIGS deposited by selenization of the metal-precursor has a random or (112) plane preferred orientation. The CIGS (220) plane was more effective in the diffusion of Cd2+ ions into the surface of the CIGS layer during chemical bath deposition of the CdS buffer layer to form a beneficial buried homojunction at the CIGS surface than the CIGS (112) plane. In this paper, a thin $MoSe_2$ layer (~50 nm) was formed intentionally by exposing a glass/Mo substrate to Se flux at 550°C in a vacuum evaporator. Then, CIGS was deposited by using a conventional three-stage co-evaporation. The formation of thin $MoSe_2$ layer was confirmed by XRD, TEM, EDS and XPS. The pre-formed thin $MoSe_2$ layer suppressed the preferential growth of $(InGa)_2Se_3$ on the (300) plane and therefore suppressed the CIGS growth on the (220) plane, which are characteristic of the three-stage CIGS deposition process. Furthermore, the addition of the $MoSe_2$ layer resulted in a cell efficiency improvement, primarily due to the enhanced fill factor and open circuit voltage.

12:10 PM

(ICACC-S7-028-2017) Nanostructured Zinc Oxide: Enabling multi-functionality through modulation of material aspect (Invited)

I. Concina*[1]; 1. Luleå University of Technology, Sweden

In the perspective of conjugating green chemistry as a tool for environment respectful material preparation and functional versatility, ZnO appears as a candidate holding an actual potential as flexible platform in different applications: its polymorphism, together with a wide band gap and high electron mobility, made this semiconducting metal oxide exploited since long ago in many fields, spanning from photocatalysis to gas sensing, photovoltaics, biofouling. This lecture will focus on simple and cheap synthetic strategies delivering ZnO nanostructures with enabled multi-functionality in three crucial fields: photovoltaics, gas sensing and photocatalysis. We will show how powerful simple chemistry can be in preparing single-crystal ZnO hierarchical nanostructures with tuned opto-electronic features which suit light management and charge transport needs. Emphasis will be given to green preparation approaches, material potential as well as material limits, correlation between nanostructures aspect and functionalities, in the bigger picture of application of green chemical concepts to functional nanomaterials.

12:30 PM

(ICACC-S7-029-2017) Stability Studies Of Doped Organic-Inorganic Hybrid Perovskites

I. M. Asuo*[1]; 1. Institut national de la recherche scientifique (INRS-EMT), Canada

Organic-inorganic hybrid perovskite (OIHP) materials exhibit outstanding photovoltaic and optoelectronic properties such as tunable optical properties. In the general device architecture, the OIHP material is a thin film sandwiched between two electrodes; the quality of which is of crucial importance to the photovoltaic cell performance. Within half a decade, OIHPs have attracted immense attention in the photovoltaic research community mainly because of the rapid improvement of their power cell efficiencies from 3.8% to 20%. However, the instability and the toxicity of lead-based perovskites materials in ambient humidity and in water have impeded the commercialisation of this type of photovoltaic cells. In this work, we present an alternative way of improving the stability of $CH_3NH_3PbX_3$ by doping with thiocynate ions. The thin films were deposited under ambient conditions via spin coating of the precursors followed by a solvent treatment. Scanning electron microscopy, X-ray diffraction and Raman spectroscopy were used to observe the morphology, the chemical composition and structure, as well as the crystallinity of the grown thin film. The optimization of the optical and electrical properties of the thiocyanate-doped $CH_3NH_3PbX_3$ films and the performance of the photovoltaic devices built with these films will be discussed in details.

S8: 11th International Symposium on Advanced Processing and Manufacturing Technologies for Structural and Multifunctional Materials and Systems (APMT11)

Novel Ceramic Processing II

Room: Coquina Salon B
Session Chairs: Zhengyi Fu, Wuhan University of Technology; Richard Sisson, WPI

8:30 AM

(ICACC-S8-009-2017) Aquacasting: A new UV based reactive tape casting concept (Invited)

T. Graule*[1]; C. Durif[1]; P. Ozog[1]; D. Kata[2]; 1. Empa, Switzerland; 2. AGH University of Science and Technology, Poland

Aquacasting is a novel approach for tape casting of gradient structures incorporating water based binder and UV curable, reactive latex dispersions. The process was described recently by Durif et al., based on zirconia powder applications. Within the last year we further applied the technology for the shaping of nano- and microporous alumina parts with gradient structures. Zeta potential measurements were carried out to prepare stable and homogeneous dispersions and the solid load was adjusted by rheological measurements. The impact of different concentrations of the photoinitiator on the curing process was investigated, The problem of foam formation in the dispersion was eliminated by using antifoaming agent and by adjusting its concentration as well. As final result fully cured and flexible green tapes with the maximum thickness of 400 μm and crack free sintered samples were obtained. The presentation gives a general outline about the opportunities to apply Aquacasting for different types of zirconia and alumina powders.

9:00 AM

(ICACC-S8-010-2017) The Challenge and the Opportunity for Manufacture of Zinc Magnesium Oxide Ceramics

J. Yang*[1]; T. Lin[1]; J. Young[1]; B. Manett[1]; E. Medvedovski[2]; 1. SCI Engineered Materials, USA; 2. Endurance Technologies Inc., Canada

Zinc Magnesium Oxide (ZnxMg1-xO) semiconducting ceramics have received particular attention due to its versatility in optoelectronics for solar and photonic applications. In CIGS thin film solar cells, ZnxMg1-xO transparent conductive films have been developed as the CdS replacement for the buffer layer to achieve better band alignment between p-type active absorption layer and n-type electrode layer. In Photonics, with the addition of MgO, the wide bandgap of ZnO can be driven down to near ultraviolet region for LED and photodetectors application. Sputtering using ceramic targets provides more consistent film compositions and structure with higher properties vs. reactive sputtering from metallic targets. The manufacturing challenges of ZnxMg1-xO ceramic sputtering targets include limited solubility of Mg in ZnO, as well as the reaction of MgO with water in colloidal processing. In this work, we introduce a process of ZnxMg1-xO ceramic targets with Mg contents ranging from 5% to 25% through pressureless sintering.

9:20 AM

(ICACC-S8-011-2017) Conductive glass-carbon composites for heating elements produced by two component powder injection molding (PIM)

J. C. Schilm*[1]; A. Rost[1]; A. Mannschatz[1]; A. Mueller-Koehn[1]; T. Moritz[1]; A. Michaelis[1]; 1. Fraunhofer Gesellschaft, Germany

In conventional glass shaping processes, glass is heated considerably above the glass transition point T_g, and formed by pressurized gas, the own weight or cooled Steel tools which involves high temperatures and high energy consumption. Shaping processes for glass components usually require high temperatures where the glass melt has a low viscosity. This in turn makes it difficult or even impossible to manufacture complex shaped components. Powder injection molding (PIM) is exceptional suited for shaping complex components since they are produced in near net shape. In this work the PIM process has been investigated for glass powders and composites thereof. The functionalization of a glass based PIM feedstock by the composite principle is achieved by doping with particles like ceramics, carbon or coloring pigments. Results in the development of a two component PIM-process for combining glass composites with different properties such electrical conductivity and insulation are presented. The key issue is adjusting the feedstock properties to enable co-sintering of both composites. Co-sintering of components consisting of two different composites has been realized. Resistance of the conductive component can be adjusted in the range from 1 to 1000 Ohm/cm. Electrical heating experiments show that sintered glass-carbon components can reach more than 300°C.

9:40 AM

(ICACC-S8-012-2017) Manufacturing Near-Net Shaped Silicon Nitride Specimens with Aligned Microstructures via Room-Temperature Injection Molding

L. Rueschhoff*[1]; J. Youngblood[1]; R. Trice[1]; 1. Purdue University, USA

A novel processing technique of injection molding aqueous silicon nitride suspensions at room temperature is proposed to overcome common forming difficulties associated with silicon nitride and other ceramic systems. Injection molding not only allows for the production of near-net complex shapes, but also affords the opportunity to use the shear stresses during mold filling to align high aspect ratio microstructural features. In the present work, highly-loaded aqueous silicon nitride (45vol.%) suspensions have been dispersed using commercially available comb-polymer water reducing admixtures, commonly used in cementitious mixtures. The yield-pseudoplastic rheological properties of the suspensions are desirable for injection molding and are tailored through polymer type, concentration, molecular weight, and ceramic loading. The aqueous based suspensions and low pressures (< 150 kPa) used during injection make this an economic and environmentally friendly process. Parts formed via this process possess high sintered densities (up to 98%TD) and complete conversion to the beta-silicon nitride phase, proving this as a viable process for production of robust silicon nitride parts. Finally, the use of the additive manufacturing process of direct writing ceramic suspensions will be explored as another means for the production of parts with aligned microstructures.

10:20 AM

(ICACC-S8-013-2017) New Sintering Process for High Density and Limited Grain Growth with Plastic Deformation as Dominating Mechanism (Invited)

Z. Fu*[1]; 1. Wuhan University of Technology, China

Traditionally, sintering is the densification process of powder compacts, in which interparticle pores are eliminated by atomic diffusion driven by thermal energy. The atomic diffusion may cause unavoidable grain growth, besides contributes to densification. For polycrystalline materials, higher density generally results in improvement of properties like strength and toughness, while the undesirable grain growth results in degradation. Attempts have been made to gain full density, while keep the grains with limited growth. A new method was proposed to sinter ceramic powders at a lower temperature close to the onset point of grain growth, at the same time apply a higher pressure to the compact. Fully dense micro-sized or nano-sized ceramics with limited grain growth were made by the new route. The sample shows excellent mechanical properties. The plastic deformation under high pressure and at high temperature is proved to be the dominating mechanism for the densification process.

10:40 AM

(ICACC-S8-014-2017) Observation of particles in silica slurry with high-concentration

S. Tanaka*[1]; Y. Nagasawa[1]; 1. Nagaoka University of Technology, Japan

Particles behavior in a high-concentration slurry must be fully understood for controlling particle packing structure in a powder compact. Rheological behavior are usually measured for characterization of the dispersed state. We examine the internal structure of dispersed or flocculated slurries in the high-concentration by the confocal laser scanning fluorescent microscopy (CLSFM). Interaction between particles was measured by the colloidal probe atomic force microscopy. Rheological measurement was also conducted for characterizing slurries. In experimental, mono-dispersed silica spherical particles and glycerol aqueous solutions containing a little fluorescent dye were used. Matching refractive index of glycerol solution with silica particles, we are able to observe the internal state of particles directly. In slurry, either PAA (Polyacryl ammonium acid) or PEI (Polyethylene imine) was used as an additive. CLSFM observation showed that particles were dispersed in slurries with PEI. In contrast, in a PAA added slurry, small agglomerate of several particles were observed well. The interaction force between particles in PEI system was the repulsive force, the result explained the observation result. Rheological measurement suggested that PEI or PAA added slurries shows Newtonian or shear-thinning behavior slightly, respectively. The particles states in the slurry were corresponded to the rheological properties.

11:00 AM

(ICACC-S8-015-2017) Synthesis of Nanocrystalline Ultrahigh Temperature Ceramic Powders via Rapid Single Step High Temperature Spray Pyrolysis

P. Foroughi*[1]; A. Behrens[1]; Z. Cheng[1]; 1. Florida International University, USA

Borides and carbides of transition metals are considered as ultrahigh temperature ceramics (UHTCs) possessing melting points above ~3000°C and they find important applications in aerospace and other fields due to their unique properties. Here, we report for the synthesis of nanocrystalline UHTCs powders using a new single step high temperature spray pyrolysis process. In this method, soluble precursors for oxides and carbon are dissolved in non-aqueous solvents and then atomized into micron-sized droplets using a nebulizer. The fine droplets are then transported by argon carrier gas furnace preheated to temperature as high as ~1675°C and go through a series of reactions including solvent removal, pyrolysis, and then in situ carbothermal (CTR) reaction all in one single pass within a minute. The high temperature spray pyrolysis incorporating in situ CTR as described is not only an inexpensive technique but also is a continuous process allowing higher production rate. Preliminary results show nanocrystalline tantalum carbide (TaC) was successfully synthesized using this technique at 1650°C from precursors of tantalum chloride, phenolic resin, and solvents of 1-pentanol (for $TaCl_5$) and ethanol. The study also reveals new knowledge about the composition-processing-morphology interrelationships in the formation of nanocrytalline UHTCs via this method.

11:20 AM

(ICACC-S8-016-2017) Numerical Analysis of Inhomogeneous Behavior in Friction Stir Processing by Using a new Coupled Method of MPS and FEM

H. Serizawa*[1]; F. Miyasaka[2]; 1. Osaka University, Japan; 2. Osaka University, Japan

As the method for estimating the residual stress produced in friction stir processing (FSP), a combined method between the moving particle semi-implicit method (MPS) and finite element method (FEM) has been developed and the flow behavior during the friction stir welding (FSW) of two aluminum alloy plates was studied. The computational results revealed that the longitudinal plastic strain near the FSW tool is not governed by only the temperature distributions and the influence of plastic flow should be taken into account. However, because the heat input density of volumetric heat source model for thermal analysis of FEM is obtained from the average temperature of the heat generation area computed from MPS, the detailed information in MPS result might be lost. So, in this research, a new coupled method of MPS and FEM was developed where the heterogeneous heat density distribution obtained from MPS was directly employed as the heat source density for thermal analysis and the applicability of this coupled method was examined. From the elastic-plastic FEM analysis using the transient temperature distributions obtained by the coupled method, it was revealed that the longitudinal plastic strain distributions near the tool can be predicted by employing the concept of boundary temperature which is as same as the annealing temperature in the welding.

11:40 AM

(ICACC-S8-017-2017) The Aqueous Corrosion Response of Ti(C,N)-Ni_3Al Cermets With Various Reaction-Formed Binder Contents

Z. Memarrashidi[1]; K. P. Plucknett*[1]; 1. Dalhousie University, Canada

In the current work the corrosion mechanisms of Ti(C,N) based cermets are analysed in a 3.5 wt.% NaCl aqueous solution. Cermets were prepared with 20 or 30 vol.% of in-situ reaction-formed Ni_3Al metal binder. The assessment of corrosion involved a variety of electrochemical measurements, including open circuit potential, potentiodynamic polarisation and cyclic polarisation, together with systematic analysis of the post-corrosion solution chemistries using inductively coupled plasma optical emission spectroscopy. The corrosion potential and current density were determined, following Tafel extrapolation, allowing estimation of the corrosion rates. It is demonstrated that the main corrosion mechanism for this type of cermet is mainly dissolution of the metallic phase during oxidation attack, and thus this response increases with the amount of binder in the cermet. It was also observed that addition of N content into the ceramic phase is beneficial to the corrosion characteristic of TiC based cermets, through significant refinement in the grain size of the hard phase.

S9: Porous Ceramics: Novel Developments and Applications

High SSA Ceramics I

Room: Coquina Salon G

Session Chair: Paolo Falcaro, Graz University of Technology

8:30 AM

(ICACC-S9-027-2017) Mesoporous K/Fe-Al-O Nano-Fiber Catalyst by Electrospinning (Invited)

G. S. Grader*[1]; G. E. Shter[1]; O. Dinner[1]; O. Elishav[1]; V. Halperin[1]; V. Beilin[1]; 1. Technion - Israel Institute of Technology, Israel

Sub-micron sized fibers are of great interest in the field of heterogeneous catalysis. Such fibers can potentially provide superior transport properties between the catalytic sites and the bulk. Providing the fibers with internal porosity that allows for larger catalytic surface, better mass transport and catalyst efficiency and lower deactivation rates is a major objective in this research. In this work we focus on the synthesis of K/Fe-Al-O ceramic fibers with mesoporous structure. Fibers mats were prepared by electrospinning a solution of metal acetylacetonates in an organic solvent. The specific metal precursors and controlled thermal treatment gave rise to a promising mesoporous structure with fibers diameter in the 100-800 nm range. The formation of this structure was investigated and explained in terms of shrinkage rate vs. weight loss rate and metal acetylacetonates phase transitions. The increased understanding in fiber morphological evolution is a step towards new possibilities in mesoporous fiber formation and heterogeneous catalysis.

9:00 AM

(ICACC-S9-028-2017) Quantitative evaluation of adsorption-photodecomposition property on mesoporous silica-titania (Invited)

M. Inada*[1]; K. Hayashi[1]; J. Hojo[1]; 1. Kyushu University, Japan

Mesoporous silica-titania has a high photocatalytic activity for removal of acetaldehyde gas because titania nanoparticles are highly dispersed into mesoporous silica with high specific surface area. In this study, the adsorbability and photodecomposition property were evaluated quantitatively from kinetic equations. The calculation from pseudo-first order equation did not agree with experimental results, suggesting the necessity of adsorption-step. Therefore, new kinetic equations were constructed from three elemental processes, consisting of direct photodecomposition, adsorption and adsorption-photodecomposition. Experimental data were well reproduced by the improved model. The adsorbability strongly affects the removal of acetaldehyde in the early stage from gas phase and the photodecomposition after strong adsorption is important for the complete decomposition of acetaldehyde.

9:30 AM

(ICACC-S9-029-2017) Ultraviolet to near infrared-light-driven photocatalysis of mesoporous SiO_2-TiO_2 deposited with Au nanorods

T. Okuno*[1]; G. Kawamura[1]; H. Muto[1]; A. Matsuda[1]; 1. Toyohashi University of Technology, Japan

Many approaches have been proposed such to make TiO_2 sensitive to the visible and even near-infrared light. Among various light-trapping techniques, the use of metallic nanostructures that support surface plasmon resonance (SPR) is an efficient route for broadening and enhancing the light absorption of TiO_2 through electric field enhancement and hot electron injection. In these studies, mainly Au nanospheres (NSs) are adsorbed on or embedded in TiO_2, while Au nanorods (NRs) with broadly tunable aspect-ratio dependent longitudinal SPR (LSPR) were integrated with TiO_2 only in a limited number of studies. In this study, Au NSs or Au NRs were selectively deposited on TiO_2 with mesoporous SiO_2 containing TiO_2-nanocrystals by photodeposition method. The effect of the shape of Au nanoparticles on the photocatalysis was investigated.

High SSA Ceramics II

Room: Coquina Salon G

Session Chair: Miki Inada, Kyushu University

10:10 AM

(ICACC-S9-030-2017) Ceramics for Metal-Organic Frameworks (MOFs) based devices (Invited)

P. Falcaro*[1]; 1. Graz University of Technology, Austria

Metal organic frameworks (MOFs) are a class of ultra-porous materials with exceptionally high accessible surface area due to the framework produced by the inorganic nodes coordinated by organic ligands. An increasing number studies are investigating MOFs for a variety of functional applications including sensing, microelectronics, energy production, drug delivery and microfluidics. However, MOF-based device fabrication requires synthetic protocols for the controlled functionalization of the porous crystals and the integration in suitable platforms. Ceramics have been recently employed for the synthesis, crystal engineering, functionalization, and patterning of MOFs. This presentation will summarize the recent progress in this emerging field.

10:40 AM

(ICACC-S9-031-2017) Mesoporous 3D polymer derived TiN/Si_3N_4 nanocomposites monoliths as platinum supports for H_2 generation from $NaBH_4$

A. Lale*[1]; U. B. Demirci[1]; S. Bernard[1]; 1. Institut Europeen des Membrane, France

PEMFC-based systems are alternatives to current energy conversion technologies due to their potential to directly convert H_2 into electrical energy but these still have several issues. One of the most critical issues faced by PEMFCs is the H_2 source to meet the energy requirements for civil vehicle applications. 95% of H_2 is produced from CH_4 which means evolution of CO_2. Liquid-phase hydrogen carriers like alkaline solutions of $NaBH_4$ are attractive alternatives because of zero emission and high efficiency. However, the hydrolysis of NaBH4 is exothermic, very alkaline and requires catalysts. Hence we develop porous TiN/Si_3N_4 nanocomposites (NC) for the growth of metallic Pt (<1 wt%) nanoparticles and the use this nanocatalysts for the catalytic hydrolysis of $NaBH_4$. The NC is synthesized by coupling the polymer-derived ceramic route with a templating approach using activated carbon as mesoporous template and polytitanosilazane as preceramic polymer. Due to the unusually high SSA (=1200 m^2/g) of the mesoporous nanocomposite monoliths, they demonstrate very high performance. Their performance for the generation of hydrogen from $NaBH_4$ is compared to organosilicon-derived SiC, Si_3N_4 and SiCN materials. Results show that the metal-supported NC are very interesting nanocatalysts in pursuit of practical implantation of $NaBH_4$ as a hydrogen source for fuel cell.

11:00 AM

(ICACC-S9-032-2017) Ultralight polymer-derived ceramic aerogel for effective wide bandwidth electromagnetic absorption properties

G. Shao*[1]; W. Zhao[1]; R. Zhang[2]; L. An[3]; 1. Zhengzhou University, China; 2. Provincial key laboratory of Aviation Materials and Application Technology, Zhengzhou University of Aeronautics, China; 3. University of Central Florida, USA

Electromagnetic (EM) pollution and interference is severely impacting the information security and the health of humans due to the rapid development of wireless equipments, electronic devices and telecommunication in both civil and military fields. Electromagnetic absorption material (EAM) is an effective method to solve this problem by attenuating the unwanted electromagnetic wave into thermal energy. In this presentation a novel electromagnetic absorption material, polymer-derived ceramic aerogel (PDCA), with ultralight weight (0.19g/cm^3), strong absorption and wide absorption bandwidth was synthesized by hydrosilylation gelation and freeze drying techniques. The electromagnetic absorption property of PDCA is easily tuned by varying pyrolysis temperatures. The minimum RL is -43.37 dB@7.6 GHz, -42.01 dB@12.5 GHz, and -31.69 dB@17.3 GHz of the SiCN aerogels pyrolyzed at 1000°C, 1200°C and 1400°C, respectively, with the relative effective band width of 3.8 GHz, 6.6 GHz and 4.2GHz. The excellent EM absorption properties indicated that polymer derived SiCN aerogel is a promising EM absorption material for practical applications.

11:20 AM

(ICACC-S9-033-2017) Polymer-derived Si-O-C-N aerogels as sorbent materials for water purification

E. Zera*[1]; E. Brancaccio[3]; M. C. Bruzzoniti[2]; A. Geleta[1]; G. D. Sorarù[1]; 1. University of Trento, Italy; 2. University of Turin, Italy; 3. SOLID Power SpA, Italy

Polymer derived ceramic aerogels belonging to the Si-O-C-N system are synthesized crosslinking a preceramic polymer in a diluted solution followed by supercritical drying and pyrolysis in controlled atmosphere. These aerogels show hierarchical porosity ranging from some microns to few nanometers together with high specific surface area in the range 100 - 300 m^2/g. Moreover their structure contains several types of chemical bonds such as Si-O, Si-N, Si-C and graphitic C=C bonds. This unusual combination of hierarchical porosity, high SSA and broad range of chemical functionalities makes these PDC aerogels ideal candidates as sorbent materials for water purification. In this work we proposed to use SiOC and SiCN aerogels pyrolized in inert (N_2) and active (NH_3, CO_2) atmospheres as adsorbents for heavy metal ions (Cr and Ni) to purify contaminated water. The materials showed rapid and simultaneous adsorption of both Ni and Cr(III) cations and Cr(VI) anions, suggesting the presence of a wide variety of active groups on the surface, related to the starting pre-ceramic polymer and to the atmosphere of pyrolysis.

S10: Virtual Materials (Computational) Design and Ceramic Genome

Modelling by DFT methods

Room: Ponce DeLeon

Session Chairs: Per Eklund, Linkoping University; Katsuyuki Matsunaga, Nagoya University

8:30 AM

(ICACC-S10-024-2017) Theoretical insights and predictions of mechanical properties and electronic structure of ternary layered borides MoAlB using density functional theory (Invited)

Y. Bai*[1]; X. Qi[1]; N. Li[1]; F. Kong[1]; X. He[1]; R. Wang[1]; 1. Harbin Institute of Technology, China

Density functional theory is employed to provide the theoretical insights and predication of crystal structure, electronic structure and mechanical properties of a ternary nanolaminated and layered transition metal boride, MoAlB. The calculated configuration including lattice parameters, internal coordinates, bond length and bond angle well matches with experiments. The metal-like electronic structure, e.g. finite density of states at Fermi energy (2.75 States/eV/Unit cell), contributes to the physical origin of high electric conductivity of MoAlB. The strong covalent bonding is present between B and B atoms, as well as Mo and B atoms, while the Mo-Al and Al-Al bonds are relatively weak. From the estimated full set of second-order elastic constants, the bulk moduli B, shear moduli G, Young's moduli E and Poisson ratio μ are 207 GPa, 137 GPa, 336 GPa and 0.23, respectively. Of much importance, the low G/B ratio of 0.66 provides some evidence for the observed high damage tolerance. The vacancy formation energy of Mo, Al and B atoms are 2.71 eV/atom, 1.21 eV/atom, and 1.06 eV/atom, respectively, which results in the selective oxidation of Al and high oxidation resistance of MoAlB. The anisotropic compression behavior originates from the changing bond stiffness and bond angle as a function of pressure.

9:00 AM

(ICACC-S10-025-2017) Chemical bonding and electronic-structure in MAX phases and MXenes as viewed by density functional theory and x-ray spectroscopy (Invited)

M. Magnuson*[1]; 1. Linkoping University, Sweden

The electronic structures and chemical bonding in the 2D ceramic materials MAX-phases and MXenes were investigated by ab initio electronic structure calculations in comparison to x-ray spectroscopy. Synchrotron radiation techniques such as bulk-sensitive soft X-ray absorption and emission are shown to be particularly useful for detecting detailed symmetry in the electronic structure and yield anisotropy information about internal monolayers and interfaces. 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 to the experimental data. By varying the constituting elements and structures, 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 MAX-phase materials. Angle- and polarization-resolved measurements reveal differences in the occupation of the orbitals across and along the laminate basal plane. For MXenes, the role of functional –OH, –O and –F temination groups and their local symmetries as well as phonons at the interfaces are discussed.

9:30 AM

(ICACC-S10-026-2017) Defect Stability and Transport in SrTiO3/PbTiO3 Superlattice from Density Functional Theory (Invited)

H. Xu*[1]; L. Zhang[1]; V. Cooper[2]; P. Kent[3]; 1. University of Tennessee, USA; 2. Oak Ridge National Lab, USA; 3. Oak Ridge National Lab, USA

In this study, the stability and transport properties of oxygen vacancy in paraelectric and ferroelectric SrTiO3/PbTiO3 superlattice are investigated using density functional theory (DFT). The effects of charge state, octahedral rotation and interface on the oxygen vacancy formation energies are examined and analyzed. Different diffusion paths are identified and the vacancy diffusion energy barrier of each path is determined. In addition, the effects of number of layers in the superlattice, (e.g. 1/1, 2/2, and 3/3) on the migration energy barriers are revealed. This information is incorporated into a kinetic Monte Carlo model to predict the dominant sites of oxygen vacancy under different conditions. This study provides detailed analysis of defect stability and transport properties in oxide superlattices and fundamental insights of how to turn the defect transport to achieve desired properties.

10:20 AM

(ICACC-S10-027-2017) Modeling of Point Defect Ordering in Nonstoichiometric ZrC and TiC with B1 Structure

G. Zhou[1]; J. Wang[1]; H. Wang*[1]; J. Zhang[1]; J. Wang[1]; R. Yang[1]; 1. Institute of Metal Research, Chinese Academy of Sciences, China

Point defects significantly alter physical and mechanical properties of materials. However, point defect ordering with the atomic-scale details remains largely unclear. In this work, vacancy and interstitial behaviors in nonstoichiometric ZrC and TiC with B1 structure are systematically studied with high-throughput algorithms under the multiscale framework. Atomistic simulations indicate that the interaction between carbon vacancies (V_C) in nonstoichiometric ZrC and TiC results in ordered arrangement of V_C, depending on vacancy concentration. The stability of these ordered configurations are subsequently checked against time and temperature with meta-dynamics and objective kinetic Monte Carlo simulations. Due to low formation energy or high C migration energy, some are long-term stable, providing resistance against irradiation. For comparison, point defect ordering other solid-state systems with face-centered cubic structure is also shown, e.g., the pentavacancy in aluminum.

10:40 AM

(ICACC-S10-028-2017) Construction of Energy Diagrams of Mn^{4+} and Ce^{3+} in Oxides Based on First-Principles Calculations (Invited)

K. Ogasawara*[1]; 1. Kwansei Gakuin University, Japan

In order to improve the color rendering properties and production cost of white LEDs, red phosphors activated with Mn^{4+} are drawing attention. Although the multiplet energies of transition metal ions in crystals are generally analysed based on the Tanabe-Sugano diagrams, they cannot be directly used for the theoretical design of the novel Mn^{4+}-doped phosphors due to the following two reasons. The first reason is that the Tanabe-Sugano diagrams cannot associate the local structure to the absolute multiplet energy since they just represent the relationship between the crystal field strength and the multiplet energy. The second reason is that they can be applied only to high symmetry systems such as O_h or T_d. In order to overcome these problems, we have constructed various energy diagrams of Mn^{4+} in oxides in terms of structural parameters such as bond lengths or bond angles by performing first-principles calculations using model clusters with various symmetries such as O_h, D_{4h}, D_{3d}, C_{4v}. Since rare-earth ions such as Ce^{3+} are also important activator ions for phosphors, we have performed similar calculations for Ce^{3+} in oxides and created corresponding energy diagrams. In this talk, our recent results on the various diagrams representing the structure-property relationships of Mn^{4+} and Ce^{3+} in oxides will be presented.

11:10 AM

(ICACC-S10-029-2017) Perovskites for efficient oxygen evolution reactions: Design guidelines from ab initio calculations (Invited)

H. Tahini*[1]; X. Tan[1]; S. Smith[1]; 1. UNSW, Australia

Oxygen evolution reactions are the bottleneck for the overall water splitting reactions. While good catalysts exist for hydrogen evolution, catalysts for OER still suffer from large overpotentials which prohibit the effective utilization for electrocatalytic water splitting. Designing efficient and cheap catalysts can be greatly aided by guidelines derived via computational techniques. Here we will focus on perovskites, looking at ways for enhancing and modulating their catalytic activities via doping, and by exploring new structures such as the emerging hexagonal phases which are promising candidates. In these materials the different local environments and coordinations of the transition metal active sites display different redox potentials which can be tuned for the requirements of water oxidation.

11:40 AM

(ICACC-S10-030-2017) Ab initio Modeling of the Electronic Structures and Physical Properties of Alkali Doped Silicate Glass

K. Baral*[1]; W. Ching[1]; 1. University of Missouri, Kansas City, USA

Ab initio molecular dynamics (AIMD) has been used for the simulation of sodium silicate glass in the range of 0 - 50 mol% of Na_2O using the previously constructed near-perfect continuous random network model of amorphous SiO_2 containing 162 atoms with a periodic boundary conditions as the seed model. The simulated $(Na_2O)_x(SiO_2)_{1-x}$ glasses are critically analyzed as a function of x and validated comparing with the available experimental results. The structural, electronic, mechanical and optical properties are calculated using the first-principles density functional theory method packages (VASP and OLCAO). The calculated mass density and refractive index increase in the series with increasing concentration of sodium oxide which is in good agreement with the experimental results. The results on mechanical properties are analyzed.

S12: Materials for Extreme Environments: Ultrahigh Temperature Ceramics (UHTCs) and Nano-laminated Ternary Carbides and Nitrides (MAX Phases)

Materials Design, New Composition and Composites IV

Room: Tomoka B

Session Chairs: Anindya Ghoshal, US Army Research Laboratory; Jesus Gonzalez-Julian, Forschungszentrum Juelich

8:30 AM

(ICACC-S12-028-2017) The Science of Entropy-Stabilized Materials for Ultra-High Temperature Applications (Invited)

E. Opila*[1]; P. Hopkins[1]; D. Brenner[2]; J. Maria[2]; S. Curtarolo[3]; J. Luo[4]; K. Vecchio[4]; 1. University of Virginia, USA; 2. North Carolina State University, USA; 3. Duke University, USA; 4. University of California, San Diego, USA

The number of known materials that can be used for ultra-high temperature applications is relatively limited, even when just considering the melting point. When considering other performance parameters such as mechanical stability, thermal conductivity, thermal shock and oxidation resistance, the list of viable materials becomes even smaller. To drive this area forward beyond traditional considerations such as improved microstructures and densification, new materials classes need to be explored and characterized. With this in mind, we have been using a combination of high throughput thermodynamic and first principles modeling, experimental prototyping, and advanced bulk processing to explore the viability of using entropy-stabilized materials for these applications. This talk will provide an overview of this effort, including recent results on the synthesis and properties of new classes of entropy-stabilized carbides, nitrides, and borides, focusing on oxidation resistance. These materials are unique in the field of entropy-stabilized alloys in that they contain both entropic and ordered sublattices, which gives them the potential for materials engineering to meet the demands of ultra-high temperature applications.

9:00 AM

(ICACC-S12-029-2017) Performance Optimization of ZrB_2-$MoSi_2$-SiC Dual Composite Architectures for High Temperature Structural Applications

R. J. Grohsmeyer*[1]; G. Hilmas[1]; W. Fahrenholtz[1]; S. Failla[2]; F. Monteverde[2]; C. Melandri[2]; D. Sciti[2]; 1. Missouri University of Science and Technology, USA; 2. Institute of Science and Technology for Ceramics, Italy

Dual composite (DC) architectures have been shown to increase wear resistance while maintaining or increasing fracture toughness in WC-Co composites for room-temperature applications using multiple, discretely segregated subcomposites with individually engineered microstructures. Composites of zirconium diboride (ZrB_2) and molybdenum disilicide ($MoSi_2$) are candidates for similar DC architectures for high-temperature structural use in extreme environments due to the high-temperature ductility and oxidation resistance of $MoSi_2$. Further, additions of silicon carbide (SiC) can resolve thermal expansion mismatch in ZrB_2-$MoSi_2$ DC architectures. After systematically measuring the mechanical properties of individual ZrB_2-$MoSi_2$ subcomposites at both room temperature and 1500°C in air, multiple DC architectures were prepared by synthesizing granules of a subcomposite composition and dispersing them in a continuous matrix of another subcomposite composition with different properties. This research investigates effects of differing mechanical and oxidative properties of granule and matrix subcomposites, as well as incorporation of a weak BN-based granule coating to aid crack deflection. Flexure strength and fracture toughness at room temperature and 1500°C in air of DC architectures will be reported and compared to corresponding traditional particulate subcomposites.

9:20 AM

(ICACC-S12-030-2017) 40% increase in Cr_2AlC self-healing rate by minute Si additions

L. Shang[1]; K. Pradeep[1]; S. Sandlöbes[2]; M. to Baben[3]; J. M. Schneider*[1]; 1. RWTH Aachen Univeristy, Germany; 2. Institut für Metallkunde und Metallphysik, Germany; 3. GTT-Technologies, Germany

The effect of Si additions on the self-healing kinetics of Cr_2AlC based coatings is investigated. Oxidation experiments were performed at 1120°C in air for 4 hours for Cr_2AlC and $Cr_2Al_{1-x}Si_xC$ ($0 < x \leq 0.06$) coatings. The crystal structure, microstructure and chemical composition of the as-deposited as well as oxidized coatings have been investigated. Alloying Cr_2AlC with up to 0.7 at% Si causes an increase in self-healing rate of up to 40 ± 17%. Electron microscopy and atom probe tomography data support the notion that the here reported Si concentration induced 40% increase in self-healing rate is enabled by the Si concentration induced, and hence concomitant, increase in nucleation density of the healing agent.

9:40 AM

(ICACC-S12-031-2017) Pressureless sintering of TiC from submicron carbide powders

Z. Fu*[1]; R. Koc[1]; 1. Southern Illinois University Carbondale, USA

Utilizing laboratory-synthesized submicron carbide powders, monolithic TiC was sintered to a relative density of ~96% through pressureless sintering. Firstly, submicron TiC powders were synthesized from a carbon coated precursors method. Densification studies were performed in the temperature range of 1500 to 1800°C for one hour without applied external pressure. Microstructures and mechanical properties were characterized as a function of the sintering temperature. Results indicate that 1700°C is the optional sintering temperature. Sample sintered at 1700°C has a relative density of ~96%, fine microstructures, a high hardness, and moderate fracture toughness. With an increased sintering temperature of 1800°C, sample has a similar relative density of ~96%, coarse microstructures, and a lower hardness. A comparison of the densification for commercial available TiC powders was also performed. Commercial available TiC was not able to be sintered to a full density even at the temperature of 1800°C.

Processing-microstructure-property Relationships of Existing Systems I

Room: Tomoka B

Session Chairs: Leszek Chlubny, AGH University of Science and Technology; Joseph Halim, Drexel University

10:20 AM

(ICACC-S12-032-2017) MeB_2 ultra-high temperature ceramics (UHTCs) With Hexagonal-metal Ductile-like Behavior

E. Zapata-Solvas*[1]; D. Gomez-Garcia[2]; A. Dominguez-Rodriguez[2]; W. Lee[1]; 1. Imperial College London, United Kingdom; 2. University of Seville, Spain

UHTCs are promising candidates for hypersonic applications as a consequence of their high melting points, in excess of 3000°C for ZrB_2 and HfB_2 UHTCs. The UHTCs community has traditionally focused on development of more oxidation-resistant UHTC composites as a consequence of the poor oxidation resistance of monolithic UHTCs, which has led to the choice of SiC-reinforced MeB_2 (where Me is Zr or Hf) as baseline materials for extreme environments. However, SiC-reinforced MeB_2 are structurally unstable and show poor creep resistance above 1800°C which highlights the need for more fundamental understanding of the thermo-mechanical properties of monolithic MeB_2. Plastic properties of monolithic HfB_2 have been studied from 900 to 2000°C. Plastic deformation onset starts at temperatures as low as 900°C with a flow stress ~1 GPa and

a brittle-to-ductile transition at ~ 1850°C. In addition, plastic deformation is controlled by dislocation sliding as in hcp metals, such as magnesium. Deformation as high as 100 % without structural damage has been measured. Observed deformation mechanism is identical to that of hcp metals. This behavior could have great technological applications and also throw some light on the development of a new generation of UHTCs, in which the oxidation resistance is improved as well as structural stability by preservation of this exceptional plastic behavior.

10:40 AM

(ICACC-S12-033-2017) Tribological, Nanomechanical and Oxidation Properties of the Cold-sprayed MAX phase Ti_2AlC Coating

A. Loganathan*[1]; C. Rudolf[1]; C. Zhang[1]; T. Laha[2]; B. Boesl[1]; A. Agarwal[1];
1. Florida International University, USA; 2. Indian Institute of Technology Kharagpur, India

In the present work, Ti_2AlC (MAX phase) was successfully cold-sprayed on the Inconel substrate. No phase change was observed after coating. Typical features of MAX phase such as kinking and buckling were observed on the fractured surface of the Ti_2AlC coating. The wear behavior of the cold sprayed coatings at room temperature and high temperature (600°C) were studied. Nanomechanical properties of the Ti_2AlC cold-sprayed coating were examined. Micro/high load nanoindentation and scratch test were carried out at room temperature and high temperature conditions (300°C). Microstructure and deformation mechanism in the coatings after nanoscratch test at room temperature and high temperature were ascertained. During nanoscratch at room temperature, the scratch path displayed a brittle nature with more fracture and chipping. However, for the nanoscratch at high temperature, the scratch path displayed a ductile nature with ploughing and cutting. Further, the oxidation behavior of the Ti_2AlC coating at high temperature was studied.

11:00 AM

(ICACC-S12-034-2017) Microstructure Evolvement and Mechanical Properties of Tantalum Carbides (Ta_2C) Composites Enhanced by the Addition of SiC

H. Liu*[1]; L. Liu[2]; F. Ye[2]; 1. University of Manchester, United Kingdom;
2. Harbin Institute of Technology, China

Tantalum carbide, in the family of the Ultra-High Temperature Ceramics, is a promising candidate for aerospace application. The current problem of the tantalum carbide is the difficult to achieve the high flexure strength and high fracture toughness at the same time. In this work, high performed Ta_2C composites were fabricated by Spark Plasma Sintering at 1900°C under 30 MPa starting from Ta, TaC and SiC powders. XRD, SEM and EBSD were applied to study the phase transformation and microstructure evolvement. With the SiC concentration increasing, the compositions of the composites evolved from Ta_2C and Ta_5Si_3 to Ta_2C, TaC and Ta_5Si_3. The room temperature mechanical properties were measured as well. Compared with the pure Ta_2C (507±45 MPa, $6.4\pm0.7 MPa \bullet m^{1/2}$) of coarse grains, Ta_2C composites in this work had a highly laminated microstructure and a good combination of high flexure strength (714±21MPa) and fracture toughness ($13.9\pm0.2 MPa \bullet m^{1/2}$). Due to the generation of the laminated structure within the TaC grains, the mechanical properties of TaC dominated composites were greatly enhanced (571±36 MPa, $7.6\pm1.1 MPa \bullet m^{1/2}$), compared with other TaC composites. Additionally, EBSD mapping gave a clue of how this laminated microstructure evolved as SiC concentration increased, which could help us understand the phase transformation of Ta_xC_y in function of Ta:C ratio.

11:20 AM

(ICACC-S12-035-2017) Alkali treated $Ti_3C_2T_x$ MXenes (Alk-$Ti_3C_2T_x$) and their dye adsorption performance

W. Zheng*[1]; Z. Sun[1]; P. Zhang[1]; X. Qin[1]; Y. Wang[1]; Y. Zhang[1]; 1. School of Materials Science and Engineering, Southeast University, China

Due to the "pillar effect" of trapped alkali metal ions (Li^+, Na^+ and K^+), "swelling effect" of penetrated solvent molecules and "repulsion force" of surface functional groups (-OH, -F and/or -O) between the $Ti_3C_2T_x$ nanosheets, the interlayer spacing of $Ti_3C_2T_x$ MXene expands considerably after alkalization treatment with LiOH, NaOH and KOH aqueous solution (remarked as LiOH-$Ti_3C_2T_x$, NaOH-$Ti_3C_2T_x$ and KOH-$Ti_3C_2T_x$), respectively, which results in enhanced removal rate for methylene blue (MB) compared with pristine $Ti_3C_2T_x$. Moreover, the Congo red (CR) anionic dye adsorption performance for pristine $Ti_3C_2T_x$ and Alk-$Ti_3C_2T_x$ was studied and the results show that both pristine $Ti_3C_2T_x$ and Alk-$Ti_3C_2T_x$ exhibit fairly slow removal rate for CR. This preferential adsorption is primarily ascribed to the tuned functional groups, ions exchange and favorable electrostatic interactions between the dye molecules and the MXene surfaces after alkalization treatment. In addition, we found that NaOH-$Ti_3C_2T_x$ possesses the highest adsorption capacity for MB, which can be compared with some commercial active carbon. All these results indicate that Alk-$Ti_3C_2T_x$ are competitive candidates for wastewater treatment.

11:40 AM

(ICACC-S12-036-2017) Effect of synthesis and sintering parameters on densification and mechanical properties of Titanium Diboride (TiB2) ceramics

A. M. Celik*[1]; R. A. Haber[1]; W. Rafaniello[1]; 1. Rutgers University, USA

Titanium dioxide, boron carbide and carbon powders were mixed using two different mixing methods; ball milling and magnetic stirring. The precursors then reacted at different temperatures to form fine-sized and single-phase TiB_2 powders using borothermic reduction reactions. Sintering of synthesized powders carried out under vacuum using Spark Plasma Sintering (SPS) method to fabricate dense samples. Chemical analysis, phase determination, microstructure investigation, and mechanical property measurements were performed to evaluate bulk ceramics. Monolithic TiB_2 ceramics with high density values and good mechanical properties were manufactured using in-house synthesized powders and various parameters during the synthesis and sintering processes were examined.

S13: Advanced Materials for Sustainable Nuclear Fission and Fusion Energy

Processing, Joining, and Coating for Ceramic Fuels and Materials

Room: Coquina Salon H
Session Chairs: Monica Ferraris, Politecnico di Torino;
Christian Deck, General Atomics

8:30 AM

(ICACC-S13-001-2017) Production of Fully Ceramic Microencapsulated Fuels with Low Enriched Uranium for Irradiation Testing (Invited)

K. Terrani*[1]; J. Kiggans[1]; J. McMurray[1]; C. Ang[1]; 1. Oak Ridge National Lab, USA

Fully Ceramic Microencapsulated (FCM) fuel is produced by encapsulation of tristructural isotropic (TRISO) fuel particles a dense SiC matrix. This dense ceramic matrix represents an additional barrier to fission product release beyond the various coating layers in the TRISO fuel particle. The SiC matrix also offers excellent oxidation resistance, even in high pressure steam environments. Therefore, this fuel form is considered a primary example of an accident tolerant fuel (ATF). The ongoing effort at Advanced Fuels Campaign, US DOE Fuel Cycle R&D program is focused on production of LEU (7.3% U-235 enrichment) FCM pellets at ORNL for irradiation testing INL's Advanced Test Reactor (ATR). This is a critical step in development of this fuel concept to establish the ability of this fuel to retain fission products under prototypical irradiation conditions. This study summarizes the fuel fabrication activates for the irradiation campaign starting in 2017.

8:50 AM

(ICACC-S13-002-2017) Design, Evaluation and Manufacturing of Integrated TRISO-Inspired Fuel

S. Harrison*[1]; J. Pegna[1]; J. Schneiter[1]; R. Goduguchinta[1]; K. Williams[1]; E. Vaaler[1]; K. Shirvan[2]; B. Hiscox[2]; 1. Free Form Fibers, USA; 2. Massachusetts Institute of Technology, USA

Free Form Fibers (FFF) has collaborated with MIT to evaluate the Fuel-in-Fiber (FiF) concept using computational modeling. FiF is a patented new technology that utilizes a combination of novel additive manufacturing steps to form a TRISO-inspired fuel in fiber form. Cells containing nuclear fuel with concentric cylindrical structures analogous to TRISO particles are micro-embedded inside a silicon carbide filament. These filaments are designed to serve as multifunctional structural reinforcement to a Ceramic Matrix Composite fuel. FFF initially formulated FIF concept as a potential accident tolerant fuel (ATF) approach, in consideration of development efforts to avoid Fukushima-type events. The MIT Nuclear Science and Engineering department performed a series of neutronic performance modeling studies using the SERPENT Monte-Carlo code package. These analyses evaluated the impact of varying fuel types, pitch spacing of fibers, and matrix material selection using specified fuel-in-fiber designs. The resulting data show that advantageous ATF design choices are possible. Fiber manufacturing development efforts continue progressing at FFF's facilities. Among these advances include additional fiber production rigs coming on-line, continuous SiC fibers are increasing in length, and a multiple laser head production system is being assembled and trialed. An overview of FFF's SiC fiber capabilities will be presented.

9:10 AM

(ICACC-S13-005-2017) Residual Stress Free Joining of Silicon Carbide Ceramics (Invited)

Y. Kim*[1]; S. Jang[1]; 1. University of Seoul, Republic of Korea

Silicon carbide (SiC) ceramics and SiC-based composites play an important role in the field of high temperature structural applications. Wide applications of these materials require further improvement of technologies for the joining of SiC and SiC-based composite materials. There are many possible techniques for joining SiC to themselves such as diffusion bonding, reaction bonding, eutectic phase joining, glass-ceramic joining, adhesive and preceramic polymer joining. However, all the above techniques have resulted build-up of residual stresses around the joined area and/or interfaces because of the difference in thermal expansion coefficients between SiC and an interlayer. The residual stresses are often detrimental to mechanical properties of joined SiC ceramics. The present presentation deals with factors influencing the joining of SiC ceramics realized by eutectic-phase joining. The effects of processing parameters on the joint quality will be illustrated in the present presentation. The residual stress free joined SiC ceramics showed almost the same mechanical properties with the monolithic SiC ceramics. In addition, the joined parts showed almost the same thermal conductivity with the material without joined interlayer.

9:30 AM

(ICACC-S13-006-2017) Vacuum Plasma Spray Metallic Coatings on SiC Composite Claddings

J. S. O'Dell*[1]; L. L. Snead[2]; C. Ang[3]; Y. Katoh[3]; 1. Plasma Processes, LLC, USA; 2. Massachusetts Institute of Technology, USA; 3. Oak Ridge National Lab, USA

The unfortunate events at the Fukushima nuclear power plants highlighted the need for enhancing the accident tolerance of nuclear fuel. Of particular concern is the potential overheating of standard zirconium alloy cladding in a loss of coolant accident. To address this issue, SiC composites have been identified as a high risk, high reward candidate for future nuclear fuel claddings. However, the inherent open porosity present in the majority of fiber-based composites and the unavoidable propagation of a fine network of cracks under modest load are serious concerns for a fully-ceramic composite clad design. Therefore, the development of a hybrid design comprised of a SiC composite cladding with a dense, metallic layer on the interior or exterior of the cladding has been proposed. During this effort, Vacuum Plasma Spray processing methods are being developed to produce a dense, metallic coating on the exterior of SiC composite claddings. To date, both zircaloy and chromium coatings have been produced on SiC composites using VPS processing. Characterization of the coatings includes microstructural examination, X-ray analysis, leak testing, and adhesion strength testing. In addition, a series of zircaloy coated SiC composite claddings along with baseline composite samples are being irradiated in the Massachusetts Institute of Technology Reactor. The characterization and irradiation results will be presented.

10:10 AM

(ICACC-S13-007-2017) Improving the damage-tolerance of Zircaloy cladding by integrated ceramic coatings

J. Zhang*[1]; Y. Zhang[2]; G. Bai[2]; J. Wang[1]; 1. Institute of Metal Research, Chinese Academy of Sciences, China; 2. Life Management Technology Center, Suzhou Nuclear Power Research Institute, China Nuclear Power Technology Research Institute, China

The development of accident-tolerant fuel/clad system during Loss-of-coolant accident (LOCA) has been considered as a 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. Key requirements for successful accident tolerant fuel (ATF) coatings include: i) good oxidation resistance, both in air and water vapor; ii) coefficient of thermal expansion match; iii) chemical compatibility, under both normal and LOCA conditions. It is also desirable to have proper thermal conductivity for maintaining the heat exchange efficiency. MAX phases display a high resistance to oxidation and ion irradiation from which a promising coating materials. Integrated ceramic coatings were synthesized onto Zircaloy substrate by PVD method. 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.

10:30 AM

(ICACC-S13-008-2017) Microstructure and mechanical properties of Cr-based coatings on SiC cladding

C. Ang*[1]; C. Kemery[3]; K. Terrani[2]; J. Kiggans[4]; Y. Katoh[1]; 1. Oak Ridge National Lab, USA; 2. Oak Ridge National Lab, USA; 3. NEO Industries, USA; 4. Oak Ridge National Lab, USA

Fuel cladding based on SiC_f-SiC composites are an attractive and ambitious concept that faces critical challenges. These are the difficulty in gas-retention from operational stress-induced microcracking, and radiolysis-assisted, aggravated hydrothermal corrosion. Barrier coatings based on Cr are investigated as a solution to these two issues. TEM, SEM and XRD examined the microstructure of the coatings, and showed significant microstructural differences in crystallite strains, grain boundary impurities, defect density and crystallite size. Residual stress analysis (by ASTM B975) was conducted on deposited coatings, and determined that detrimental processing stresses from chemical deposition of Cr were much higher than vapor deposition. Tensile adhesion tests (by ASTM D4541) and scratch indentation tests (by ASTM E2456) were adapted for use on small-size coupons to determine the change in mechanical properties. These lay the groundwork for evaluation and down-selection of in-pile coated specimens in the future. Research sponsored by the Advanced Fuels Campaign of the Fuel Cycle R&D program, Office of Nuclear Energy, US Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

10:50 AM

(ICACC-S13-009-2017) Fabrication of SiC coated zircaloy-4 fuel claddings by solid-state reaction method and their adhesion properties

X. Geng*[1]; X. Huang[1]; G. Wen[1]; 1. Harbin Institute of Technology, China

Hydrogen release from the reaction between Zr-based alloy claddings and water steam, raises concerns over safty (may cause explosion) of light water reactors (LWRs) during a loss of coolant accident. This study provides a potential method through solid-state reaction to deposite SiC coatings onto zircaloy-4 substrates in order to enhance the accident tolerance of LWR fuel claddings. Within experiments, SiC/zircaloy-4 diffusion couples were subjected to high temperature heat treatment. Results from various characterisation techniques indicated that reaction occoured, therefore bonding of SiC with zircaloy-4 claddings. Moreover, thermodynamic calculations supported the above experimental phenomena. In addition, both nanoindentation and the tensile tests were performed to evaluate the adhesion properties of the SiC-coated zircaloy-4 samples.

S14: Crystalline Materials for Electrical, Optical and Medical Applications

Optical Material 3

Room: Tomoka C
Session Chairs: Nerine Cherepy, Lawrence Livermore Nat'l Lab; Claudia Wickleder, University of Siegen

8:30 AM

(ICACC-S14-024-2017) Review of Phosphor Identification and Synthesis Methods (Invited)

J. McKittrick*[1]; J. Ha[1]; Z. Wang[1]; G. Hirata[2]; O. Graeve[1]; S. Ong[1]; 1. UC San Diego, USA; 2. Center for Nanoscience and Nanotechnology, Mexico

A higher quality white light can be generated through use of phosphor blends with a near UV-emitting (380-410 nm) LED, instead of a yellow-emitting phosphor and a blue-emitting LED. There are several basic properties that an ideal near UV phosphor should simultaneously possess. The quantum efficiency should be as high as possible and the excitation maximum should fall in the range where near UV LED emission is most efficient. Because of higher photon flux from LEDs, the phosphors are expected to operate at elevated temperatures, which require them to have reduced thermal quenching behavior. The phosphor must not degrade chemically or thermally and the radiative lifetime of the activator ions should be short in order to reduce saturation effects and nonlinear, nonradiative recombination. Sub-micron or nanosize phosphors are needed for reduced light scattering. It must be possible to economically manufacture the phosphor on a large scale. Finally, the phosphor should be non-toxic, and the fabrication, use, and disposal of the phosphor should be as environmentally benign as possible. These topics will be addressed in this presentation. Also discussed is a combined first-principles modeling and experimental approach for the identification of phosphor hosts and activators. Phosphor synthesis methods and resultant particle characteristics will be discussed in the context of luminescence intensity.

9:00 AM

(ICACC-S14-025-2017) Synthesis of nano oxides by water assisted solid state reaction at low temperature (Invited)

K. Toda*[1]; 1. Niigata University, Japan

Progress of the solid state reaction required to fulfill two conditions; "Thermodynamics" and "Kinetics". Many researchers claimed that ionic-diffusion in ionic crystal is very slow at low temperature. However, final product layer spatially separates the raw materials at the interface in the course of solid state reactions. The mass transport of raw materials occurs through the thermodynamically stable product layer. Observed data of ionic-diffusion are not true diffusion rate. Recently, we disclosed a novel water assisted room temperature solid state reaction (WASSR) method to synthesize the nano-ceramic materials in a single phase form at low temperature. This process is very simple and can synthesize the ceramic materials just by mixing of raw materials added a small amount of water. In this study, we present the machanism and practicability of WASSR method.

9:30 AM

(ICACC-S14-026-2017) Luminescent Property of Deep-red $La_2MgGeO_6:Mn^{4+}$ Phosphor Synthesized by Polymerized Complex Method

M. Watanabe*[1]; T. Yoshizawa[1]; S. Kim[2]; K. Seki[1]; K. Uematsu[1]; K. Toda[1]; M. Sato[1]; 1. Niigata University, Japan; 2. Sejong University, Republic of Korea

The phosphor-converted white light-emitting diodes (LEDs) have attracted much attention as the light source for "indoor plant cultivation", which grow plants in a controlled environment permitting stable supply of vegetables without the influence from the outside. Far-red light around 730 nm is especially significant for preparation of higher-value vegetables, because it prevents aggravation of eating quality and excessive growth of vegetables. Currently, the combination of the LED and the blue light excitable red LED emitting round 660 nm has been reported as a candidate for the next artificial light source redeemed red light. However, the intensity of its far-red emission is insufficient. Novel blue light excitable deeper-red phosphors, therefore, are required in order to develop an artificial light source. Mn^{4+} doped phosphors are excited by blue light and exhibit red emission corresponding to the $^4A_2 \rightarrow {}^4T_2$ and $^2E \rightarrow {}^4A_2$ transitions of Mn^{4+}. $La_2MgGeO_6:Mn^{4+}$ is a good candidate for deep-red phosphor. In this study, we synthesized $La_2MgGeO_6:Mn^{4+}$ phosphors by polymerized complex method, exploiting polymerization between ethylene glycol and citric acid and obtained $La_2MgGeO_6:Mn^{4+}$ homogeneously distributed Mn^{4+}, resulting in high efficiency.

10:10 AM

(ICACC-S14-027-2017) Phase Change Sintering of $BaCl_2$ Transparent Ceramics (Invited)

T. Shoulders[1]; R. M. Gaume*[1]; 1. University of Central Florida, USA

Over the past few years, the optical ceramics lab at UCF has developed a procedure for hot pressing barium chloride, a naturally birefringent material exhibiting good performance as a europium-doped gamma ray scintillator. The major features of the fabrication are the containment of powders in an oxygen and moisture-free environment throughout the process and the creation of a complex state of stress by surrounding barium chloride with a carefully matched pressure-transmitting medium. Characterization of sintered samples has revealed that the optimized hot pressing conditions of 450°C and 100 MPa nominal gauge pressure produce a large molar fraction of optically isotropic cubic phase in addition to a small fraction of the equilibrium orthorhombic phase. The phase change and compaction of orthorhombic $BaCl_2$ powders is discussed on the basis of a pressure-induced martensitic transition and subsequent plastic flow densification mechanism. For the phase change component, we introduce a simple model to predict the phase boundary as a function of the state of stress. The model is validated by stress calibration using a well-known pressure induced transition and applied to the $BaCl_2$ material system.

10:40 AM

(ICACC-S14-028-2017) Transparent Polycrystalline Ceramics for Demanding Optical Applications (Invited)

M. R. Pascucci*[1]; 1. CeraNova Corporation, USA

Transparent ceramics are finding increasing use in demanding applications where glasses are limited, and single crystals are not practical. Defense and commercial applications include: infrared transparent materials for sensor windows (flat panels, deeply concave shapes, conformal shapes), transparent armor, scintillators for medical diagnostics, laser host materials, and advanced lighting. Processing of ceramic powders into high quality optical components is critically dependent upon the properties of the starting materials, controlling the balance between grain growth and densification, and eliminating scattering and absorption. CeraNova's processing technology enables densification at lower temperatures and produces a fine-grained, homogeneous microstructure. In addition to high transparency, the fine grain size of CeraNova optical ceramics imparts higher hardness and improved strength compared to larger grained materials. Properties of CeraNova's fine-grained, high strength transparent alumina (CeraLumina™) and spinel ($MgAl_2O_4$) will be discussed.

11:10 AM

(ICACC-S14-029-2017) Development of transparent ceramics for optical and photonic applications (Invited)

Y. Wu*[1]; 1. Alfred University, USA

Polycrystalline transparent ceramics are emerging as a highly promising alternative to single crystal materials and glasses for a wide of optical and photonic applications. For development of high quality of transparent ceramics, it is necessary to investigate the fundamental issues associated with the processing of the materials. The work in this presentation is to understand the mechanisms which control optical transmittance and anti-site defect behavior in optical materials to minimize defects that cause light scattering and absorption. An emphasis is also included on improving the understanding of the effects of processing techniques on birefringence behavior in anisotropic transparent ceramics.

11:40 AM

(ICACC-S14-030-2017) Toughening transparent polycrystalline yttria-stabilized zirconia ceramics with functional yttria coatings

M. Rubat du Merac*[1]; M. Bram[2]; O. Guillon[2]; 1. Technical University Darmstadt, Germany; 2. Forschungszentrum Juelich, Germany

Polycrystalline transparent ceramics have the potential to meet demanding applications that no other materials can satisfy, but their use is limited because their toughness remains far below theoretical. Polycrystalline yttria-stabilized zirconia (YSZ) is one promising transparent ceramic with high hardness, strength, and chemical and thermal resistance combined with excellent optical properties. Transparent polycrystalline YSZ bodies were made by pressureless sintering and hot-isostatic pressing and were coated with yttria by electron-beam physical-vapor deposition (EB-PVD). Upon thermal treatment, yttria's lower coefficient of thermal expansion compared to YSZ imparted a residual surface compressive stress that in some instances more than doubled fracture toughness as measured by indentation. Moreover, transmittance was increased due to antireflection caused by yttria's lower refractive index and the increase could be adjusted over certain wavelengths by varying the coating thickness. Fabrication of transparent YSZ bodies and EB-PVD yttria coatings, microstructure, and optical and mechanical properties of the coated bodies will be discussed.

3rd Pacific Rim Engineering Ceramics Summit

Challenges and Opportunities I

Room: Coquina Salon C

Session Chairs: Shaoming Dong, Shanghai Institute of Ceramics, Chinese Academy of Sciences; Hisayuki Suematsu, Nagaoka University of Technology

1:30 PM

(ICACC-PACRIM-027-2017) Synthesis of cuprate superconductors under high oxygen partial pressure and improvement of the critical current density properties by insertion of water molecules (Invited)

H. Suematsu*[1]; A. Sklyarova[1]; T. Nagumo[1]; H. Shinoda[1]; T. Aoba[2]; T. Suzuki[3]; T. Nakayama[1]; K. Niihara[3]; 1. Nagaoka University of Technology, Japan; 2. Toyohashi University of Technology, Japan; 3. Nagaoka University of Technology, Japan

Among high-T_c superconductive cuprates, $Ba_2Ca_{(n-1)}Cu_nO_y$ and $Sr_2Ca_{(n-1)}Cu_nO_y$ (02(n-1)n) phases[1,2] show unique characteristics. They do not have any toxic heavy metal elements and are stable under high oxygen partial pressures. At ambient pressure, they absorb water and other organic molecules in the crystal near the room temperature to stabilize. These derivative phases also show superconductivity so that the 02(n-1)n phases are now becoming important precursors for development of novel organic-inorganic superconductors which cannot be synthesized by conventional methods. Water molecule insertion in $Sr_2Ca_2Cu_3O_y$ shows improvements in the critical current density and the irreversivility field properties without changing its T_c (105 K). In this presentation, a high oxygen partial pressure synthesis technique using a cubic anvil type press is explained. Then, superconductive properties of $Sr_2Ca_{(n-1)}Cu_nO_y$ ($02^{(Sr)}23$) and the derivative phases are exhibited.

2:00 PM

(ICACC-PACRIM-028-2017) Porous preform structure and interphase effect on RMI ultra high temperature ceramic matrix composites (Invited)

S. Dong*[1]; X. Chen[1]; Y. Kan[1]; X. Zhang[1]; 1. Shanghai Institute of Ceramics, Chinese Academy of Sciences, China

Carbon fiber reinforced ultra high temperature ceramic (UHTC) matrix composites have been considered as good candidate materials for thermal structural elements due to their high mechanical properties, good thermal shock resistance and superior ablation/oxidation resistance, ect. Reactive melt infiltration (RMI), based on the infiltration of molten Si-Zr alloy in porous fiber reinforced preforms, is often employed to fabricate UHTC matrix composites. For RMI UHTC matrix composites, structure of porous fiber reinforced preforms and interphase existing between carbon fiber and UHTC matrix are important factors to determine RMI process and composite properties. In present study, a sol-gel method is used to prepare the porous preform and multilayered $(PyC\text{-}SiC)_2$ interphase is adopted. The influence of sol-gel parameter on the microstructure of the porous body and the relationship between composite properties and porous body structure are investigated. The morphologies and microstructures of the interphase before and after RMI are contrasted by means of SEM and TEM. It is found that the degraded interphase exhibited typical features of Zr aggregation layer and SiC residue layer around the fiber. A possible mechanism of reaction-fusion is proposed based on the characterization results and thermodynamic/heat transfer calculations..

2:30 PM

(ICACC-PACRIM-029-2017) High-temperature strength of ZrB$_2$-based ultra-high temperature ceramics (Invited)

G. Zhang*[1]; 1. Donghua University, China

ZrB$_2$-based ceramics are regarded as the baseline materials in ultra-high temperature ceramics (UHTCs) family. In this work, the high temperature bend strength of ZrB$_2$-based ceramics, mainly the ZrB$_2$-SiC (ZS), ZrB$_2$-SiC-ZrC (ZSZ) and ZrB$_2$-SiC-WC (ZSW) ceramics were investigated up to 1600°C in high purity argon atmosphere and the mechanisms of the improved high-temperature bending strength in the ZSW sample were discussed. The specimens were dense ceramics fabricated by hot pressing using self-synthesized high purity ZrB$_2$ and commercial SiC powders as raw materials. The different distribution features of the residual oxygen impurity in ceramics were analyzed by EDS. The distribution of the W in ZSW specimen was analyzed by TEM. The residual thermal stresses in different samples were evaluated by both Raman spectroscopy (RS) and X-ray diffraction (XRD). The temperatures at which the stresses began to accumulate were calculated. At the same time high-temperature resistance to deformation of ZrB$_2$-SiC-based ceramics was evaluated via four-point flexure test under different conditions. The relationships between the high-temperature bending strength and the above behaviors for ZrB$_2$-SiC-based ceramics were discussed, which provides a train of thought and method for designing of materials.

3:20 PM

(ICACC-PACRIM-030-2017) Effects of filler materials on the properties of ceramic composites made by precursor impregnation and pyrolysis process (Invited)

S. Lee*[1]; J. Kim[1]; A. Mohan[1]; 1. Korea Institute of Materials Science, Republic of Korea

SiC or carbon fiber fabrics were used for the fabrication of the SiC$_f$/SiC or C$_f$/Si(B)CN ceramic matrix composites (CMC) by precursor impregnation and pyrolysis (PIP) method. The SiC$_f$/SiC CMCs made with different polycarbosilane (PCS) precursors showed elegant fracture behavior with relatively high strength (200 - 310 MPa). However, the CMCs suffered from strong deterioration at 1500°C. For gaining a deeper understanding of the strength degradation of the CMC at high temperature, the high temperature thermal behavior as well as the corresponding phase characterization of the PCS and polycarbosilazane (PCSZ) precursor derived ceramics was analyzed. The oxygen which was contained in the precursors mainly caused the deterioration of properties at 1500°C. The dispersion and infiltration of oxide or non-oxide fillers in the 2D-woven fiber fabrics were performed in order to get over the high temperature deterioration of the matrix phase. Among the four filler materials tested, SiC was most appropriate in terms of mechanical properties and thermal stability. However, SiO$_2$ layer which was formed on the surface of SiC filler deteriorated the high temperature properties of the CMC. The problem could be solved by removing the oxide layer before PIP process. By the optimization of the filler, the high temperature strength of the CMC could be improved at and above 1500°C.

3:50 PM

(ICACC-PACRIM-031-2017) Selection of the liquid-phase sintering additive for SiC and the fabrication of SiC$_f$/SiC composites by EPD combined with hot pressing (Invited)

D. Yoon*[1]; P. Fitriani[1]; K. Raju[1]; A. Sharma[1]; J. Park[2]; 1. Yeungnam University, Republic of Korea; 2. KAERI, Republic of Korea

We are going to present on the selection of liquid-phase sintering additives for SiC as well as the fabrication technique SiC$_f$/SiC composites. For the selection of sintering additives, various kinds of metals, metal oxides and rare-earth nitrates were examined as sintering additives for β-SiC by considering the standard Gibbs formation free energy under hot pressing conditions (1973 – 2123 K). The conclusions drawn from thermodynamic considerations were compared with the experimental observations, where Al, Mg and their oxide forms were found to be only effective sintering additives among main group elements, while all of the rare-earth elements enhanced the density of SiC. In order to fabricate the SiC$_f$/SiC, both AC- and DC-electrophoretic deposition (EPD) combined with ultrasonication were applied for the matrix infiltration into the 2D-woven Tyranno SA-3 fabric preform. For this, the zeta potential for SiC slurries was optimized for EPD followed by hot pressing at 1750°C for 2 hr under 20 MPa. Associated with the fabrication of dense SiC$_f$/SiC composites having a planar and tubular shape, experimental results on the slurry properties, designing a new mold for tube fabrication, the degree of infiltration along with the resultant composite properties will be presented.

4:20 PM

(ICACC-PACRIM-032-2017) Grain-oriented polycrystalline ceramics prepared by colloidal processing using magnetic field (Invited)

S. Tanaka*[1]; 1. Nagaoka University of Technology, Japan

Crystal orientation process using a magnetic field has been expected to improve various functional properties of polycrystalline ceramics. A grain oriented polycrystalline alumina ceramic with nearly single-crystal transparency was fabricated using this method. A well-dispersed slurry consisting of fine particles was prepared using an effective dispersant, which is the Na salt of 4,5-dihydroxy-1,3-benzenedisulfonic acid. The c-axis of alumina grains was oriented along the direction parallel to the magnetic field. Using HIP, the transmittance of the specimen with a thickness of 0.8 mm was over 75% at a wavelength of 650 nm. We also report a colloidal processing using ultra-violet, UV, curable resin and the strong-magnetic-field. An issue of the method using a magnetic field is to take a long time for solidification in magnetic field. UV curable resin is expected to be consolidated in several seconds. Concentrated colloids of (Sr,Ca)$_2$NaNb$_5$O$_{15}$ slurry were prepared from the UV curable acrylic resin, photoinitiator, and dispersant. The colloid was casted on a PET film and set into superconducting magnet. After duration in magnetic field, UV light was irradiate to the tape for polymerization in the magnetic field. XRD showed that the particles were oriented well in the colloid with UV curable resin. Orientation degree depended on duration time and magnetic flux density.

FS1: Geopolymers, Chemically Bonded Ceramics, Eco-friendly and Sustainable Materials

Synthesis, Processing Microstructure

Room: Coquina Salon E

Session Chair: Waltraud Kriven, University of Illinois at Urbana-Champaign

1:30 PM

(ICACC-FS1-001-2017) Geopolymers based on natural and synthetic metakaolin: A critical review of published scientific papers (Invited)

J. Davidovits*[1]; 1. Geopolymer Institute, France

Much of the original research into geopolymers was conducted on calcined kaolinitic clay precursors known as metakaolins. The chemical formula for kaolinite is Si$_2$O$_5$Al$_2$(OH)$_4$. From a geopolymer standpoint we may write ΞSi-O-Al-(OH)$_2$ with the covalent aluminumhydroxyl - Al-(OH)$_2$ side groups of the poly(siloxo) hexagonal macromolecule [Si$_2$O$_5$]$_n$. Metakaolin results from the dehydroxylation of the -OH groups in kaolinite. The reactive molecule is an alumino-silicate oxide Si$_2$O$_5$Al$_2$O$_2$, coined MK-750 in order to pinpoint the calcination temperature. It comprises two Al-oxyde types: ΞSi-O-Al=O (alumoxyle), Al in 5-fold coordination, and

ΞSi-O-Al(O_2), Al in 4-fold coordination. The kiln technology determines the desired chemical reactivity. The geopolymer chemistry was invented 40 years ago with natural metakaolins. The review discusses the correlation between reactivity, calcination methods, MAS-NMR spectroscopy, reaction mechanism and applications, for natural and synthetic metakaolins.

2:30 PM

(ICACC-FS1-002-2017) What is the Smallest Size of Geopolymer Particles?: Production of Geopolymer Nanoaggregates, Their Characteristics and Emerging Applications (Invited)

D. Seo*[1]; 1. Arizona State University, USA

Geopolymer materials are usually obtained as monoliths as they are traditionally used in building applications. Importantly, geopolymer is an innately nanostructured material in that it has a dense gel network structure consisting of primary aluminosilicate nanoparticles (10 - 50 nm) that are strongly fused together throughout the body of the geopolymer. By adjusting the geopolymer resin compositions with high alkali concentrations, we demonstrate that geopolymerization process can be controlled to produce submicron-sized highly structured nanoaggregates of geopolymer. The relationship between their synthetic conditions and resulting pore and aggregate structures can be established. 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 potential applications of the new materials in polymer-nanocomposites and antimicrobials. Interesting extension of the synthetic method will be given in terms of supported metal oxide synthesis using the geopolymer nanoaggregates as an ideal high-surface area support.

3:20 PM

(ICACC-FS1-003-2017) The influence of amorphous/crystalline ratio in the aggregates on the polycondensation of high strength geopolymer composites

E. Kamseu*[1]; A. Nana[2]; R. C. Kaze[3]; C. Leonelli[4]; 1. Local Materials Promotion Authority/MIPROAMLO, Cameroon; 2. University of Dschang, Cameroon; 3. University of Yaounde I, Cameroon; 4. Universita Studi Di Modena, Italy

The mix design of geopolymers allow the possibility to reproduce natural stones using essentially various natural solid solutions. In general, the geochemical history of the natural solid solutions affect the dissolution and polycondensation. In this work selected sodique, potasique and sodo/potasique solid solutions (feldspaths and granite) are investigated for the design of high strength geopolymer composites as for the replacement of dimensioned stones. The amorphous/crystalline ratio of the solid precursors were used to understand the role of dissolved and undissolved fraction into the strength development and durability of high strength geopolymer composites. It was concluded that a high strength and durable matrix is a product of chemico-mechanical equilibrium of phases contained into the composites including the pore volume and pore-size distribution which is significant for the life cycle of geopolymers composites.

3:40 PM

(ICACC-FS1-004-2017) Thermal behavior of geopolymer cements using sodium waterglass from rice husk ash and waste glass as alternative activators

H. Tchakouté Kouamo*[1]; C. Henning Rüscher[2]; 1. University of Yaoundé I, Cameroon; 2. Leibniz Universität Hannover, Germany

The aim of this work was to study the thermal behavior of geopolymer cements using sodium waterglass (NWG) from rice husk ash and waste glass as alternative alkaline activators. The geopolymer cements obtained using sodium waterglass from rice husk ash (G1) and the one obtained with sodium waterglass from the waste glass (G2) were treated at room temperature for 28 days and then heated at 200, 400, 600 and 800°C. The results show that the geopolymer cements lose their compressive strength from room temperature to 400°C. At 600°C, the compressive strength of geopolymer cements increases relative up to 200 and 400°C. At 800°C, the reduction of compressive strength of geopolymers is assigned to the total evaporation of the rest of structural water. However, it is higher than that measured at ambient temperature for G2 but lower for G1. The total mass loss of G1 and G2 are about 12.57 and 15.04%, respectively. This suggests that G2 are more condensed geopolymer structure indicating that it could a very suitable material for fire resistant application. The results indicate that NWG from rice husk ash and waste glass could be served as suitable alternative activators for producing metakaolin-based geopolymer cements with high-temperature performance.

4:00 PM

(ICACC-FS1-005-2017) Effects of nucleation seeds on Alkali-Activated Binders (Invited)

D. Sarbapalli[1]; S. Puligilla[1]; P. Mondal*[1]; 1. University of Illinois at Urbana-Champaign, USA

Various nano-sized additives such as alumina, silica, zinc oxide, and titanium dioxide were added in small dosages (1% by weight of precursor) as external nucleation seeds to gain new insights into the reaction mechanism of alkali-activated fly ash-slag and metakaolin binders. The effects on the reaction mechanism was studied by monitoring heat evolution though isothermal calorimetry and rate of hardening through ultrasonic wave reflection (UWR). Selective chemical dissolution (salicylic acid-methanol and hydrochloric acid attack) along with Fourier transform infrared spectroscopy with spectral subtraction was used to quantify the amount of products formed and the amount of unreacted fly ash at different ages. Calorimetry and UWR results for hydroxide activated fly ash-slag binders with 1% addition of nano silica and nano alumina show significant increase in the rate and extent of reaction although the addition of nano zinc oxide strongly retards the reaction. Interestingly, the nano alumina and nano silica seeds do not change the rate and extent of reaction of hydroxide activated metakaolin binders indicating differences in the reaction mechanism between binders made with fly ash and metakaolin.

4:30 PM

(ICACC-FS1-006-2017) Geopolymer foams: Influence of the processing route on chemical composition and properties (Invited)

M. Arnoult*[1]; S. Rossignol[1]; G. Gasgnier[2]; 1. Laboratoire SPCTS, France; 2. Imerys Ceramics, France

Mineral foams are expected to be used in many technological applications. Thanks to their low heat conductivity, good heat resistance, and good acoustic properties, this type of material is suitable for insulation applications. Porous ceramics can be obtained with several ways such as replica, sacrificial template and direct foaming methods. The aim of this work is to study processing routes that can be used to produce geopolymer foams. In this work, foams were obtained using a sacrificial template and direct foaming methods. An emulsion was prepared with an alcaline silica solution and different types of oil. Finally, the metakaolin was added to the reactive mixture and cured at room temperature. On the other hand, various foaming agent have been used to create porosity. A comparison of the different processes used to produce foams is investigated in this work. First, all the reactive mixtures were studied by FTIR monitoring in order to evaluate the material formation. Then, thermal conductivity was measured. The results obtained indicate that the processing route used has an influence on the chemical composition as well as on foam properties such as porosity, thermal conductivity and density.

5:00 PM

(ICACC-FS1-007-2017) The Corrosion of Kaolinite by iron minerals and its effects on geopolymerization (Invited)

R. C. Kaze[2]; L. M. Beleuk a Mougam[3]; A. Nana[3]; E. Kamseu*[1]; C. Leonelli[4];
1. Local Materials Promotion Authority/MIPROAMLO, Cameroon;
2. University of Yaounde I, Cameroon; 3. University of Dschang, Cameroon;
4. Universita Studi Di Modena, Italy

Iron-rich aluminosilicate (laterites) with disordered structure due to the corrosion of kaolinite by iron minerals were investigated as solid precursors for geopolymerization. The particle size distribution, the BET surface area, the chemical and mineral composition of two raw laterites were found to vary with temperature. The raw materials in the range of temperature 25 < T < 500°C shown behavior similar to that of metakaolin. At temperature higher than 500°C, the coarsening of particles, the decrease of BET surface area correspond to an initial sintering explaining the poor polycondensation/geopolymerization and decrease of strength. The increase of temperature of calcinations of laterites between 25 and 500°C reduced the setting time of geopolymer products without changing significant the flexural strength, that remained between 4 and 6 MPa. The addition of amorphous silica from RHA increase exponentially the flexural strength that goes from 4 to 20-30 MPa according to the concentration of RHA. It was concluded that the addition of silicate allow the development of a significant reactive and equilibrated matrix in the ternary $Fe_2O_3(FeO)$-Al_2O_3-SiO_2 system.

FS2: Advanced Ceramic Materials and Processing for Photonics and Energy

Photonics, Electronics and Sensing II

Room: Halifax A/B

Session Chairs: Fiorenzo Vetrone, Institut National de la Recherche Scientifique; Rafik Naccache, Concordia University

1:30 PM

(ICACC-FS2- 026- 2017) Hybrid electrospun heterostructures for low-cost light-emitting device architectures (Invited)

S. G. Cloutier*[1]; I. Ka[1]; R. Nechache[1]; J. Benavides[1]; L. Gerlein-Reyes[1];
X. Guo[1]; 1. École de technologie supérieure, Canada

In this report, we discuss the creation of hybrid heterostructures using electrospinning (for fibers) and cojetting (for particulates). As we show, solution-based precursors can be controllably & uniformly deposited using low-cost electrospraying methods prior to conversion. Resulting devices can offer superior performances compared to organic semiconductors, conducting polymers or colloidal self-assembled systems. As we demonstrate, the same approach can be used (1) to deposit a metal-oxide collector, (2) deposit active light-emitting material sutructures (fibers and particles) and (3) to deposit high-performance transparent electrodes. As such, a complete device architecture can be deposited using the same setup configuration with well-controlled deposition parameters. This approach can be used to produce hybrid heterostructures with otherwise incompatible material systems to explore new approaches to producing better low-cost light-emitting device architectures.

2:00 PM

(ICACC-FS2- 027- 2017) Quantum dot based luminescent solar concentrator (Invited)

H. Zhao*[1]; 1. INRS, Canada

Luminescent solar concentrators (LSCs) are of current interest as a platform for solar energy harvesting, complementary to traditional photovoltaics, as they can dramatically improve energy harvesting efficiency and simultaneously reduce the expensive photovoltaic module cost. Colloidal quantum dots (QDs) are attractive for use in LSCs, due to their high quantum yield, broad absorption spectrum, size-tunable emission and enhanced chemical and photo stability with respect to organic chromophores. However, their small Stokes shift results in reabsorption losses that hinder the realization of large-area devices. Here, we demonstrate for the first time the application of "giant" $CdSe/Cd_xPb_{1-x}S$ QDs as high performance LSCs. These QDs were embedded in a polymer matrix, resulting in highly transparent composites with the absorption spectrum in the range 300 to 600 nm. The efficiency obtained by applying this giant QDs in a large-size LSC (up to 7 cm in length) is ~1.15%. In addition, we also synthesized near infrared (NIR) PbS/CdS core/shell QDs for LSC. The as-synthesized QDs allow for good matching with the solar spectrum, from UV to NIR region. The LSC exhibits the highest value (6.1% with a geometric factor of 10) for optical efficiency compared to the reported NIR QD-based LSCs. These results highlight the efficient approach to grow the alloyed giant QDs or PbS/CdS QDs for high efficiency LSCs in photovoltaics.

2:30 PM

(ICACC-FS2- 028- 2017) Carbon Dots: From Synthesis to Application (Invited)

F. Victoria[2]; J. Jesu Raj[1]; F. Vetrone[1]; R. Naccache*[2]; 1. Institut National de la Recherche Scientifique, Canada; 2. Concordia University, Canada

In recent years, nanomaterials (defined as materials < 100 nm in a single dimension) have garnered a significant interest for the development of novel applications in the physical and life sciences. This is especially true for luminescent nanoparticles, which have been investigated for the development of sensors, imaging/diagnostic probes, display and solar cell applications. Recently, a relatively new class of luminescent nanomaterials, namely carbon dots (CDs) has come to light. CDs, sometimes known as carbongenic dots, are carbon, oxygen, nitrogen and hydrogen containing materials with the first two elements typically accounting for ~90% of their elemental composition and contributing to their low chemical toxicity profile. Moreover, they are typically water dispersible and can be prepared from an abundant number of inexpensive sources. While they are small in size (typically 1-5 nm), they can offer a high quantum yield of emission, a process that is controlled through passivation of the surface with an organic reagent. Combined with their small size and versatile optical properties, developing CDs as multifunctional probes can be achieved. Here, we focus on the thermal decomposition and microwave synthesis of CDs and their surface passivation in order to achieve high emission quantum yields. We investigate the role of the excitation wavelength, pH and temperature on the resultant optical properties.

3:20 PM

(ICACC-FS2- 029- 2017) Optical Properties of Biosilicas in a Rice Plant (Invited)

H. Imai*[1]; K. Sato[1]; A. Yamauchi[1]; T. Ishigure[1]; Y. Oaki[1]; N. Ozaki[2]; 1. Keio University, Japan; 2. Faculty of Bioresource Sciences, Akita Prefectural University, Japan

Rice plants accumulate silicate ions in their body and produce various kinds of amorphous silicas called "plant opal". The biogenic silicas would play many important roles, such as enhancing mechanical strength, preventing diseases and feeding damages, and encouraging photosynthesis. However, the properties of plant opals have not been sufficiently clarified on the basis of experimental evidence. In the present work, we investigated the optical properties of two kinds of plant opals: silicified long cells called "silica plates" that cover the surfaces of leaf blades and fan-shaped silica embedded inside leaf blades. We analyzed the optical properties of silica plates and fan-shaped silica by two-dimensional optical simulation and optical experiments using real plant opals. The simulation and experimental results showed that micrometric projections on silica plates diffuse visible light effectively on leaf blades. Thus, the plant opal would perform as a solar diffuser panel for chloroplasts and promote photosynthesis.

3:50 PM

(ICACC-FS2- 030- 2017) Multi-Functional Nanoplatforms Based on Rare Earth Doped Nanoparticles (Invited)

F. Vetrone*[1]; 1. Institut National de la Recherche Scientifique, Canada

In recent years, rare earth doped nanoparticles have been proposed for a number of exciting applications in a wide-range of fields including nanomedicine, biosensing, bioimaging, nanothermometry, and recently, energy and environment. This is due, primarily, to their inherent ability to convert low-energy near-infrared (NIR) excitation light to higher energies spanning the UV, visible, and NIR regions of the spectrum via a process known as upconversion. Inherent to the rare earths, it results from the multitude of 4f electronic energy states, many of which are spaced equally and long-lived, that facilitate the absorption of multiple low energy photons to populate the higher energy emitting states. Thus, upconversion is a multiphoton process, but unlike other two-photon (or multi-photon) excited materials, the need for expensive ultrafast lasers is eliminated since the simultaneous absorption of multiple photons is not required. Here, we will examine and give an overview of the process of upconversion in rare earth doped upconverting nanoparticles (UCNPs) and present a perspective on both their applicability in various applications. Finally, we will demonstrate that the intelligent combination of diverse materials, with different properties, will allow for the engineering of novel multifunctional nanostructures, which can usher in a new era for UCNPs in the fields of renewable energy and nanomedicine.

4:20 PM

(ICACC-FS2- 031- 2017) Microwave assisted synthesis of $Y_2O_3:M^{3+}$ downconversion and upconversion nanophosphors (Invited)

M. S. Toprak*[2]; A. M. Khachatourian[2]; C. Vogt[1]; E. M. Vasileva[2]; I. Stjugov[2]; M. Mensi[2]; S. M. Popov[2]; 1. KTH Royal Institute of Technology, Sweden; 2. KTH Royal Institute of Technology, Sweden

Properties of rare-earth based nanophosphors depend strongly on various factors such as dopant composition, dopant concentration, crystallinity, homogeneity, particle size and crystallite size. Effective control of these parameters can enhance, or significantly influence the performance of the phosphor material. Nano-sized particles of $Y_2O_3:Tb^{3+}$, $Y_2O_3:Tb^{3+},Yb^{3+}$, and $Y_2O_3:Tm^{3+},Yb^{3+}$ are prepared with a microwave assisted solution-based method followed by calcination. The structural and luminescent properties of nanophosphors are investigated by XRPD, TEM, PL analysis and time-resolved spectroscopy. Effective control of the reaction parameters and doping concentration resulted in uniform nanostructures with downconversion (DC) and upconversion (UC) characteristics. The calcined particles are polycrystalline cubic Y_2O_3 phase. Upon ultraviolet (UV) excitation $Y_2O_3:Tb^{3+}$ particles exhibit DC emissions due to $^5D_4 \rightarrow {}^7F_J$ transitions of Tb^{3+}. Upon 980 nm excitation, Tb^{3+} and Yb^{3+} co-doped Y_2O_3 particles exhibit intense green UC emissions from Tb^{3+} ions. For the Tm^{3+} and Yb^{3+} co-doped Y_2O_3 particles intense blue, weak red, and intense near infrared UC emissions of Tm^{3+} are observed. The UC mechanisms involved in these emissions and energy transfer between Tm^{3+}, Tb^{3+} and Yb^{3+} is studied by considering their spectroscopic properties.

S1: Mechanical Behavior and Performance of Ceramics & Composites

Processing - Microstructure - Mechanical Properties Correlation I

Room: Coquina Salon D

Session Chairs: Jordi Seuba, INSA Lyon (Mateis); Hans-Joachim Kleebe, Technical University Darmstadt

1:30 PM

(ICACC-S1-042-2017) Processing and mechanical properties of porous ceramic-polymer composites with unidirectional porosity (Invited)

J. Seuba*[1]; S. Meille[1]; S. Deville[2]; 1. INSA Lyon (Mateis), France; 2. CNRS (UMR3080), France

Macroporous ceramics are extensively used in filtration, biomedical applications, thermal insulation, and electrochemical and catalytic processes. However, their overall performance is usually maximized increasing the total pore volume, at expenses of their mechanical stability. One strategy to mitigate this issue is to optimize the pore architecture in terms of pore directionality, size, and shape. In the last decade, ice-templating (or freeze casting) has emerged as a powerful technique to process unidirectional macroporous materials in a relatively simple, versatile, and low cost way. The high anisotropy exhibited by these materials reinforces the orientation of the main solicitation, but also severely weakens the direction perpendicular to it. This feature presents a major drawback for their industrial spread, particularly in applications with a multiaxial stress solicitation. In this presentation, I will detail several strategies to enhance the mechanical resistance in both directions, parallel and perpendicular to the porosity. In particular, I will focus on our efforts to coat the ceramic scaffold with a polymer and its effects on strength and work of fracture.

2:00 PM

(ICACC-S1-043-2017) Quantifying the Effect of Templated Growth on the Crystallinity and Density of Polycarbosilane-Derived Silicon Carbide

T. Key*[2]; I. Wolford[1]; D. Petry[2]; M. O'Malley[1]; M. Cinibulk[1]; 1. Materials and Manufacturing Directorate, USA; 2. UES Inc., USA

Temperatures in excess of 1400°C are typically required to fully convert and crystallize polycarbosilane (PCS) pre-ceramic polymers to SiC. Limited by the current commercially available fibers, pyrolysis temperatures and times of PIP-based SiC/SiC ceramic matrix composites (CMCs) often result in a significant amount of non-crystalline material remaining in the matrix. In support of PIP-based matrix processing and modeling, the impact of various SiC seed powders on the density and crystallinity of STARFIRE SMP-10 (a commercial PCS) was quantified after processing at temperatures between 1100°C and 1600°C. Seed powders were seen to dramatically improve the crystallinity of pre-ceramic polymer derived SiC. The thickness of the amorphous layer on the powder's surface, rather than surface area itself, was found to have the greatest influence on inducing templated growth.

2:20 PM

(ICACC-S1-044-2017) The effect of diamond grits on the microstructures and properties of polycrystalline diamonds (PCDs) sintered by HPHT

L. Deng*[1]; 1. Element Six, United Kingdom

Polycrystalline diamonds (PCDs) are diamond powders which are sintered together under high pressure and high temperature conditions. This study investigates the sintering process of several diamond grits with different sizes under the assistance of catalytic metal cobalt. The effects of various treatments, the sintering pressure and temperature on the PCDs' microstructure and mechanical properties are discussed. The relationship between the diamond grit sizes and the mechanical properties of PCDs are studied. The microstructures of the PCDs are characterised by SEM and the mechanical properties are investigated by wear and fracture tests. Finally, according to these findings, the various applications of PCDs in the abrasion area are presented.

2:40 PM

(ICACC-S1-045-2017) Correlation Between Microstructure and Mechanical Properties of Boron Suboxide B_6O Ceramics

H. Kleebe*[1]; 1. Technical University Darmstadt, Germany

B_6O is a potential candidate for superhard materials with a hardness of 45 GPa measured on single crystals. In recent studies it was found that mixtures of Al_2O_3/Y_2O_3 can be utilized as an effective sintering aid. Therefore, liquid phase sintering of B_6O can be achieved with such oxide additives. Microstructure evolution as a function of additive composition during SPS/FAST processing and resulting mechanical properties were investigated in detail. A strong dependence of the resulting microstructure and the corresponding mechanical response was found. The material revealed characteristic triple junctions filled with amorphous residue composed of B_2O_3, Al_2O_3 and Y_2O_3, while no clear evidence of grain-boundary films was observed along internal interfaces. Mechanical testing revealed on average a hardness of 33 GPa, a fracture toughness of approximately 4.5 $MPa(m)^{1/2}$ and a strength value of 520 MPa. Scanning electron microscopy (SEM) and conventional as well as high-resolution transmission electron microscopy (TEM) analysis allowed for the correlation of the intrinsic microstructure with the measured mechanical properties, in particular, with the fact that, independent of additive composition and volume fraction, no further increase in fracture toughness could be achieved.

3:20 PM

(ICACC-S1-046-2017) Microstructure and mechanical properties of B_4C--TiB_2--SiC composites fabricated by reactive hot pressing from the B_4C–TiC–Si as raw materials

W. Wang*[1]; 1. Wuhan University of Technology, China

High-performance B_4C--TiB_2--SiC composites were fabricated with a new reaction system of B_4C–TiC–Si by reactive hot pressing. The chemical reaction processing, microstructure evolution and mechanical properties of composites were studied. The results show that The chemical reaction processing was controlled by the diffusion of boron atoms and hindered by the accumulated product layer of TiB_2 and C. The melting Si can offer another diffusion path for boron atoms, thereby promoting the reaction process. The Vickers hardness, flexural strength, and fracture toughness of the fabricated B_4C--TiB_2--SiC composites respectively reached 34--36 GPa, 650-700MPa, and 6-7MPa $m^{1/2}$. Both the intergranular fracture resulting from mismatch in thermal expansion coefficient between TiB_2 and SiC and the transgranular fracture resulting from the network structure of TiB_2 can enhance the fracture toughness of the TiB_2–SiC composite.

3:40 PM

(ICACC-S1-047-2017) Effects of the addition of white graphene to hydroxyapatite: Processing, Testing, and Characterization

T. Aguirre*[1]; T. B. Holland[1]; 1. Colorado State University, USA

The effects of adding boron-nitride nanoplatelets (BNNP) to spark plasma sintered (SPS) hydroxyapatite (HA) were investigated. Because of high stiffness it is hypothesized that adding BNNP to HA will increase fracture toughness (K_{IC}) without increasing cytotoxicity. HA, a naturally occurring mineral in bones and teeth, displays poor K_{IC} (≤ 1.0 MPa) and nanophase reinforcement has been attempted through the use of nanotubular and nanoplate structures with promising, though scattered, results. Carbon nanotubes and nanoplatelets have been shown to improve the K_{IC} of HA, but have contradictory biocompatibility reports as opposed to the promising aspects of BNNP. However, there has been little to no investigation of HA-BNNP composites. Because biocompatibility and excellent mechanical properties BNNP are ideal candidates for toughening HA. Through rapid heating rates available through SPS these samples are able to be quickly fabricated with nanometric grain sizes, which has been shown to increase mechanical properties. Sintered samples will be tested to 4 point bending to characterize their flexural strength as well as K_{IC}. Vicker's hardness testing was used to assess the hardness as well as the indentation fracture toughness. Currently, sinterability, fracture toughness, and microstructural development in these composites are demonstrating very promising results in our testing.

4:00 PM

(ICACC-S1-048-2017) Densification and Mechanical Properties of Spark Plasma Sintered Si_3N_4/ZrO_2 Nanopowder

A. Sayyadishahraki*[1]; K. Nekouee[2]; E. Taheri-Nassaj[1]; A. Hassanzadeh[3]; 1. Tarbiat Modares University, Islamic Republic of Iran; 2. Sahand University of Technology, Islamic Republic of Iran; 3. Islamic Azad University, Islamic Republic of Iran

Fully dense Si_3N_4-ZrO_2 composites, where ZrO_2 content varied from 0 to 30 vol.%, were produced by Spark Plasma Sintering (SPS) at 1600°C under pressure of 30 MPa for 10 min. The Si_3N_4, Y_2O_3 stabilized ZrO_2 and 2 wt.% Al_2O_3 (sintering aid) were used as starting powders and mixed with high energy ball milling. The milled powder had an average particle size of 75 nm. The XRD patterns of SPSed composites showed that the entire ZrO_2 constituent was in the form of tetragonal phase and no tetragonal to monoclinic phase transition was observed. Mechanical properties evaluation revealed that the hardness of the composites decreased from 16.6 to 13.2 GPa, while fracture toughness increased from 5.8 to 7.1 MPa $m^{1/2}$ with the increase of ZrO_2 content from 0 to 30 vol.%, respectively. The improvement in the fracture toughness was attributed to the presence of the stress-induced phase transformation from tetragonal to monoclinic ZrO_2 as the dominant toughening mechanism in the SPSed Si_3N_4-ZrO_2 composites. Tribological behavior of SPSed Si_3N_4-30 vol% ZrO_2 composite against steel DIN-Ck45K was studied using a pin-on-disk tribometer under dry conditions. The results showed that the composite possessed good wear resistance including friction coefficient and specific wear rate of 0.67 and 3.41×10^{-6} mm^3/Nm respectively, which makes it as a promising candidate for tribological applications.

4:20 PM

(ICACC-S1-049-2017) Inconel 625-Tungsten Carbide Coatings Obtained by Laser Cladding

D. Kata*[1]; J. Huebner[1]; J. Lis[1]; 1. AGH University of Science and Technology, Poland

Laser cladding was used to prepare Inconel 625-WC composite coatings useful for high temperature turbine blades. Three powder mixtures with different Inconel 625 - WC weight ratio (10, 20 and 30 weight % of WC) were prepared. Coatings were made using following process parameters: laser beam diameter ø ≈ 500 μm, powder feeder rotation speed - 7 m/min, scanning velocity - 10 m/min, laser power - 220 W changed to 320 W, distance between tracks - 1 mm changed to 0.8 mm. Microstructure of coatings was examined by Leica DM2500M optical microscope and NOVA NANO SEM 200. Hardness of samples were investigated by Future-Tech FM-700 Hardness tester with Vickers indenter. Samples were tested under load of 200 g for 15 seconds. Additionally, nanohardness test was performed on samples containing 30 wt % of WC in order to investigate hardness of different phases that appeared in the samples, using CSM instruments diamond Berkovich nanoindenter under load of 30 mN. Coatings produced by laser cladding were crack and pore free, chemically and structurally homogenous. High cooling rate during cladding process resulted in fine microstructure of material. Hardness improved with addition of WC from 396.3 ± 10.5 HV for pure Inconel 625, to 469.9 ± 24.9 HV for 30 weight % of WC. Tungsten carbide dissolved in Inconel 625 which allowed formation of intergranular eutectic that contains topologically close packed (TCP) phases

4:40 PM

(ICACC-S1-050-2017) Chromium alloyed $MoSi_2$-composite for improved low temperature oxidation resistance

E. O. Strom*[1]; 1. Sandvik Heating Technology, Sweden

The terminal portion of a $MoSi_2$ heating element, Kanthal Super 1800, has been modified in order to improve its resistance to accelerated low temperature oxidation, so called pesting. A hybride terminal consisting of a transition zone containing substantially $(Mo,Cr)Si_2$ and a silica based oxide phase is joined to a regular $MoSi_2$-based terminal (KS 1800). Thermogravimetric analysis (TGA) has been carried out on both materials at 350-550°C showing a remarkably lower weight gain on the Cr-alloyed sample. Samples exposed isothermally in a laboratory furnace at 450°C for 3576 hours gave at hand a much thinner oxide scale on the $(Mo,Cr)Si_2$-based material. XRD and SEM investigations have been carried out to examine reaction products that form on the oxidized surfaces. Due to evaporation of chromium-containing oxide species and reduced creep resistance at elevated temperatures the $(Mo,Cr)Si_2$-based transition zone of modified heating elements should preferably not be exposed to temperatures exceeding 1200°C. However, by adding chromium to the silicide its oxidation rate is significantly suppressed in the pesting temperature regime (400-550°C), which would extend life time of heating elements suffering from detrimental low temperature oxidation.

5:00 PM

(ICACC-S1-051-2017) Modeling the Mass Yield and Molecular Weight Distribution of Polycarbosilane

T. Key*[2]; G. Wilks[1]; T. Parthasarathy[2]; D. King[2]; M. Cinibulk[1]; 1. Air Force Research Laboratory, USA; 2. UES Inc, USA

Low molecular weight (MW) polycarbosilane is often present in preceramic polymers and enhances viscosity for the purpose of composite infiltration. Due to the increased volatility of low MW chains, a model was developed to semi-empirically determine the MW distribution and then predict the yield and evolution of the MW distribution as a function of temperature and time for Starfire SMP-10 (a commercial polycarbosilane). The enthalpy of vaporization, the temperature dependence of the enthalpy of vaporization, the temperature dependence of the normal boiling point and a representation of the molecular weight distribution were fit using a series of thermo-gravimetric measurements, involving isothermal holds on a particular batch of SMP-10. Once calibrated for SMP-10 in this fashion, the molecular weight distribution of different batches of SMP-10 could be fit using a thermo-gravimetric measurement involving a reduced temperature-time series. The model was then predictive of mass loss over time for temperatures below the onset of curing (>90°C). Control of volatiles allows for improved SiC yield, reduced processing time and minimizing void/bubble formation.

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

Smart Functional Ceramic Coatings

Room: St. John

Session Chair: Eugene Medvedovski, Endurance Technologies Inc.

1:30 PM

(ICACC-S2-039-2017) Review: High-Temperature, Thin Film Sensors for CMCs (Invited)

O. Gregory*[1]; 1. University of Rhode Island, USA

A key enabling technology to support ceramic matrix composite (CMC) engine component development is strain and temperature measurement. Many issues arise when attempting to instrument CMC's, considering they will be exposed to supersonic velocities, and gas temperatures approaching 1500°C. Thin film sensors are ideally suited for instrumenting CMC components, since they are deposited directly onto the surface of the CMC and become an integral part of the surface. Thin film sensors do not require adhesives and can survive the extremely large "g" forces. However, critical to their performance is the formation of a stable dielectric coating to electrically isolate the sensors from the CMC substrate. Traditional metallic thin film thermocouples, including those based on platinum/rhodium, can suffer from reliability issues above 900°C due to selective oxidation, as well as thinning and poor adhesion to oxide surfaces due to de-wetting of the films. Seebeck coefficients as large as 160 $\mu V/°C$ at 1300°C have been measured for ceramic thin film thermocouples based on indium-tin-oxide (ITO) alloys. These same ITO alloys do not suffer from oxidation effects and also exhibit relatively large gage factors. The merits of using metallic and ceramic thin film sensors for CMC's will be reviewed and the most promising approaches for electrical isolation, temperature and strain measurement will be presented.

2:00 PM

(ICACC-S2-040-2017) Surface Mountable, High-Temperature Strain Gages for CMC's

V. Wnuk*[1]; O. Gregory[2]; 1. HPI, USA; 2. University of Rhode Island, USA

A key enabling technology to support ceramic matrix composite engine component development is the ability to measure strain at temperatures up to 1300°C. Thin film semiconductor strain gages are ideally suited for instrumenting CMC components. The high gage factor is necessary for stress analysis. Thin film sensors are deposited directly onto a surface of a component so that the instrumentation becomes an integral part of the surface of a component. Deposition is usually done in a sputtering machine with a large vacuum chamber. However, when large components have to be instrumented, the size of the component to be instrumented will be limited by the size of the vacuum chamber in the sputtering system. High temperature thin film strain gages can be batch fabricated on a large alumina preform and divided into individual strain gage coupons. Batch quantifying of gage factor can be established before usage on the CMC part. Thin film strain gages based on indium tin oxide (ITO) were fabricated onto a very thin substrate in such a way that the individual strain gage coupons could be easily cemented to the surface of CMC components. The advantages in using surface mountable semiconductor sensors to instrument CMC's for strain measurement will be discussed and results of testing these ultra-high temperature backed sensors relative to other strain gages used to instrument CMC's will be presented.

2:20 PM

(ICACC-S2-041-2017) High-Resolution, High Stability Temperature and Strain Sensors for Advanced CMC's

K. Rivera*[1]; T. Muth[1]; J. Rhoat[1]; O. Gregory[1]; 1. University of Rhode Island, USA

As more SiC-SiC ceramic matrix composites or CMC's are being used in the hot sections of gas turbine engines, there is a greater need for low drift rate, high-resolution sensors to measure the strain and temperature in these harsh environments. Currently, few choices exist for in-situ monitoring of these SiC-SiC CMC components. Thin film thermocouples, for example, are ideally suited for such harsh environments, since they have a very small thermal mass and are non-invasive due to their low profile. However, they can suffer from stability issues at elevated temperatures due to the small diffusional distances associated with thin films. Alternatively, thermocouples utilizing the SiC-SiC CMC itself as one thermoelement and platinum as the other thermoelement were fabricated on CMC's. Here, the bulk properties of SiC determine the sensor performance rather than those of a deposited thin film. In addition, extraordinarily large thermoelectric powers (180 μV/°K) were realized with this Pt:SiC(CMC) thermocouple due to the contribution of the SiC in the CMC. Similarly, strain gages utilizing the SiC-SiC CMC itself as the active strain element were fabricated on CMC's. Here, the SiC in the CMC contributes to very large gage factors. The advantages of using this new approach for strain and temperature measurement as well as fabrication details and experimental results will be presented.

2:40 PM

(ICACC-S2-042-2017) Magnetic studies of Copper incorporated Iron Nitride Thin Films

H. Kamat*[1]; X. Wang[1]; J. Parry[2]; H. Zeng[2]; 1. Alfred University, USA; 2. University of Buffalo, USA

Iron nitride thin films are being considered in various applications due to their high saturation magnetization values. Their magnetic properties can be tuned for specific applications, by either adding a non-magnetic layer or by adding a third element to the iron nitride films during fabrication. For example, stacked layers of iron nitrides and copper nitrides have been investigated as promising candidates for magnetic tunnel junctions. In this paper we report a systematic study of copper incorporated iron nitride thin films fabricated by co-sputtering iron and copper in an environment of N_2 and Ar using reactive magnetron sputtering. These thin films have been deposited on Si [100] substrate and the copper sputter power and nitrogen flow rates have been systematically varied to study their effects on the structure and properties of the film. The films have been characterized by grazing incidence x-ray diffraction, grazing incidence x-ray reflectivity, field emission scanning electron microscopy, energy dispersive spectroscopy and x-ray photoelectron spectroscopy. Finally, the magnetic properties of these films have been measured using a vibrational sample magnetometer and the values correlated with the structure and composition of the films.

Advanced Multifunctional Ceramic Coatings

Room: St. John
Session Chair: Otto Gregory, University of Rhode Island

3:20 PM

(ICACC-S2-043-2017) Boride-Based Coatings for Protection of Cast Iron against Wear

E. Medvedovski*[1]; J. Jiang[2]; M. Robertson[2]; 1. Endurance Technologies Inc., Canada; 2. National Research Council of Canada, Canada

The components made of cast irons require protection against wear in severe service conditions. Surface engineering can be used to prevent mechanical failures of cast iron components due to excessive friction-related wear. The iron boride-based coatings can be applied on the entire working surfaces of large size complex shape cast iron components through the thermal diffusion process. Tribological properties of these coatings obtained have been studied using the pin-on-disc test configuration in simulating application conditions. The obtained iron boride coatings demonstrated significantly lower wear losses compared to bare cast iron, stable behavior of coefficient of friction during time and no structural degradation and spalling. The superior wear resistance of boride-based coatings on cast iron is dealt with the combination of their high hardness, specific "saw-tooth" double-layer morphology obtained through the thermal diffusion process, diffusion-related bonding to the substrate, self-lubricating thin "tribofilm" formed during friction and high thermal and chemical stability.

3:40 PM

(ICACC-S2-044-2017) Evaluation of protective coatings for superheater tubes for waste to energy plants

S. Molin*[1]; L. Mikkelsen[2]; M. Chen[1]; P. Hendriksen[1]; 1. Technical University of Denmark, Denmark; 2. Babcock & Wilcox Vølund A/S, Denmark

High temperature corrosion remains a main challenge in operation of superheater tubes in power plants. Exposition to highly corrosive gases, containing alkali compounds can lead to rapid and breakaway corrosion. In this work coatings based on alumina and yttria are deposited on superheater tubes and are evaluated for their corrosion performance. Novel deposition methods are used to prepare dense coatings at low temperatures. Exposures in air and in simulated flue gas are performed in a specially designed rig. Weigh gain, oxide thickness and composition are determined in order to quantify protective behavior of the layers.

4:00 PM

(ICACC-S2-045-2017) Controlling the phase composition in Lanthanum Tungstate for Hydrogen Gas Separation Membranes by Plasma Spray-Physical Vapor Deposition

D. A. Marcano*[1]; G. Mauer[1]; R. Vaßen[1]; 1. Forschungszentrum Juelich, Germany

Lanthanum tungstates are crystalline mixed proton-electron conductors which are attractive for dense hydrogen gas separation membranes operating above 600°C. In this work, dense $La_{6-x}WO_{12-d}$ (x=0.3-0.6) membranes directly deposited on porous metallic substrates by Plasma Spray-Physical Vapor deposition (PS-PVD) were studied. The layers were investigated by means of high temperature X-ray diffraction (HT-XRD), scanning electron microscopy (SEM), inductively coupled plasma atomic emission spectroscopy (ICP-AES) and the feedstock evaporation during PS-PVD by optical emission spectroscopy (OES). Starting from a powder consisting of cubic La_6WO_{12} and the secondary phase $La_6W_2O_{15}$, it was found that independently of the oxygen partial pressure during deposition, coatings consisted of the same phases: cubic La_6WO_{12}, La_2O_3 and hexagonal LaWO. Previous work on tape-casted membranes showed that the formation of $La_6W_2O_{15}$ led to crack formation in the membranes. With PS-PVD as processing route however, it was possible to control the formation and limit the amount of secondary phases. Additionally, it was found that once secondary phases are formed, they are stable during annealing in air.

4:20 PM

(ICACC-S2-046-2017) Microstructure and Properties of Room Temperature Deposited, Thick $BaTiO_3$ Dielectric Films

P. Sarobol*[1]; A. Vackel[1]; J. Adamczyk[1]; T. D. Holmes[1]; M. Rodriguez[1]; J. Griego[1]; H. Brown-Shaklee[1]; 1. Sandia National Laboratories, USA

$BaTiO_3$-based dielectrics are being explored for high temperature stable capacitor applications to enable high power electrical switching devices. The high sintering temperature of $BaTiO_3$ (T>1000°C) often prevents successful integration with low melting point substrates--glass, metal, or plastic. In this work, we demonstrate integrated high density $BaTiO_3$-based thick films at room temperature (RT) utilizing a novel, solid-state deposition process, Aerosol Deposition (AD). In AD, high velocity submicron particles impact, deform, and consolidate as coatings on substrates, at RT, under vacuum. The AD $BaTiO_3$ film crystal structure, grain size, residual strain, and dielectric properties were investigated. Preliminary results from XRD analysis showed an in-plane crystallographic strain in our AD films were ~1%, corresponding to ~1GPa compressive stress. The impact of this significant crystallographic strain on dielectric properties will be discussed. This work is supported by Office of Electricity and Laboratory Directed Research and Development Program at Sandia National Laboratories. Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. DOE's National Nuclear Security Administration under contract DE-AC04-94AL85000.

4:40 PM

(ICACC-S2-047-2017) Effect of Microstructural Characteristics on Thermal and Electrical Properties of Thermal Spraying Deposited Ceramic Coatings

F. Azarmi*[1]; E. Mironov[2]; 1. North Dakota State University, USA; 2. Skolkovo Institute of Science and Technology, Russian Federation

Aluminum oxide ceramic samples were fabricated using a thermal spraying deposition technique. In general, the global properties of thermal sprayed coating materials are different from those of conventionally processed. This paper investigates thermo-electrical properties of ceramic coatings using available theoretical, numerical simulation, and experimental techniques. Due to anisotropic properties of coating materials, all properties were measured in both in transverse and longitudinal microstructural directions. Numerical simulation was also performed on both transverse and longitudinal directions. Comparison between the results from the analytical models, experimental techniques, and numerical simulation helped us to better understand the influence of microstructural characteristic on the thermal and electrical conductivity of thermal spraying deposited ceramic materials.

S3: 14th International Symposium on Solid Oxide Fuel Cells (SOFC): Materials, Science and Technology

Mechanical Integrity

Room: Crystal
Session Chair: Vijay Srivastava, GE Global Research

1:30 PM

(ICACC-S3-035-2017) Redox stable anode materials for SOFC

T. Thomas*[1]; E. Sabolsky[1]; H. Qi[1]; X. Liu[1]; J. Zondlo[2]; R. Hart[3]; E. Jezek[3]; 1. West Virginia University, USA; 2. West Virginia University, USA; 3. GE Global Research Centre, USA

The solid-oxide fuel cells (SOFCs) may undergo unwanted redox cycles during application and this may cause dimensional instability for the anode electrode. The state-of-the-art Ni/YSZ anode experiences a linear expansion of up to 1% when oxidized causing an irreversible microstructural change. Researchers are focusing on the development of many new SOFC anodes for internal reforming, lower temperature operation, and poison resistance, but there is a lack of study on the redox stability of the materials. In this work, the redox stability of various perovskite and layered-perovskite materials were tested by controlled-atmosphere dilatometry. $La_{0.35}Sr_{0.65}TiO_3$ (LST) and $Sr_2MgMoO_{6-\delta}$ (SMM) samples were subjected to 5 hours redox cycles in air and forming gas. Their linear expansion at isothermal conditions were compared to commonly used anode materials like Ni/YSZ and GDC. For example, it was found that 38% porous LST experienced a reversible linear expansion of 0.013% at an isothermal of 800°C. The change in microstructure of these samples after redox cycling were also studied. The electrical performance were also measured by four-point conductivity. Apart from the above mentioned compositions, the redox performance of other non-traditional compositions with various doping strategy will also be discussed in this work.

1:50 PM

(ICACC-S3-036-2017) Fracture Mechanisms in Solid Oxide Fuel Cell Anode Supports

K. Kwok*[1]; P. Jørgensen[1]; J. Wei[2]; G. Pecanac[2]; H. Frandsen[1]; J. Malzbender[2]; 1. Technical University of Denmark, Denmark; 2. Forschungszentrum Jülich GmbH, Germany

Mechanical reliability poses a major challenge for solid oxide fuel cells (SOFC). Current understanding on the failure modes and mechanical properties of ceramic components is limited, and needs to be improved for developing mechanically robust SOFC. Fracture of ceramic cells is one of the prominent failure modes that degrade SOFC performance. This work is an investigation into the fracture behavior of anode supports made of porous nickel (oxide) yttria-stabilized zirconia Ni(O)-YSZ. The focus is to elucidate how fracture interacts with the microstructure of the anode supports. We study the fracture paths in partially cracked anode supports obtained from double torsion experiments by detailed fractography. It has been identified that operative fracture mechanisms include brittle fracture of NiO and YSZ phases, debonding of Ni/YSZ interface, and most importantly plastic necking of Ni phase. To quantitatively characterize microstructural parameters affecting the fracture behavior, we reconstruct three-dimensionally the crack surfaces using focused ion beam serial sectioning and scanning electron microscopy (FIB-SEM). Analytical models relating the microscopic mechanisms with fracture toughness of anode supports are provided and compared against measurements.

2:10 PM

(ICACC-S3-037-2017) Effect on Sintering Aid and YSZ Short Fibers on Densification and Contact Strength of Solid Oxide Fuel Cells

Y. Chou*[1]; J. Bonnett[1]; J. W. Stevenson[1]; 1. Pacific Northwest National Lab, USA

The mechanical integrity of 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 temperature which led to poor solid-state sintering and low strength. To improve the contact strength, one could use precious metals. However, the cost would be prohibitive. In previous work, we have demonstrated the strength can be improved by engineering cathode surface. In this work we will continue the work by sintering aid such as CuO and Bi_2O_3 as well as YSZ short fibers. Sintering aid or YSZ short fibers will be mixed with LSM20 cathode at various % and sintered at 800-950°C 2h. Density will be measured and bulk strength will be determined with diametral compression test. Ceramic bilayers couples will be joined at elevated temperatures with candidate LSM20 contact and optimum sintering aid as well as YSZ short fibers. Uniaxial tensile strength will be conducted on joined couples at room temperature. Fracture surface analysis will be applied to correlate with strength results. At the end, candidate cathode contact compositions will be tested on a 2"x2" cell in a PNNL's generic stack fixture to assess its effect on thermal cycle stability as well as stability at 800°C. Cell performance and microstructure analysis will be reported.

2:30 PM

(ICACC-S3-038-2017) Characterization of the ferroelasticity of SOFC cathode materials

K. Shishido*[1]; 1. Tohoku University, Japan

The mechanical properties of SOFC components should be precisely understood to prevent mechanical degradation. $La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_{3-\delta}$ (LSCF6428) is one of the typical cathode materials for SOFCs.

LSCF6428 is known to have ferroelasticity at low temperature (about 298 K ~ 800 K). When a ferroelastic material is exposed to a mechanical stress, domains start to reorient to relax the stress. Such a property may affect the mechanical stability and stress state of SOFCs under operation. In this study, the change of the crystal orientation caused by the ferroelasticity at room temperature was evaluated by inducing the macro and micro stress and using EBSD. The uniaxial compression test and EBSD were performed for the polycrystalline LSCF6428. The domain component, where its hexagonal c-axis is almost parallel to the compression direction, increased in intensity during compression. This behavior agrees with previous study on $LaCoO_3$-based materials. However this domain reorientation was not observed in all grains whose c-axis is parallel to the compression stress. It is because the direction of macro stress and doesn't always correspond with that of the stress applied an individual grain. Therefore, the stress state of an individual grain should be controlled for quantitative understanding of influence on mechanical behavior by ferroelasticity.

Novel Processing and Design

Room: Crystal
Session Chair: Andre Weber, Karlsruhe Institute of Technology (KIT)

3:10 PM

(ICACC-S3-039-2017) Development of electrodes for solid oxide electrolysis cells using microstructure control techniques (Invited)

H. Shimada*[1]; T. Yamaguchi[1]; H. Sumi[1]; Y. Yamaguchi[1]; K. Nomura[1]; Y. Fujishiro[1]; 1. National Institute of Advanced Industrial Sience and Technology (AIST), Japan

We have investigated high performance electrodes for solid oxide electrolysis cells by various microstructure control techniques such as porous structure control and starting ceramic powder synthesis techniques. Although higher reactant utilization leads to high efficiency and flexibility in solid oxide electrolysis cell (SOEC) systems, it increases the concentration polarization resistance. To decrease the concentration polarization resistance, porous structure of fuel electrode was controlled via extrusion process. Also, a functional layer to improve fuel electrode reaction was developed using nano-composite powders prepared via spray pyrolysis. Our SOEC showed considerable improvement in fuel utilization and polarization resistance. In our presentation, we will introduce these results in detail and other our resent activity for SOECs.

3:40 PM

(ICACC-S3-041-2017) Fabrication of anode-supported solid oxide fuel cell with direct pore channel in the cermet structure to improve the electrochemical performance

T. Lin*[1]; H. Kuo[1]; J. Kuo[1]; C. Yeh[1]; W. Kao[1]; M. Liao[1]; Y. Chen[1]; R. Lee[1]; 1. Institute of Nuclear Energy Research, Taiwan

An anode-supported solid oxide fuel cell consisting of a NiO-YSZ anode, YSZ electrolyte, and YSZ-LSM || LSM composite cathodes has been investigated. In order to enhance the diffusivity of fuel gas to reach the triple phase boundary for electrochemical reaction, we use tape casting to produce thin strip of green tape anode substrate. A pore array perforation and forming an anode supporting substrate through sintering has been executed, followed by thin film processes to produce an unit cell with low diffusion impedance in the fuel gas electricity generation operation, and lastly removing a nickel depleted layer on the surface of the anode with precise abrasion. By the approach, it can effectively enhance the performance of the cell and provide power output with long term stability. The cell consists of a NiO-YSZ anode, YSZ electrolyte, and YSZ-LSM || LSM composite cathodes and the thicknesses of YSZ, YSZ-LSM, and LSM layers are 8, 15, and 40 μm, respectively. In an electrical performance test of the cell produced by green tape perforation, it shows that the electricity generation efficiency may raise a percentage of 25 %, and the gas diffusion impedance can be lowered by a percentage of 40 %.

4:00 PM

(ICACC-S3-042-2017) Thermal strain control in micro-machined solid oxide fuel cells

M. Hadad*[1]; P. Muralt[1]; 1. Ecole Polytechnque Federale de Lausanne (EPFL), Switzerland

The importance of small-scale energy storage and supply devices has increased with the growing power consumption of portable electronic devices. Today, all such devices are depending on Li-ion batteries. The limited energy density of Li-batteries leads to large research activities for improving the latter, or to find alternatives. With increasing interests on environmental aspects fuel cells are probably the best alternative for commonly used batteries. The fuel cells performance is directly related to the active membrane area and operation conditions. The quality of the thin films is also an important factor. So far, the ideal membrane size has not been formulated since the limitations are strongly related to the fabrication process. We proposed a new design to fabricate micro-SOFCs membrane for durable performance. The idea is to facilitate the deflection when residual stress, or thermal stress is imposed during fabrication and operation of the cell. Unpredictable buckling with a high risk of cracking is thus avoided. We were able to fabricate > 200μm wide membranes without any buckling, which is very favorable for the mechanical stability of the membrane. The idea is based on a predefined corrugation that controls deformation strain. Moreover, we have successfully deposited 600 nm thick Yttrium stabilized zirconia over the corrugated structures with high conformity and uniformity using magnetron sputtering.

4:20 PM

(ICACC-S3-043-2017) Towards a Scalable and Automated Impregnation Process for Solid Oxide Fuel/Electrolysis Cells

E. H. Daas[2]; J. T. Irvine[3]; E. Traversa[4]; S. Boulfrad*[1]; 1. Hamad Bin Khalifa University – Qatar Foundation, Qatar; 2. King Abdullah University of Science and Technology, Saudi Arabia; 3. University of St Andrews, United Kingdom; 4. Xi'an Jiaotong University, China

The triple phase boundary (TPB) sites are key element in solid oxide fuel and electrolysis cells (SOCs). Larger TPB leads to improved performance of the electrodes and thus the device. Impregnation based processes were established to enhance the performance by a substantial increase of the TPB density. A porous backbone structure, usually formed by the electrolyte material, is impregnated with a liquid precursor of the electrode active phase. After appropriate heat treatment, the impregnated solid phase is synthesized in the form of a very fine structure on the top of the porous scaffold. This leads to TPB much larger than with the usual powder mixing methods, in addition to an excellent percolation of both solid phases. The literature is full of impressive results with different cathode and anode materials. However, because it is usually manually performed, its irreproducibility remains a major problem. In this work Inkjet printing was used to impregnate porous yttria stabilized zirconia (YSZ) structure by a liquid precursor of $La_{0.8}Sr_{0.2}MnO_3$ (LSM). The process was optimized in order to control the 3D dispersion of the impregnated liquid in the porous scaffold. After only one printing-firing cycle, pure LSM phase was formed with particle size around 20 nm. The polarization resistance was equal to 0.29 Ω cm^2 at 700°C, among the best reported in the literature for these materials.

S5: Next Generation Bioceramics and Biocomposites

Bioceramics I

Room: Coquina Salon F
Session Chairs: Alberto Vomiero, Lulea University of Technology; Ajay Karakoti, Ahmedabad University; Akiyoshi Osaka, Okayama University

1:30 PM

(ICACC-S5-001-2017) Nanoscale structure and properties of biocompatible materials (Invited)

F. Rosei*[1]; 1. INRS, Canada

Modifying the nanostructure/chemistry of materials allows to optimize their properties. Our strategy rests on creating nanopatterns that act as surface cues, affecting cell behavior. Chemical oxidation creates unique topographies, becoming a general strategy to improve biocompatibility. Our treatment selectively inhibits fibroblast growth while promoting osteogenic cell activity in vitro. Enhancement of mechano-biocompatibility may occur by coating with spider silk. Improvement of antibacterial properties using laser and plasma strategies will also be discussed, as well as perspectives in nanoscale thermometry.

1:50 PM

(ICACC-S5-002-2017) Effect of etching on the surface modification of silica nanostructures for tuning the uptake and release of hydrophobic drugs

A. S. Karakoti*[1]; H. Thawani[2]; S. Singh[2]; 1. Ahmedabad University, India; 2. Ahmedabad University, India

Silica nanoparticle based nanocarriers or nanosilica are widely used in drug delivery applications on account of their excellent stability, biocompatibility, inertness and ability to undergo surface modifications by various silane coupling agents. In the present study, solid and mesoporous silica nanoparticles (MSNs) were synthesized using conventional methods. These nanoparticles were etched using a combination of HF/water, HF/ethanol and their mixtures. Docetaxel, a hydrophobic drug, was loaded on as-synthesized and etched nanostructures and its total loading, as well as, release was studied at pH 7.4 and pH 4 in phosphate buffer saline. It was found that increasing the surface area of SNPs, by etching its surfaces, increased the drug loading however, the drug adsoprtion was also influenced by the polarity of the resulting surfaces. After etching in HF/Ethanol, hydrophobic drug was bound tightly on silica surface and was released slower as compared to unetched surfaces. Silica particle before and after etching were characterized using DLS, TEM and FTIR and the surface chemistry was probed using XPS. A comparative study of the DTX loading and release from as-synthesized and etched-SNPs and the role of different surfaces in tuning the uptake and drug release will be presented.

2:10 PM

(ICACC-S5-003-2017) Multifunctionalized Fe3O$_4$@SiO$_2$ on SiO$_2$ Dual-Faced "Janus" Nanoparticle for Targeted Drug Delivery (Invited)

D. Shi*[1]; 1. University of Cincinnati, USA

Nanotechnology offers highly advanced targeting and efficient delivery of therapeutic agents. Therapeutic agents which alone possess disseminated, non-specific delivery. Recently reported dual-faced nanoparticles composed of a polystyrene core with a hemispherical coating of magnetite (Fe_3O_4) imbedded polystyrene (PS) has allowed for multifunctionalization of folic acid for folate receptor targeting on cancer cells as well as conjugation and subsequent delivery of doxorubicin, a potent chemotherapeutic which natively possesses no selective action. Transmission electron microscopy reveals monodisperse "Janus" nanoparticles. Folic acid was covalently attached to carboxylate groups on the surface of the PS core introduced through the 4,4'-azobis(4-cyanovaleric acid) initiator using carbodiimide coupling with a poly(ethylene glycol) bis(amine) spacer. Doxorubicin was covalently functionalized to the magnetite imbedded PS coat through hydrazone bond formation with adipic acid dihydrazide crosslinked to 3-(triethylsilyl)propyl isosyanate included in the PS coat. A pH dependent release of doxorubicin was observed with a lower pH driving greater cumulative release profiles. Folic acid functionalization of doxorubicin loaded nanoparticles significantly lowered the median lethal dose in-vitro when compared against doxorubicin loaded nanoparticles without folic acid functionalization.

2:30 PM

(ICACC-S5-004-2017) Antifouling characteristics of alginic acid deposited on stainless steel substrate (Invited)

T. Yoshioka[3]; K. Tsuru[2]; S. Hayakawa[3]; A. Osaka*[1]; 1. Okayama University, Japan; 2. Kyushu University, Japan; 3. Okayama University, Japan

Antifouling characteristics are of common requirements for implant materials in constant contact with blood. So far some hydrophilic coatings on the implant materials have been widely accepted for practice. Here, the integrity of such coatings with material surface is an urgent subject since it would be sometimes degraded in aqueous environments like in our body. In this study, we immobilized hydrophilic molecular-layers of alginic acid on an oxide layer of stainless steel (SUS) substrate via the APS-mediated covalent bonding or ionic interactions, and examined their antifouling property using bovine serum albumin (BSA). [APS: aminopropyltriethoxysilane] Interestingly, the covalently immobilized alginic-acid molecules prevented BSA adsorption, while the directly bonded ones due to the ionic interactions favored the BSA adsorption. FT-IR RAS spectroscopic studies indicated that those behaviors were interpreted on the basis of the chemical bonding states of the carboxyl groups of alginic acid on to the oxide layer. We therefore further discussed the potential roles of the carboxyl group of alginic acid for the antifouling property based on the proteins adsorption experiments in different pH conditions, and on chemical modifications of the carboxyl group.

3:10 PM

(ICACC-S5-005-2017) Structural investigation of biominerals surface and organo-mineral interface through solid state NMR (Invited)

T. Azaïs*[1]; W. Ajili[1]; G. Laurent[1]; S. Von Euw[1]; N. Nassif[1]; 1. Université Pierre et Marie Curie, France

In this communication, we will show how solid state nuclear magnetic resonance (ssNMR) can allow the structural characterization of surfaces and interfaces for natural biominerals with a focus on bone mineral. Bone mineral consists of nanoplatelets structurally close to hydroxyapatite $Ca_{10}(PO_4)_6(OH)_2$. It has been proposed since the late 50's that bone mineral consists of a crystalline apatitic core covered by an independent mineral domain. We will show how a combination of advanced solid state NMR experiments applied to fresh and intact bone samples leads to an unprecedented structural characterization of that outer domain: in particular, we demonstrated that the surface layer of bone mineral is amorphous, highly hydrophilic and composed of divalent ions: Ca^{2+}, HPO_4^{2-}, CO_3^{2-}. Finally, we will discuss the benefit of using dynamic nuclear polarization (DNP) combined to ssNMR to selectively enhanced NMR signals from bone mineral surface species. Indeed, DNP is a recently developed technique that allows the magnetization transfer from radicals unpaired electrons to nuclei inducing a huge enhancement of the NMR signal (up to 150 !). The organo-mineral interface of bone mineral can be readily investigated through 13C-31P REDOR experiments in few hours instead of weeks using conventional ssNMR.

3:30 PM

(ICACC-S5-006-2017) Charge dynamics in composite nanosystems (Invited)

A. Vomiero*[1]; 1. Lulea University of Technology, Sweden

Charge dynamics (i.e. the processes of charge generation, dissociation and collection) plays a critical role in several advanced applications based on composite nanosystems, including solar energy, water splitting, nanothermometry. In most of them, semiconducting nanocrystals exhibiting quantum confined effects (the so-called quantum dots, QDs) act as light absorbing materials, which are able to generate excitons as a consequence of photon absorption. Managing the photogenerated charges enables the exploitation of different processes, including charge separation and collection in photoelectrochemical systems for energy conversion or tuning photoluminescence properties in luminescent nanoprobes. Key element for driving the processes to targeted applications is the modulation of composition and size of the nanomaterials, which determines the electronic band structure of the systems and its functional properties. We will illustrate different examples of composite systems. (i) “Giant” composite core-shell QDs, in which modulation of core-to-shell interface induces a single- to double-color photoluminescence; (ii) Near-infrared QDs with increased Stokes shift due to suitable electronic structure of the core and shell, to be applied in luminescent solar concentrators; (iii) Composite TiO_2 mesoporous film sensitized by “giant” QDs with high charge injection from the photoexcited QD to the TiO_2 anode, for excitonic solar cells and water splitting.

3:50 PM

(ICACC-S5-007-2017) Tantalum and Tantalum-based Ceramic Coatings for Increasing the Biocompatibility of Conventional Metal Implant Alloys (Invited)

J. Stiglich*[1]; B. Williams[1]; R. Narayan[2]; 1. Ultramet, USA; 2. University of North Carolina, USA

Tantalum has long been used as an implant material in bone and soft tissue, and Ultramet developed and licensed a process for fabricating open-cell tantalum metal foam bone implants that is approved by the Food and Drug Administration. Under NASA funding Ultramet developed a process to diffuse highly corrosion-resistant tantalum metal into the surface of conventional stainless steels and superalloys to improve acid corrosion resistance of precision valve components. A thin, metallurgically bonded surface layer was established that grades from pure tantalum to a mixture of tantalum and the substrate elements. The tantalum surface layer precisely replicates intricate substrate features and does not require machining. This diffusion coating can increase the biocompatibility of conventional metal implant alloys, and the ability to harden the surface via partial or full conversion to tantalum nitride has been demonstrated. Additional surface treatments to alter texture may be used to improve biological functionality (e.g. enhance bone formation) of dental and orthopedic implants.

4:10 PM

(ICACC-S5-008-2017) Control of Interlayer Distance of Octacalcium Phosphate Using Dicarboxylate Ions (Invited)

T. Yokoi*[1]; M. Kamitakahara[2]; C. Ohtsuki[3]; 1. Japan Fine Ceramics Center, Japan; 2. Tohoku University, Japan; 3. Nagoya University, Japan

Octacalcium phosphate (OCP) is known as one of bioresorbable materials. OCP has a layered structure and incorporates dicarboxylate ions in the interlayer by substitution of HPO_4^{2-} and resulting in formation of inorganic-organic hybrid materials. OCP with incorporated dicarboxylate ions is called as OCP carboxylate (OCPC). Physicochemical properties, such as solubility, of OCP were controlled by incorporation of dicarboxylate ions. Additionally, incorporation of pharmaceutical molecules into OCP may lead development of calcium phosphate biomaterials with drug release function. OCPCs have potential to be an innovative ceramic biomaterial. Kinds of dicarboxylate ions which can be incorporated into OCP have been investigated. As the next step, studies about control approach of the interlayer distance of those layered substances, namely OCPCs, were worthwhile for expanding calcium phosphate chemistry as well as development of OCPC biomaterials. In this presentation, we introduce our synthetic process of OCPCs using calcium carbonate and phosphoric acid as starting materials as well as control of interlayer distance using two kinds of aliphatic dicarboxylate ions and using a dicarboxylate ion with side chain.

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

Thermal Energy Conversion and Energy Storage IX

Room: Tomoka A

Session Chair: Xin Guo, Huazhong University of Science and Technology

1:30 PM

(ICACC-S6-031-2017) In-situ TEM Probing of Nanoscale Surface Coating Layer on Mitigating Capacity Fading of Lithium ion Battery (Invited)

C. Wang*[1]; 1. Pacific Northwest National Laboratory, USA

Capacity fading is always a big challenge for lithium ion batteries. The fading can be contributed by a range of factors, such as degradation in cathode, anode, electrolyte, and the interface between them or a combination and coupling of all these factors. Recent studies have shown that applying nanometer-thick coating layers on either anode (such as Si nanoparticle) and cathode (such as lithium transition metal oxide) can enhances cyclability and capacity retention. However, it is far from clear how the coating layer function from the point of view of both surface chemistry and electrochemi-mechanical effect. In this presentation, we use ex-situ and in-situ TEM to evaluate the functioning mechanism of coating effect on the lithiation kinetics of Si anode and lithium transition metal oxide cathode. Evaluating from the point of view of both surface chemistry and electrochemi-mechanical effect, we found that the coating layer can play dual roles of both beneficial and detrimental. Our findings suggest the significance of the coupled electrochemi-mechanical effects of surface coatings in the design of high-performance Si-based anodes for lithium ion batteries. For the case of cathode, it appears that the surface chemical reaction will be the dominate factor.

2:00 PM

(ICACC-S6-032-2017) Carbon-Based Composite Electrodes for High-Performance Energy Storage Devices (Invited)

T. Liu[1]; B. Lee[1]; S. Lee*[1]; 1. Georgia Institute of Technology, USA

Lithium-ion battery has become a leading energy storage technology for various applications from consumer electronics to electric vehicles. However, large-scale energy storage applications, such as smart grid and renewable energy storage, further drive the development of low-cost and sustainable electrode materials. Therefore, cost-effective organic electrode materials have been extensively studied to replace conventional transitional metal based inorganic cathodes for lithium-ion batteries. We also studied the charge storage properties of various carbon materials, including carbon nanotube, graphene, small organic molecules, and polymers in lithium- and sodium-cells. We demonstrated enhanced charge storage properties of these carbon-based electrodes by utilizing the surface redox reactions with lithium and sodium ions. These carbon-based electrodes can deliver high specific capacities over 120 mAh/g with enhanced rate-capability in both lithium- and sodium-cells. We also attempt to introduce external redox-active inorganic materials into the porous carbon electrodes to further increase the energy density of the composite electrodes.

2:30 PM

(ICACC-S6-033-2017) 3D Printed carbon materials enabled energy-related applications (Invited)

K. Fu*[1]; L. Hu[1]; 1. University of Maryland, USA

Additive manufacturing (AM) technique, known as three-dimensional (3D) printing, has attracted much attention in industry and academia in recent years. 3D printing has been developed for advanced materials, architectures, and systems in a broad range of applications. Printable inks are the most important component for 3D printing, which are directly related to material preparation, printing strategy, and architectural design of the final 3D printed products. Recently, graphene oxide (GO) has shown noteworthy printing capabilities and unique viscoelastic properties as an aqueous dispersion wherein its rheological behavior varies considerably with the graphene oxide concentration. Studies about GO in 3D printing have been extensively reported, particularly using the porous 3D graphene structures to demonstrate various energy-related applications. In this circumstance, understanding the very recent development of 3D printed graphene oxide and its extended applications to address energy-related challenges and bring new concept for material designs are becoming urgent and important. This presentation will discuss recent development in 3D printing of emerging devices for energy-related applications, including energy storage applications and thermal energy applications at high temperature and future designs and developments of 3D printing technology based on carbon materials for energy-related applications and beyond.

Thermal Energy Conversion and Energy Storage X

Room: Tomoka A

Session Chair: Ryoji Funahashi, National Institute of Advanced Industrial Science and Technology (AIST)

3:20 PM

(ICACC-S6-034-2017) Recent Progress in Thermoelectric Complex Sulfides (Invited)

E. Guilmeau*[1]; P. Ventrapati[1]; T. Barbier[1]; C. Bourgès[1]; P. Lemoine[2]; O. I. Lebedev[1]; 1. CNRS CRISMAT, France; 2. Institut des Sciences Chimiques de Rennes, France

Recently, copper-containing ternary sulphides have been attracting much attention for thermoelectric applications because most of the main components comply with the actual requirements of low cost, earth abundant, and less toxicity. A non-exhaustive list includes p-type tetrahedrite $Cu_{12-x}Tr_xSb_4S_{13}$ (ZT ~ 0.8 @ 700K) with Tr = Mn, Fe, Co, Ni, Zn ($x \leq 2$), colusite $Cu_{26}V_2Sn_6S_{32}$ (ZT ~ 0.56 @ 663K), bornite Cu_5FeS_4 (ZT ~ 0.55 @ 550K), Cu_2SnS_3 (ZT ~ 0.56 @ 750K), Cu_2ZnSnS_4 (ZT ~ 0.35 @ 700K) or n-type $Cu_4Sn_7S_{16}$ (ZT ~ 0.21 @ 700K). A common feature of tetrahedrite, colusite, bornite and $Cu_4Sn_7S_{16}$ phases is their intrinsically low thermal conductivities κ originating from high structural complexities, i.e. large number of atoms per unit cell and large Grüneisen parameter. In addition, low-energy vibration mode of Cu atom out of the $[CuS_3]$ triangular planar unit in tetrahedrites explains its ultra low thermal conductivity. Same phenomena was supposed in $Cu_4Sn_7S_{16}$. In the case of the p-type Cu_5FeS_4 bornite and $Cu_{26}V_2Sn_6S_{32}$ colusite, the low lattice thermal conductivity value was mainly attributed to the structural complexity and mass difference between Cu, V and Sn atoms. During this presentation, recent progress on these thermoelectric complex sulfides will be described. Synthesis, processing, shaping, as well as structural and microstructural features will be reported, together with electrical and thermal properties.

3:50 PM

(ICACC-S6-035-2017) Thermoelectric Performance of Ternary and higher Cu Chalcogenides (Invited)

H. Kleinke*[1]; 1. University of Waterloo, Canada

Thermoelectric materials can convert waste heat - otherwise lost - into useful electricity. The Seebeck effect has been utilized since decades in space exploration missions from Voyager to Curiosity, and currently TE materials are being investigated for commercial waste heat recovery in stationary applications as well as passenger and heavy duty vehicles. In turn, the opposite effect - converting electricity into temperature gradients - is used for (Peltier) cooling in portable coolers and laptops, as well as possibly for temperature control in car seats. As one of the emerging technologies, certain obstacles remain to be addressed, including efficiency and availability of these materials. Copper chalcogenides are among the materials classes of interest, because the disorder and mobility of the copper ions typically causes a very low thermal conductivity, advantageous for the thermoelectric energy conversion. On the other hand, this mobility has led to device decay, as the copper ions moved to one side of the material, resulting in growing copper wires. Therefore, we set out to contain this movement within isolated Cu clusters, by adding additional large cations such as barium ions to impede Cu mobility. In this talk, I will present our efforts into this materials family, which were evidently successful in uncovering at least one such new material with the desired properties.

4:20 PM

(ICACC-S6-037-2017) Continuously Graded Microstrucutre in a Thermoelectric ZnO Material

C. L. Cramer*[1]; J. Gonzalez-Julian[2]; P. Colasuonno[1]; T. B. Holland[1]; 1. Colorado State University, USA; 2. Forschungszentrum Juelich, Germany

ZnO is a promising thermoelectric generating (TEG) material because of its high Seebeck coefficient and doping feasibility. Also oxide thermoelectrics are of interest for waste heat recovery in the mid- to high-temperature ranges. Properties of thermoelectrics have been exploited to make homogenous improvements with carrier concentration, and in some cases non-homogenous functionally graded materials (FGM) have been achieved. Nanostructuring and atomic layering have been done to achieve decreased thermal conductivity, but one way to improve overall efficiency and widen the useful temperature range of bulk material involves a thermal conductivity gradient. To achieve this, the grain sizes must be graded significantly. Functionally graded ZnO is fabricated with water sintering strategies with spark plasma sintering (SPS) as well as advanced, modified tooling, and the grain size gradient in 10 mm of fully dense material is almost an order of magnitude. The basis of the manufacturing is SPS under a large enough thermal gradient. The effects of this system are compared to isothermal sample camparisons or represenations, and possible constrained sintering effects are presented. Finally, the thermoelectric measurements of the FGM are measured and compared to samples with uniform grain size, and the effects are presented for efficiency and useful temperature bandwidth expansion.

4:40 PM

(ICACC-S6-039-2016) Impact of Advanced Transmission Electron Microscopy (TEM) to the structure /properties of energy materials (Invited)

O. I. Lebedev*[1]; M. Freire[1]; D. Berthebaud[1]; A. Maignan[1]; V. Pralong[1]; 1. Laboratoire CRISMAT, UMR 6508 CNRS/ENSICAEN/UCBN, France

Advanced TEM plays an increasingly important role in the understanding of the relationship between microstructure and energy material properties. The uniqueness of TEM is the ability to obtain crystallographic, chemical, electronic and atomic structural information with excellent spatial (0.5A) and energy (0.3eV) resolution simultaneously. Developing of new TEM techniques allows to localize not only heavy atoms but also light elements

(O, Li...) which is extremely important for battery materials. A new electrochemically active rock-salt type nanostructured $Li_4Mn_2O_5$ compound showing a discharge capacity of 355 mAh/g, prepared by direct mechanochemical synthesis at room temperature, was studied by advanced TEM. The magnetic and thermoelectric properties of $Bi_{1.8}V_8O_{16}$ have been studied in connection with their crystal structure characterized by advanced TEM. Notably, the presence of vanadium cations, inserted in the wider tunnels of hollandite structure, was demonstrated. It is found that the $BiCu_{1-x}OS$ (with x<0.20) oxysulfide tends to adopt a constant amount of copper vacancy corresponding to x=0.05 in the $BiCu_{1-x}OS$ formula. For larger Cu deficiencies (x>0.05 in the nominal composition), other types of structural nanodefects are evidenced such as oxysulfides of the "BiOS" ternary system which might explain the report of superconductivity for the $BiCu_{1-x}OS$ oxysulfide.

S7: 11th International Symposium on Nanostructured Materials: Functional Nanomaterials and Thin Films for Sustainable Energy Harvesting, Environmental and Health Applications

New Materials and Processing I

Room: Coquina Salon A
Session Chair: Roger Narayan, NC State University

1:30 PM

(ICACC-S7-030-2017) Soft Processing for Nano Carbons: Direct Fabrication of Functionalized Graphenes and Their Hybrids Inks via Submerged Liquid Plasma and Electrochemical Exfoliation under Ambient Conditions (Invited)

M. Yoshimura*[1]; 1. National Cheng Kung University, Taiwan

Nano-carbons like Grapnenes have greatly been interested in various fields of researches, where the large scale synthesis of nanocarbon should be free from using excess energies for firing, sintering, melting, vaporizing and expensive equipments. We, propose here Soft processing of functionalized Graphenes at ambient conditions. The soft processing provides number of advantages which includes (a) simple reaction set up,(b) at ambient conditions, (c) simple procedure and (d)less operating costs. Herewith, we have utilized "Submerged Liquid Plasma [SLP] and "Electrocemical Exfoliation[ECE] methods. SLP methods resulted the direct synthesis of Nitrogen functionalized Graphene Nano-sheets from Graphene suspension and/or Graphite electrode in acetonitrile liquids. Products contains few layers Graphene nanosheets. Unsaturated or high energy functional group have formed in the products. We could confirm those functionalized Graphenes are electrochemically active. Using pencil rods instead of Graphite rods we have also succeeded to prepare the Nano-clay/Graphene hybrids by this SLP methods. Reduction and functionalization of GO and synthesis of Graphene/Au Hybrids also realized by SLP.

2:00 PM

(ICACC-S7-031-2017) Microwave-Assisted Synthesis of Perovskite $SrSnO_3$ Nanocrystals in Ionic Liquids for Photocatalytic Applications (Invited)

A. V. Mudring*[1]; 1. Iowa State University, USA

Nano-sized $SrSnO_3$ photocatalysts have been successfully synthesized by microwave synthesis in various ionic liquids (ILs) followed by a moderate heat treatment process. The influence ILs with various counter cations such as 1-butyl-3-methylimidazolium ($[C_4mim]^+$), 6-bis(3-methylimidazolium-1-yl)hexane, butylpyridinium and tetradecyltrihexyl phosphonium ($[P_{66614}]^+$) and bis(trifluoromethanesulfonyl)amide ($[Tf_2N]^-$) as the anion on the structure, crystallization and morphology of the products was investigated. The samples are characterized by X-ray diffraction, TG, SEM, BET surface area analysis, X-ray photoelectron spectroscopy, UV–Vis diffuse reflectance spectroscopy, and Raman and IR spectroscopy. Acording to stucture characterization using X-ray diffraction, and Raman analyses, all samples crystallized phase pure in the orthorhombic perovskite type of structure. Investigation of the sample morphology using SEM shows that, based on the IL, the obtained $SrSnO_3$ exhibits different morphologies. For these particles, the highest photocatalytic activity for H_2 evolution and for photo-hydroxylation of terephthalic acid under UV irradiation. The improved photocatalytic activity of the sample prepared in $[C_4Py][Tf_2N]$ can be attributed to the synergistic impacts of the relatively large surface area associated with the nanosized crystals and the appropriate energy band structure.

2:30 PM

(ICACC-S7-032-2017) Multifunction metal oxides shell on carbon nanotubes core for clean energy and other applications (Invited)

D. Chua*[1]; 1. National University of Singapore, Singapore

In this review, we will show that hybrid metal oxide/carbon nanotubes shell/core structures effectively enhances and extends the range of applications. Several applications will also be reviewed here. For example, metal oxide tip-coated carbon nanotubes (such as MoO3 and WO3) have proved to be excellent schottky electron emitters while platinum coated carbon nanotubes formed excellent catalytical activity for PEM fuel cell applications. Other metal-oxide composites such as ZnO can have dual-functional properties allowing good electron emission with photoluminescence properties. We can further extend the applications into biomedical materials where carbon nanotubes can be used as the seed layer for hydroxyapatite growth. We will further show that 2D structures can be incorporated on these carbon nanotubes with clean energy applications such as PEM fuel cell and hydrogen evolution reactions.

3:00 PM

(ICACC-S7-033-2017) Bottom-up processing oxide nanosheets into 3-D porous solids and their topothermal conversions

T. Hey*[1]; P. Metz[1]; T. Gubb[1]; K. Fuller[1]; S. Misture[1]; 1. Alfred University, USA

Nanosheets derived from bulk layered oxides are promising for use in applications of catalysis and energy storage. Starting from highly crystalline materials, we demonstrate that layered oxides can be chemomechanically exfoliated to produce stable suspensions of nanosheets that can be flocculated through the addition of acids and salts. Homogeneous flocculation or gelation of the nanosheets yields either macroporous solids with surface areas reaching ~200m2/g or mesoporous structures comprising primarily restacked nanosheets. Topothermal conversions are demonstrated for the first time where the 3-D, high porosity frameworks are maintained but the nanosheets convert to tunnel-structure forms that can accommodate ion sorption, etc.

New Materials and Processing II

Room: Coquina Salon A
Session Chair: Monica Jung de Andrade, University of Texas, Dallas

3:30 PM

(ICACC-S7-034-2017) Defective films for water splitting (Invited)

Y. Yang*[1]; 1. University of Central Florida, USA

Defect-engineering was developed to create sufficiently abundant active sites towards efficient water splitting. More specifically, three strategies were utilized: i) electrochemically anodic treatment was used to generate porous oxide films with high surface area and large amount of surface oxygen vacancies; ii) no further thermal treatment was used to maintain an amorphous structure of the film; iii) binary metal oxides were fabricated to introduce more defects at atomic scale. Enabled by those defective features, these porous binary metal oxide films delivered highly efficient water splitting (oxygen and hydrogen generation) capabilities in alkaline solution. A full water splitting electrolyzer developed by using these defective films were also demonstrated.

4:00 PM

(ICACC-S7-035-2017) Porous Structure based High Performance Electrocatalysts for Low Temperature Fuel Cells and CO_2 conversion (Invited)

J. Lee*[1]; 1. POSTECH, Republic of Korea

In this presentation, a simple and scalable method to produce ordered-intermetallic FePt nanotubes by electrospinning will be presented. When tested at cathode catalysts, under the US Department of Energy's reference condition, the activity of face-centered-tetragonal (fct) FePt NTs surpasses that of commercial Pt/C. In an accelerated degradation test at 1.4 V for 3h, the degradation activity rate of fct-FePt NTs is only 10%, whereas that of commercial Pt/C catalysts is 65%. This approach would provide simple route to support-free intermetallic nanotube structure with superior kinetic activity and higher durability than those of commercial Pt/C catalyst. We will also present the effect of the pore size and doping site position on the single-cell performance of metal-free catalysts using wee-defined ordered mesoporous carbon systems. Well-defined large pore sized mesoporous carbons were synthesized by simple self-assembly of block copolymers with carbon and silicate precursors. The single-cell tests prove that control of pore size and doping-site positon directly affects the cell performance by changing the mass-transport properties and utilizing the doping sites in the catalyst layer. NPOMC with precisely controlled doping sites to be near the large mesopores exhibited a remarkable on-set potential and achieved 70% of the maximum power density of Pt/C.

4:30 PM

(ICACC-S7-036-2017) Extremely flexible inorganic-organic multilayer moisture barriers grown by atomic layer deposition and plasma polymerization

S. Cho*[1]; 1. SungKyunKwan University, Republic of Korea

Inorganic-organic multilayer moisture-barrier layers were grown by atomic layer deposition (ALD) and plasma enhanced chemical vapor deposition (PECVD). The inorganic and organic layers were ALD-grown Al2O3 and plasma-polymerized layers, respectively. By structuring the inorganic-organic multilayers, the moisture barrier showed the reduced water vapor transmission rate as well as the enhanced flexibility. The flexible moisture barrier could be utilized as an encapsulation layer for moisture-sensitive flexible organic devices such as organic light emitting diodes or organic photovoltaic cells. We further developed a roll-to-roll equipment to fabricate the inorganic-organic multilayer barriers by the ALD and PECVD.

4:50 PM

(ICACC-S7-037-2017) Engineering Interfacial Structure in "Giant" PbS/CdS Quantum Dots for Solar Energy Conversion

L. Jin*[1]; H. Zhao[1]; A. Vomiero[2]; F. Rosei[1]; 1. Institut National de la Recherche Scientifique, Canada; 2. Luleå University of Technology, Sweden

Intense efforts are boosting the development of photoelectrochemical (PEC) H_2 generation to fulfill the demand for clean energy. Colloidal quantum dots (QDs) have been employed as excellent light harvesters to sensitize wide band gap semiconductors such as TiO_2. However, bare QDs or core/thin-shell QDs could suffer from stability issue due to their surface sensitivity. Post-growth of a robust thick inorganic shell (in general>1.5 nm) can inhibit surface oxidation and the formation of surface traps of core due to better surface passivation. However, the sharp interface in type I core/shell structures may contribute to unwanted confinement of charge carriers and the interfacial defects. Here, we demonstrated the possibility to finely tune the hole transfer dynamics by properly controlling the core/shell interfacial layer, leading to transition from double- to single-colour emission as a consequence. For the first time, we reported TiO_2 mesoporous framework sensitized by the close to near infrared "giant" PbS/CdS QDs with gradient interface can produce a saturated photocurrent density ~5.3 mA/cm^2 under 1 Sun illumination, which is much higher than PbS and thin-shell PbS/CdS QDs. The as-prepared PEC device presented good stability thanks to the "giant" core/shell QDs architecture with tailored interfacial layer, keeping 78% of its initial current density after 2-hour irradiation.

5:10 PM

(ICACC-S7-038-2017) Nanofiber-supported CuS nanoplatelets as a high efficiency counter electrode for quantum dot-based photoelectrochemical hydrogen generation

F. Navarro Pardo*[1]; L. Jin[1]; H. Zhao[1]; A. Vomiero[2]; F. Rosei[3]; 1. Institut National de la Recherche Scientifique, Canada; 2. Luleå University of Technology, Sweden; 3. University of Electronic Science and Technology of China, China

Photoelectrochemical (PEC) hydrogen (H_2) production represents a clean and environmentally sustainable approach to provide energy. In a typical PEC device, the counter electrode (CE) which is where H_2 is reduced, is made with Platinum (Pt). Therefore, it is highly desired to develop robust, active and inexpensive non-precious metal-based CEs for cost-effective PEC devices. In this work, we have obtained a composite structure comprising polymer nanofibers, which act as a support for covellite (CuS) nanoplatelets; its performance was studied in a PEC system based on a TiO_2 photoanode sensitized with PbS/CdS QDs optically active in the near infrared region. PEC tests showed a photocurrent density of ~7.5 mA/cm^2 at ~0.6 V versus the RHE, similar to that of Pt foil CE. Our CE mantained 85% of the initial photocurrent density after ~1 h, similarly to the response obtained under the same conditions with the Pt foil CE (86%). Stability of the nanofiber-supported CuS was confirmed by analyzing the chemical structure, morphology and catalytic performance under cyclic operation. The ease of preparation and low cost for obtaining this composite structure along with its efficiency and long-term stability offer promising features for PEC H_2 generation and a starting point for further composition/structure research of CuS CEs and related semiconductors.

5:30 PM

(ICACC-S7-039-2017) Plasmonic Ag@TiO_2 Core-Shell Nanoparticles Doping and Their Effect on The Photovoltaic and Electrochemical Performance of Dye-Sensitized Solar Cell

P. S. Fuseini Nbelayim*[1]; G. Kawamura[1]; M. M. Abdel-Galeil[2]; W. K. Tan[1]; H. Muto[1]; A. Matsuda[1]; 1. Toyohashi University of Technology, Japan; 2. Tanta University, Egypt

A key factor in improving the efficiency of the promising low-cost, easy-to-manufacture and flexible but low power conversion efficiency (PCE) dye-sensitized solar cell (DSSC) is in improving its light harvesting efficiency (LHE). Plasmonic DSSCs were prepared with plasmonic Ag@TiO_2 nanoparticles doping in anticipation of enhancing PCE via localized surface plasmon resonance (LSPR) effect and to study the effects the doping has on the performance of the cells. A 0.25% doping gave the optimal resonance coupling for efficient cell performance, increasing PCE from 3.10% to 3.45%, with only an 18.4% charge collection efficiency. The doping increased light absorbance; increased V_{OC} and reduced charge injection efficiency as a result of conduction band edge shifting; reduced surface area of the mesoporous photoanode, consequently reducing dye loading. Additionally, it enhanced interfacial charge transfer within the photoanode; increased charge transfer resistance against recombination at the TiO_2-dye/electrolyte interface by an electron-sink effect of the core Ag NPs, but at the same time increased charge transport resistance within the photoanode. The enhanced cell performance due to the LSPR effect was intense in the maximum LSPR absorbance peak region around 420 nm.

S8: 11th International Symposium on Advanced Processing and Manufacturing Technologies for Structural and Multifunctional Materials and Systems (APMT11)

Novel Ceramic Processing III

Room: Coquina Salon B
Session Chairs: Lisa Rueschhoff, Purdue University; Yiquan Wu, Alfred University

1:30 PM

(ICACC-S8-018-2017) Densification and Microstructural Engineering of Ceramics by Flash Spark Plasma Sintering (Invited)

M. J. Reece*[1]; S. Grasso[1]; T. Saunders[1]; E. Castle[1]; P. Tatarko[1]; B. Du[1]; F. Gucci[1]; 1. Queen Mary University of London, United Kingdom

This talk will review the use of Electric Current Assisted Sintering (ECAS) techniques, such as the commonly referred to Spark Plasma Sintering (SPS), for the processing of materials. This includes the potential advantages and disadvantages of the technique. SPS has been widely applied because of its ability to produce nanostructured materials. However, the techniques has other potential advantages, such as the ability to produce non-equilibrium structures. Recent results on the flash sintering (heating rates of > 10,000°C/min) of ultra-high temperature ceramics, advanced ceramic composites, hard magnetic materials and thermoelectrics will be presented. The application of ECAS to functional materials brings additional effects, including the Peltier/Thomson effects. These effects can bring advantages and disadvantages and need to be understood in order to optimise the the microstructures engineering of materials. Experimental and modelling results will presented to quantify the magnitude these effects.

2:00 PM

(ICACC-S8-019-2017) Continuous Electric Field Assisted Sintering

T. B. Holland*[1]; 1. Colorado State University, USA

Spark Plasma Sintering (SPS) of nanogranular ceramics and metals is well-known, and is considered an effective technique for producing lab scale parts. However, scale up and process efficiency suffer due to being a batch-mode and power intensive process. Further, scale up often presents significant challenges with specimen homogeneity. We have developed a process, called Continuous Electric Field Assisted Sintering, whereby the SPS "effect" can be applied through a continuous rolling apparatus. The apparatus overcomes key limitations of current SPS and EFAS approaches including: problematic scale up, excessive thermal gradients in rapid heating of large parts, resultant gradients in properties, and slow production rates even for sample areas as low as 1 m^2. Multi-physics models currently show nearly 200-fold increases in production rate along with nearly 20-fold reductions in energy expenditures. Prototypes are being evaluated to confirm these targets using zinc oxide and cerium oxide nanogranular ceramics.

2:20 PM

(ICACC-S8-020-2017) Industrial applications of Direct Current Based Spark Plasma/Field Assisted Sintering; Large components and simultaneous multi-part operation

L. S. Walker*[1]; 1. Thermal Technology, USA

Direct current sintering also known as field assisted and spark plasma sintering has achieved great success in academia and industrial based research with its ability to rapidly produce high quality material systems. Industrial acceptance of the technology however has been slow to evolve due to limited access to large industrial scale systems for proof of concept work and experienced operators. Here we present the production of large parts (>10cm) for industrial applications and high throughput operation with multiple parts simultaneously, while achieving high part uniformity and part-to-part consistency. Applications involving sintering and diffusion bonding will be demonstrated, and material systems include both low and high temperature operation of both metals and ceramic systems of interest for industrial applications.

2:40 PM

(ICACC-S8-021-2017) Flash sintering of alumina

M. Biesuz*[1]; V. M. Sglavo[1]; 1. University of Trento, Italy

Flash Sintering (FS) represents an innovative and promising sintering route that could allow a consistent reduction of processing times and temperatures and, consequently, of cost. FS belongs to the so-called field-assisted sintering techniques, characterized by the application of an electrical field during the heating process. At an onset field and temperature a strong conductivity non linearity is manifested and the densification process occurs in few seconds. This phenomenon is also followed by a strong photoemission, which has not been completely clarified yet. Many works in the last six years have shown the applicability of this technique to different classes of ceramics (i.e. ionic, electronic and protonic conductors, semiconductors, composites…). More recent activities have demonstrated that also resistive ceramics like nearly pure α-alumina can be flash sintered although the physical mechanisms are still not definitively understood. In the present work the FS behavior of corundum was analyzed in order to understand the effect of the main process parameters (field, current, electrode material) on densification. Flash sintering is applied to pre-sintered samples with the aim to clarify the effect of the starting microstructure on the runaway for the flash effect, also allowing to distinguish surface and bulk-activated phenomena. Finally, the photoemission during the different stages of flash sintering is studied in the UV/VIS and NIR regions.

3:20 PM

(ICACC-S8-022-2017) Microwave Technology for Commercial Scale Processing of Materials

P. Apte*[1]; 1. Harper International, USA

It is generally accepted that microwave energy can provide volumetric heating and could therefore be used for energy efficient, rapid thermochemical processing. It can be especially attractive for ceramics in bulk because of their low thermal conductivity. However there are very few commercial ceramic material processing facilities based on microwave heating. One reason could be that there is no generic microwave equipment design other than larger versions of the household microwave oven. However, proper design of equipment, taking into account the material properties, their form factor, the various changes in properties during thermal processing, can enable advantageous utilization of microwave energy. Proper equipment design can help regulate the energy input and ensure that it is delivered in the appropriate locations. This will be illustrated using examples of equipment and process design for the following types of heating applications. 1. Materials which are difficult to heat from room temperature 2. Materials that have a 'doughy' intermediate stage 3. Materials where high surface area products are desired 4. Rapid binder removal

3:40 PM

(ICACC-S8-023-2017) Ultra-high temperature heat treatment of ceramics by microwave: the reactive silicon infiltration process

A. Ortona*[1]; 1. SUPSI, Switzerland

Reactive silicon infiltration (RSI) is a manufacturing process to produce Si-SiC composites by infiltrating at high temperatures porous preforms containing carbon with liquid silicon. Even if RSI is considered one of the fastest manufacturing techniques for the production of CMCs, long processing times are required to heat

and cool down the furnaces commonly heated by graphitic resistors. In this work, we show the benefits of applying microwave power to perform RSI. A new set up in which preforms are embedded in powder field modifiers is here presented. An innovative use of SiC/BN powder as field modifiers with the twofold function of being an efficient way to heat up the components uniformly and of being impermeable to molten silicon is also illustrated. Si-SiC bulk, composite and porous ceramic parts of relatively large size were successfully infiltrated in minutes. Different examples will be here presented. On the other hand microwave heating is very difficult to control properly. In order to get more insight into this promising heating technique, we performed a parametric study to evaluate the effect of the process parameters. Heating and infiltration performances were respectively monitored during the heat treatment and measured on the final samples. A discussion on the influence of those parameters on the final product's characteristics is then performed.

4:00 PM

(ICACC-S8-024-2017) Direct Joule heated (Flash) Sintering of Ionic Conductive Ceramics in a Conventional Spark Plasma Sintering Furnace Using Standard Graphite Tooling

L. S. Walker*[1]; 1. Thermal Technology, USA

Direct joule heating of ionic conductive ceramics, also known as 'flash' sintering, has attracted great interest of recent due to the extremely high sintering rates achieved through the rapid heating process. The basic approach involves heating a powder compact of an ionic conductive ceramic such as zirconia in a furnace to a temperature high enough for it to conduct electrical current and rapid joule heating to high temperature using a current controlled power supply to achieve rapid sintering. The apparatus for such an experiment is generally complex and typically involves a furnace, a dog-bone shaped powder compact, power supply and noble metal conductor leads. Control over the experiment is also generally limited to current control of the power supply. Presented here is a simplistic approach to achieve the 'flash' sintering effect in a conventional spark plasma sintering (SPS) furnace using either current control or temperature control. The method requires no pre-sintering or compaction of the powder and uses only conventional tooling typical to the SPS process without any sacrificial heaters.

4:20 PM

(ICACC-S8-025-2017) Preparation of Al_2O_3-$Y_3Al_5O_{12}$-ZrO_2 eutectic ceramic by flash sintering

J. Liu*[1]; D. Liu[2]; Y. Gao[1]; Y. Wang[2]; L. An[3]; 1. Southwest Jiaotong University, China; 2. Northwestern Polytechnical University, China; 3. University of Central Florida, USA

Al_2O_3-$Y_3Al_5O_{12}$-ZrO_2 ternary eutectic oxide ceramic was prepared by flash sintering the corresponding powder. The highly dense sample was obtained at relatively low temperature and very short time with the assist of dc electric field. As-prepared ceramics well preserve the phase composition and microstructure of the original eutectic powder. The ceramic exhibits relatively high hardness and high fracture toughness. These results clearly demonstrate the feasibility of employing the flash-sintering technique to fabricate eutectic ceramics.

S10: Virtual Materials (Computational) Design and Ceramic Genome

Modeling Environmental and Thermal Behavior

Room: Ponce DeLeon

Session Chairs: Hans Seifert, Karlsruhe Institute of Technology; Jingyang Wang, Shenyang National Laboratory for Materials Science, Institute of Metal Research

1:30 PM

(ICACC-S10-031-2017) Thermodynamic modeling of the Y-Si-C-H-O system for gas turbine applications (Invited)

H. J. Seifert*[1]; I. Markel[1]; D. M. Cupid[1]; 1. Karlsruhe Institute of Technology, Germany

For next generation power plants and gas turbines, Si-based Ceramic Matrix Composites (CMCs) are promising structural materials for the hot sections. The presence of water vapor in combustion gases requires applications of environmental barrier coatings (EBC). Combinations of yttrium silicates and yttrium oxide or silicon dioxide are most promising EBC materials and their behavior at high-temperature and in O_2/H_2O containing combustion atmospheres has to be understood. In this work, CALPHAD-type modeling (CALculation of PHAse Diagrams) in combination with key experiments was used to develop a thermodynamic dataset for the multi-component Y-Si-C-O-H system. Heterogeneous reactions between EBC and various gas atmospheres were calculated. Existing thermodynamic modeling of the Y-Si-C-O system was refined by updating the description of the Y_2O_3-SiO_2 pseudo-binary system. New descriptions of the Gibbs free energies of the silicon- and yttrium-hydroxides were developed. The updated thermodynamic description of the Y-Si-C-O-H system was used to calculate the thermochemical reactions between yttrium silicate based coatings and the SiC base material as well as with the O_2/H_2O containing combustion atmosphere. The stabilities of yttrium silicate based coatings against erosion through formation of volatile silicon- and yttrium hydroxides was thereby evaluated.

2:00 PM

(ICACC-S10-032-2017) Toward an Integrated Model for Molten Silicate Degradation of Thermal and Environmental Barrier Coatings: Phase Equilibria and Reaction Dynamics

D. L. Poerschke*[1]; C. G. Levi[1]; 1. University of California Santa Barbara, USA

Mitigating the degradation of thermal and environmental barrier coatings (TBCs and EBCs) caused by molten silicate (CMAS) deposits is viewed as a fundamental challenge to increasing gas turbine efficiency. It is particularly important to understand the thermochemical interaction between deposit and coating material in a thermal gradient. These reactions can either exacerbate the degradation by destabilizing the coating microstructure or mitigate the attack by crystallizing the melt, preventing further infiltration into the coating. Efforts to model the degradation process, necessary to accelerate the development of improved materials, are complicated by variations in the deposit compositions arising in service. The present work addresses the need to develop models that accurately capture the effects of variable deposit composition on the interaction with rare-earth based coatings. Phase equilibria data from the subsystems is combined with multi-component thermodynamic modeling of the reactions, guiding targeted experiments aimed at elucidating the factors influencing the reaction dynamics. The findings provide insight into the relative efficacy of degradation-mitigating reactions for prospective coating materials.

2:20 PM

(ICACC-S10-033-2017) Kinetic Monte Carlo Simulations of Diffusion in Environmental Barrier Coating Materials

B. S. Good*[1]; 1. NASA Glenn Research Center, USA

Ceramic Matrix Composite (CMC) components for use in turbine engines offer a number of advantages compared with current practice. However, such components are subject to degradation through a variety of mechanisms. In the hot environment inside a turbine in operation a considerable amount of water vapor is present, and this can lead to corrosion and recession. Environmental Barrier Coating (EBC) systems that limit the amount of oxygen and water reaching the component are required to reduce this degradation. A number of silicate-based materials are under consideration for use in such coating systems, including Yttterbium and Yttrium di- and monosilicates. In this work, we present results of kinetic Monte Carlo computer simulations of oxygen diffusion in Yttrium disilicate, and compare with previous work on Yttterbium disilicate. Coatings may also exhibit cracking, and the cracks can provide a direct path for oxygen to reach the component. There is typically a bond coat between the coating and component surface, but the bond coat material is generally chosen for properties other than low oxygen diffusivity. Nevertheless, the degree to which the bond coat can inhibit oxygen diffusion is of interest, as it may form the final defense against oxygen impingement on the component. We have therefore performed similar simulations of oxygen diffusion through $HfSiO_4$, a proposed bond coat material.

2:40 PM

(ICACC-S10-034-2017) Microstructural modeling of EB-PVD YSZ thermal barrier coating under thermomechanical and CMAS loading

A. Laukkanen*[1]; T. Andersson[1]; T. Suhonen[1]; 1. VTT Technical Research Centre of Finland, Finland

Thermal barrier coatings (TBCs) are typically utilized in the gas turbine hot section for protection of critical components to increase service life and improve efficiency. Design of TBC systems is a complex challenge largely carried out by trial-and-error due to the complexity of both the material solution and the service conditions. In current work, 3D microstructural modeling founded approach is developed to tackle the problem. The objective is to contribute to computational materials engineering solutions for TBCs that can be utilized in design of novel coating solutions and assess the behavior of the component during its operation. As a case study a partially-stabilized zirconia (YSZ) coating on a NiAl substrate is investigated. Microstructural model of the system is created in full 3D and loaded with operational thermal loading. The system response is discussed and evaluated with respect to microstructural characteristics, properties and modes of failure. Tomography imaging is utilized in development of the computational model. As an additional failure mode of importance the adhesion of sand particles on hot TBC surfaces is considered to evaluate the capabilities of the modeling approach. Melted calcium-magnesium-alumina-silicate (CMAS) glass deposits are introduced penetrating the top TBC layer and the following adhesive TBC failure mode is investigated.

3:20 PM

(ICACC-S10-035-2017) A modeling framework, accounting for competing degradation/healing mechanisms, for the prediction of the long term mechanical behavior of CMCs (Invited)

E. Baranger*[1]; 1. LMT, ENS-Cachan, CNRS, Université Paris-Saclay, France

SiC/SiC ceramic matrix composites have good specific mechanical properties for high temperatures in oxidizing atmosphere. However, very high lifetimes now affordable with self-healing matrices lead to expansive experimental validation strategies; predictive models are therefor needed. The confidence accorded to such models is directly related to the understanding and modeling of the different degradation and healing mechanisms. A modeling framework and the associated simulation tools are presented. It allows the prediction of the mechanical behavior and lifetime for complex thermo-chemo-mechanical loadings. Based on that framework, a macroscopic model enriched with micro information has been developed, identified and validated. This model can also be used to better understand the behavior of the composite material. Two examples are given. The first deals with the impact of the different healing layers on the final time to fracture. The second deals with the prediction of the macroscopic rupture probability while introducing probabilistic data only at a micro-scale. From that enriched macroscopic model, the focus is given to the description of the mechanical behavior of CMC yarns using a multiscale generalized finite element method MS-GFEM for which the microstructure is generated from different mechanically representative patterns.

3:50 PM

(ICACC-S10-036-2017) Ablation of a 3D C/C Composite in a Plasma: Influence of the Surrounding flow on the Kinetics and Morphology (Invited)

G. L. Vignoles*[1]; C. Levet[1]; J. Mathiaud[2]; J. Couzi[2]; B. Helber[3]; O. Chazot[3]; J. Gouriet[3]; 1. University Bordeaux, France; 2. CEA, France; 3. Von Karman Institute for Fluid Dynamics, Belgium

3D Carbon-fiber reinforced carbon composites (3D C_f/C) are widely used in thermal protection systems for atmospheric re-entry. They encounter very aggressive environments, and suffer from ablation. The Plasmatron Inductively Coupled Plasma torch of the Von Karman Institute for Fluid Dynamics offers representative conditions in ground tests, with numerous intrusive and non-intrusive diagnostics and measurements, coupled with a numerical rebuilding of the flow. Air and argon flows were used on flat and hemispherical samples ; surface temperatures ranged from 1800 to 2500°C. Post-mortem examinations were carried out by SEM micrography and digital microscope profilometry. The differences in ablation resistance between constituents and the flow field have a major contribution to the composite macroscopic and mesoscopic roughness and recession velocity. Results of a simple analytical model and of 3D numerical computations support these conclusions.

4:20 PM

(ICACC-S10-037-2017) Tailoring Phonon Anharmonicity and Its Impacts on Tunable Thermal Properties of Yttrium Silicates

Y. Luo*[1]; J. Wang[1]; 1. Institute of Metal Research, Chinese Academy of Sciences, China

Phonon engineering is a necessary yet challenging diagram to guide the future designs of novel structures and materials with desired thermal properties. In this study, phonon behaviors of yttrium silicates ($Y_2Si_2O_7$ and Y_2SiO_5) are investigated via density function theory and lattice dynamic calculations. Giant phonon anharmonicity is identified due to significant coupling between acoustic and low-lying optic phonons; and its correlation with heterogeneous atomic bonding environments is also established. Based on relaxation time approximation and Debye theory, an anomalous negative pressure dependence of intrinsic lattice thermal conductivity is predicted due to pressure-induced enhancement of phonon anharmonicity. Meanwhile, by employing quasi-harmonic approximation, we show that the thermal expansion coefficient of mono- and di-yttrium silicates have times of difference, in spite of their identical composition and similar polyhedral structural units. The discrepancy mainly stems from the nature of phonon anharmonicity, governed by diverging low-energy atomic vibration pattern of corner-sharing (in $Y_2Si_2O_7$) and isolated (in Y_2SiO_5) SiO_4 tetrahedra under perturbation of intrinsic lattice. Our investigation is expected to bring deeper physical insights for tailoring thermal properties in complex-structured oxide ceramics from the cutting-in point of phonon anharmonicity.

4:40 PM

(ICACC-S10-038-2017) Pressure induced low-lying phonon modes softening and thermal resistance strengthening in β-$Mg_2Al_4Si_5O_{18}$

Y. Li*[1]; J. Wang[1]; 1. Institute of Metal Research, China

In the whole thermal conductivity spectrum, materials at the low end are of great interest for a wide range of thermal insulation applications such as thermal insulators, thermoelectric materials and thermal barrier coatings. In principle, materials with complex structures are inherently strong in anharmonicity and weak in phonon dispersion because a vast majority of heat is trapped in flat, low velocity optical modes which are introduced with increasing structural complexity. We applied hydrostatic pressure to investigate the temperature dependent lattice thermal conductivities of β-$Mg_2Al_4Si_5O_{18}$ with a complex framework structure using first-principles calculations based on density functional theory and ab initio lattice dynamics. A rigorous analysis of phonon dispersions helps us to recognize the origin of the negative dependence of thermal conductivity on pressure and attribute this anomalous behavior to the low frequency softening phonons and strengthening anharmonicity, both of which arise from the unique corner-linked tetrahedral framework in crystal structure. This work not only reports an anomalous pressure induced reduction of lattice thermal conductivity, but also provides a key insight into the interesting thermal conductivity modification mechanism through tailoring the crystal structure of complex compounds.

S12: Materials for Extreme Environments: Ultrahigh Temperature Ceramics (UHTCs) and Nano-laminated Ternary Carbides and Nitrides (MAX Phases)

Processing-microstructure-property Relationships of Existing Systems II

Room: Tomoka B

Session Chairs: Frederic Monteverde, CNR-ISTEC; Sylvain Dubois, PPRIME Institute

1:30 PM

(ICACC-S12-037-2017) Synthesis, Characterization, and Sintering of Nanocrystalline Tantalum-Hafnium Carbide Solid Solution Powders

P. Foroughi[1]; A. Behrens[1]; Z. Cheng*[1]; 1. Florida International University, USA

Tantalum carbide and hafnium carbide are considered as ultrahigh temperature ceramics (UHTCs). These carbides form a continuous solid solution and offer the flexibility to optimize their chemical and physical properties for critical applications in aerospace and other fields. However, synthesis of single phase (Ta,Hf)C solid solution powders via conventional carbothermal reduction (CTR) reaction proved difficult due to the difference in reactivity and the presence of a miscibility gap below 887°C causing phase separation i.e., formation of two distinctive carbides instead of a single phase solid solution. On the other hand, the extremely high melting points of these carbides make the sintering process difficult. In this study, nanocrystalline (Ta,Hf)C powders were synthesized by solution processing of tantalum chloride, hafnium chloride, and sucrose followed by pyrolysis and then CTR of the yielded finely mixed tantalum-hafnium oxide(s) and carbon. Special attention was given to investigate the effects of different processing parameters on inhibiting/promoting the phase separation of (Ta,Hf)C during synthesis. In addition, results on characterizing the synthesized nano solid solution powders including their oxidation resistance and evaluating their sinterability using spark plasma sintering (SPS) and new flash sintering techniques will be presented and discussed.

1:50 PM

(ICACC-S12-038-2017) Thermal expansion of MAX phases solid solutions

T. Cabioch*[1]; P. Chartier[1]; T. Basyuk[2]; J. Halim[3]; E. Caspi[4]; M. W. Barsoum[3]; 1. University of Poitiers, France; 2. Bakul Institute for Superhard Materials, Ukraine; 3. Drexel University, USA; 4. Nuclear Research Centre, Israel

The structures and the properties of ternary compounds such as MAX phases can be modified, and even tuned, by introducing a fourth, or more, element into their structures. It is now well established that isostructural solid solutions can be obtained on the M, A and/or X sites and this compositional degree of freedom has been exploited by some to tailor the MAX phases' properties. For example the oxidation resistance of Ti_3SiC_2 improves upon the introduction of Al on the A-sites to create $Ti_3(Si_{1-x},Al_x)C_2$ solid solutionsy. Solid solution hardening effects were also reported for $(V_x,Ti_{1-x})_2AlC$. This contribution focuses on the evolution of thermal expansion coefficients (TEC) of $M_{n+1}AX_n$ compounds when solid solutions are obtained on the M, A or X sites for various values of n. $Cr_2(Al_x,Ge_{1-x})C$, $(Cr_x,V_{1-x})_{n+1}AlC_n$ with n =1, 2, 3 and $Ti_2Al(C_x,N_{1-x})$ were synthesized by conventional powder metallurgy techniques with x=0; 0.25; 0.5; 0.75; 1 and studied in situ in a X-ray diffractometer between 20°C and 800°C to determine their TEC along the a- and c-axis. This study shows that these TEC generally obey a Vegard's law as a function of x. Interestingly, whereas an almost isotropic behavior is obtained for x=1 and 3 in the system $(Cr_x,V_{1-x})_{n+1}AlC_n$, the compounds obtained for n= 2 show anisotropic behavior. The TEC of new quaternary MAX phases compouds (Mo_2TiAlC_2 and $Mo_2Ti_2AlC_3$) will also be discussed.

2:10 PM

(ICACC-S12-039-2017) Advances and challenges in $Zr_{n+1}AlC_n$ MAX phases for fission reactors

E. Zapata-Solvas*[1]; N. Ni[2]; D. Horlait[3]; S. Christopoulos[4]; A. Thibaud[1]; D. Parfitt[2]; A. Chroneos[4]; W. Lee[1]; 1. Imperial College London, United Kingdom; 2. Imperial College London, United Kingdom; 3. CNRS/IN2P3 and University of Bordeaux, France; 4. Coventry University, 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 studies the stabilization of $Zr_{n+1}AlC_n$ MAX phases by partial substitutions in the quaternary systems $(Zr,M)_{n+1}AlC_n$ and $Zr_{n+1}(Al, A)C_n$ with M=Cr and A=Si. Synthesis and sintering of MAX phases will be discussed as well as DFT calculations, indicating possible phase stability and different elements that play a role in the nucleation and stabilization of $Zr_{n+1}AlC_n$, will be shown.

Materials Design, New Composition and Composites V

Room: Tomoka B

Session Chairs: Frederic Monteverde, CNR-ISTEC; Sylvain Dubois, PPRIME Institute

2:30 PM

(ICACC-S12-040-2017) YB_2C_2: A potential layered ultra-high temperature ceramic with excellent damage tolerance

G. Zhao*[1]; J. Chen[1]; Y. Li[1]; M. Li[1]; 1. Institute of Metal Research, China

Intrinsic brittleness, poor thermal shock resistance and machinability have greatly hampered the applications of ultra-high-temperature ceramics. In this work, a noval ternary rare earth metal diborodicarbides YB_2C_2 was successfully fabricated by an in situ hot-pressing method. The electronic structure and the mechanical properties of YB_2C_2 were investigated using a combination of DFT calculations and experimental investigations. The chemical bonding in YB_2C_2 is anisotropic according to first-principles calculations, which results in anisotropic elastic properties and low shear deformation resistance. The experimental results show that the as-received YB_2C_2 ceramic possesses typical layered structure, low Young's modulus (207 GPa), low shear modulus (87 GPa), low hardness (4.2±0.3 GPa), moderate flexural strength (460±10 MPa), and high fracture toughness (4.6±0.1 MPa $m^{1/2}$). In addition, it was also proved that YB_2C_2 has good thermal shock resistance, excellent damage tolerance and easy machinability. What's more, it has excellent thermal stability under ultra-high temperature. These unique features might endow YB_2C_2 with great potential for future ultrahigh temperature applications.

2:50 PM

(ICACC-S12-041-2017) Morphology and Phase Control in Synthesis of Nanocrystalline Ultrahigh Temperature Ternary Diboride Powders

P. Foroughi*[1]; Z. Cheng[1]; 1. Florida International University, USA

Transition metal diborides such as hafnium diboride (HfB_2) and tantalum diboride (TaB_2) are considered as ultrahigh temperature ceramics (UHTCs) thanks to their extremely high melting points (~>3000°C). Some of them were reported to form solid solutions offering additional flexibility in tuning their composition and optimizing the resulting chemical/physical properties. In this study, nanocrystalline powders of ternary diboride nanocomposite and related solid solution in the system of Ta-Hf-B were synthesized by aqueous solution processing of tantalum chloride, hafnium tetrachloride, boron trioxide and sucrose followed by pyrolysis and carbothermal reduction reaction (CTR) of the yielded molecular scale mixed tantalum-hafnium-boron oxide(s) and carbon. Similar to other boron-containing ceramics such as boron carbide, noticeable morphology and particle size non-uniformity were observed during the synthesis of the ternary diboride powders via CTR. The results of study on the effects of starting materials/solvent selection and thermal processing conditions (e.g., CTR temperature, time and heating/cooling rate) on product morphology, particle size, and phase separation will be presented and the factors determining product uniformity in morphology and phase (i.e., when they consists of two phase nanocomposite versus single phase solid solution) will be discussed.

New Precusors for Powders, Coatings, and Matrix or Fibers of Composites

Room: Tomoka B
Session Chair: Surojit Gupta, University of North Dakota

3:30 PM

(ICACC-S12-042-2017) Low-temperature synthesis of hafnium diboride powder via magnesiothermic reduction in molten salt

K. Bao*[1]; S. Zhang[1]; 1. University of Exeter, United Kingdom

Phase pure hafnium diboride (HfB_2) powder was synthesized from HfO_2 and B_2O_3 using a molten-salt assisted magnesiothermic reduction technique. The effects of salt type, Mg and B_2O_3 amount, and reaction temperature on the synthesis process were examined and the relevant reaction mechanisms discussed. Among the three chloride salts (NaCl, KCl and $MgCl_2$) attempted, $MgCl_2$ showed the best accelerating effect. By using 60 mol% excess Mg and 60 mol% excess B_2O_3, phase pure HfB_2 powder of ~100 nm was obtained after 6 h firing at 950°C. Total yield of HfB_2 was ~92%.

S13: Advanced Materials for Sustainable Nuclear Fission and Fusion Energy

Advanced Reactor Materials and Chemical Compatibility

Room: Coquina Salon H
Session Chairs: Kurt Terrani, Oak Ridge National Lab;
Tatsuya Hinoki, Kyoto University

1:30 PM

(ICACC-S13-010-2017) Materials' Innovations for a Safe and Sustainable nuclear in Europe (FP7 MatISSE project) (Invited)

P. Giroux*[1]; A. Michaux[1]; L. Malerba[2]; A. Bohnstedt[3]; C. Mingazzini[4]; M. Serrano[5]; K. Nilsson[6]; P. Ho-Hune[7]; 1. CEA, France; 2. SCK-CEN, Belgium; 3. KIT, Germany; 4. ENEA, Italy; 5. CIEMAT, Spain; 6. JRC, European Commission, Belgium; 7. LGI, France

Advanced fission systems seek for enhanced sustainability and economics while keeping high safety standards. The development of those advanced reactors mainly depends on innovation in the field of materials for fuel elements and structural components, regarding their performances in operating conditions (high temperature, mechanical loading, irradiation, corrosive environments). In addition, the development of robust and predictive evaluation methods in support of the materials qualification program is an essential task within this framework. The MatISSE project (Materials' Innovations for a Safe and Sustainable nuclear in Europe), that supports the European Energy Research Alliance - Joint Programme of Nuclear Materials, aims at building a European integrated research programme on materials innovation for a safe and sustainable nuclear. The objective of this work is to optimize performances of conventional and advanced nuclear materials, including capability to forecast their behavior in operation. An overview of the results obtained since the beginning of the project in 2013 is presented, focused on materials qualification and development across low carbon energy technologies as well as technical results on (i) modelling in ferritic/martensitic steels and alloys, (ii) ceramic composites and MAX phase cermets, (iii) ODS steels and (iv) activities performed in support to ESNII reactors.

1:50 PM

(ICACC-S13-011-2017) Oxidation behaviour study of SiC/SiC composite and aluminium-based ceramics in the high temperature helium environment

J. Kalivodova*[1]; C. Sauder[3]; J. Kutzendörfer[2]; J. Berka[1]; M. Kryková[1]; 1. Centrum výzkumu Rez s.r.o., Czech Republic; 2. UCT, Czech Republic; 3. CEA, France

Generation IV gas cooled nuclear reactors are considering the use of ceramics and ceramic composites where radiation doses, environmental challenges, or temperatures (up to or beyond 1000°C) will exceed capabilities of metallic materials. This is especially true for control rods, reactor internals, refractory materials, thermal insulation materials, and for gas cooled fast reactor fuel cladding. SiCf/SiC composite has become a prime material candidate for advanced fuel cladding and it posses outstanding mechanical, thermal and electrical properties. Alumina or aluminium oxide (Al2O3) advanced ceramic materials are promising refractory material, mainly due to its high melting point. Nevertheless, it is important to understand oxidation behaviour of those ceramic materials under specific conditions simulating Gas Fast Reactor (GFR) coolant conditions. In this study, the oxidation resistance of ceramic materials under high temperature helium environment has been investigated. Subsequently the microstructural characterisation of ceramic samples was performed.

2:10 PM

(ICACC-S13-012-2017) Hydrothermal corrosion behavior of SiC fibers for CVI-SiC/SiC composites

S. Suyama*[1]; M. Ukai[1]; M. Uchihashi[1]; K. Kakiuchi[1]; H. Heki[1]; 1. Toshiba Corporation, Japan

Recently, CVI-SiC/SiC composites have been shown to be promising structural materials for use in nuclear reactor cores to enhance safety. We have started to design the structure and composition of CVI-SiC/SiC composites for light water reactor cores. In this work, we evaluated the candidate for the SiC fiber of CVI-SiC/SiC composites necessary to achieve high hydrothermal resistance. The candidate fiber of a CVI-SiC/SiC composite was selected by considering the residual strength characteristics and surface conditions. To protect the SiC fiber from hydrothermal corrosion, both a dense interface coating on every fiber and a dense coating on the entire surface of the CVI-SiC/SiC composite were found to be necessary for the core materials.

2:30 PM

(ICACC-S13-013-2017) Corrosion Behavior of Various CVD SiC Ceramics in Hydrothermal Water

W. Kim*[1]; J. Shin[1]; D. Kim[1]; H. Lee[1]; J. Park[1]; 1. Korea Atomic Energy Research Institute, Republic of Korea

SiC ceramics and composites have excellent high temperature mechanical properties, chemical stability, irradiation tolerance, and low radioactivity under neutron irradiation. Because of these superior physical/chemical properties, they have been considered for use as reactor core materials of fusion and advanced fission reactors. In recent years, there have also been efforts on applying the SiC$_f$/SiC composites to nuclear fuel claddings and fuel components of light water reactors (LWRs). Although there are a lot of technical hurdles, the hydrothermal corrosion of SiC is one of the most critical feasibility issues for the LWR application. It has been reported that the corrosion behavior of SiC in hydrothermal water is largely dependent on the fabrication methods of SiC ceramics as well as the detailed water chemistry. While the highly pure CVD SiC ceramics have been known to have a better corrosion resistance than the reaction bonded or sintered SiC ceramics, it has not been elucidated how the microstructural features affect the corrosion of different grades of CVD SiC ceramics. In this study, we compared the corrosion behavior of various grades of CVD SiCs procured from different vendors. We analysed various microstructural features and discussed the effect of microstructures on the hydrothermal corrosion of CVD SiC ceramics.

3:10 PM

(ICACC-S13-014-2017) Modular Fabrication and Characterization of Complex Silicon Carbide Composite Structures

H. Khalifa*[1]; C. P. Shih[1]; E. Song[1]; E. Novitskaya[2]; O. Graeve[2]; L. Alva[3]; H. Zhao[3]; X. Huang[3]; C. Deck[1]; 1. General Atomics, USA; 2. University of California, San Diego, USA; 3. University of South Carolina, USA

Fabrication of large, complex silicon carbide fiber-reinforced, silicon carbide matrix composites (SiC-SiC) is an enabling technology for advanced reactors. Recent efforts targeting fabrication of complex SiC-SiC composites for in-core support structures will be discussed. Bulk processing and composite joining techniques have been developed to advance the state of complex SiC-SiC structure fabrication. Mechanical strength and fracture toughness were measured for SiC-SiC made via transient eutectic phase (TEP) processing and chemical vapor infiltration. Near-net shape complex structures were fabricated containing thru-hole features. The effect of thru-hole spacing was assessed via heat transfer modelling, microhardness and open-hole tension tests coupled with digital image correlation. Enhanced complexity was achieved by bonding SiC-SiC plates together using TEP-based joining. Joint strength and microstructure were characterized by double notch shear tests and transmission electron microscopy, respectively. Bulk performance of joined complex composite structures was assessed via instrumented impact tests coupled with impulse excitation. Residual strength of complex structures was measured after impact testing. This work successfully demonstrated fabrication of robust SiC-SiC composite structures with engineered internal features. This work is supported by DOE-NE grant # DE-NE0008323.

3:30 PM

(ICACC-S13-015-2017) Conception, manufacturing and characterization of SiC/SiC claddings for IV Generation Nuclear Reactors

P. G. David*[1]; J. Blein[1]; Y. Pierre[1]; O. Caty[2]; Z. Mane[2]; 1. CEA, France; 2. LCTS, University of Bordeaux, France

Several developments and optimizations have been studied to meet the specifications of structural components in nuclear systems. For Gas cooled Fast Reactor claddings, new textile processes have been developed for manufacturing of honeycomb structures or closed tubular braids, used as fibrous reinforcement structures. Closed tubular braids have shown pressure resistance up to 1000 bars. Control of the geometry can be improved using graphite tools, during densification, and by addition of a sacrificial layer that is machined away after densification. Precision on the diameters was better than 1 % and the general and local straightness on tubes was about 0.05 mm. The internal surface smoothness has been increased through an initial Chemical Vapor Deposition step on the graphite rod used as a mandrel for pin braiding. The roughness (Ra) was lower than 5 microns. Complementary tests and characterizations are still necessary to prove that these materials are consistent with the targeted performance.

3:50 PM

(ICACC-S13-016-2017) Microstructural Evolution in Neutron Irradiated Fine-Grained Graphite

A. A. Campbell*[1]; J. Arregui-Mena[1]; Y. Katoh[1]; T. Takagi[2]; H. Kato[2]; 1. Oak Ridge National Lab, USA; 2. Ibiden Co., Ltd., Japan

Graphite undergoes complex changes to all of its physical, thermal, and mechanical properties when it is exposed to neutron irradiation. These changes are strongly dependent on the irradiation temperature and pre-irradiation properties. What is still not extensively understood is the dependence of these physical changes on the microstructure evolution as a result of exposure to irradiation. Research at Oak Ridge National Laboratory has been investigating the microstructure evolution of neutron irradiated graphite. Specimens of ETU-10 graphite have been irradiated in the High Flux Isotope Reactor at various combinations of irradiation temperature and total neutron fluence. The changes to the physical properties have been measured at each temperature/fluence combination, and a sub-set of these specimens are being investigated to understand how the microstructural features evolve. The microstructure feature that will be discussed in this presentation will be the changes to the pore structure. The meso- to macroscopic pores have been measured with optical microscopy and micro-tomography. The results of the pore structure evolution and how these relate to the observed physical property changes will be presented and discussed.

4:10 PM

(ICACC-S13-017-2017) Fracture and indentation behavior of Ti_3AlC_2-$Ti_5Al_2C_3$ and Ti_3SiC_2 after neutron-irradiation at elevated temperatures

C. Ang*[1]; P. Chad[1]; C. P. Shih[3]; P. Edmondson[1]; S. Zinkle[2]; Y. Katoh[1]; 1. Oak Ridge National Lab, USA; 2. University of Tennessee, USA; 3. General Atomics, USA

MAX phases are potential materials for advanced reactors. However, effects of neutron irradiation on mechanical properties in MAX phases and the underlying physical processes are largely unknown.

In the present work, Ti_3AlC_2-$Ti_5Al_2C_3$ and Ti_3SiC_2 were neutron irradiated to a dose of ~2 dpa at temperatures (T_{irr}) of ~400 and ~700°C, followed by indentation, equibiaxial flexural tests, and microstructural examination. There was a transition in fracture behavior between T_{irr} ~400°C and ~700°C, which was further confirmed by fractography and indentation response. After T_{irr} ~400°C, indentation response was poor, and fracture surfaces were flat facets. After T_{irr} ~700°C, fracture surfaces had bridging ligaments from destabilized kink-bands and classical indentation morphology with measurable indentation cracks. After T_{irr} ~700°C there was a reduction in fracture toughness, measured by introducing an increasing "critical flaw" size and observing the change in equibiaxial fracture strength. T_{irr} ~400°C affected the A-layer and M-A functionality. A STEM examination suggested that the abrupt change in fracture behavior was associated with retained M-M (antisite) and recovered M-A layers after T_{irr} ~600°C. Research sponsored by the Office of Fusion Energy Sciences, U.S. Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

S14: Crystalline Materials for Electrical, Optical and Medical Applications

Optical Material 4

Room: Tomoka C

Session Chairs: Joanna McKittrick, UC San Diego; Inka Manek-Hönninger, CELIA - University of Bordeaux

1:30 PM

(ICACC-S14-031-2017) Are time reversal and inversion symmetries broken in copper oxide high temperature superconductor $Bi_2Sr_2CaCu_2O_{8+x}$? (Invited)

T. Asahi*[1]; 1. Waseda University, Japan

Despite intensive experimental and theoretical studies on copper-oxide based superconducting material, the mechanism of high-temperature superconductivity is still not clear. Clarifying whether time reversal symmetry and inversion symmetry in the superconducting material is broken or not is indispensable for understanding the mechanism of the high temperature superconductivity. Although several groups have reported that time reversal symmetry is broken in the copper-oxide superconductor pseudogap region, these results are controversial. In this study, the main purpose is to investigate accurately the optical property and symmetry of $Bi_2Sr_2CaCu_2O_{8+x}$ (Bi2212) crystal with Generalized–High Accuracy Universal Polarimeter (G-HAUP). The G-HAUP enables us to measure the natural optical activity (OA) and natural circular dichroism (CD) caused by the inversion symmetry breaking and the Faraday effect and magnetic CD caused by the time reversal symmetry breaking in condensed matters. Furthermore, we can measure the strong optical quantities such as linear birefringence (LB) and linear dichroism (LD) with the G-HAUP in addition to OA and CD. We have succeeded in preparing very thin Bi2212 single crystals with ca. Tc=90 K and also in measuring the wavelength-dependence of LB, LD, optical rotatory power, and CD at room temperature with the G-HAUP.

2:00 PM

(ICACC-S14-032-2017) Characterization approaches of femtosecond direct laser writing (DLW) induced modifications inside cubic YAG crystals (Invited)

I. Manek-Hönninger*[1]; W. Gebremichael[2]; S. Rouzet[1]; M. Chamoun[1]; M. Dussauze[3]; P. Bon[4]; A. Fargues[5]; V. Jubera[5]; T. Cardinal[5]; Y. Petit[6]; L. Canioni[1]; 1. CELIA - University of Bordeaux - CNRS - CEA (UMR5107), France; 2. Amplitude Systèmes and CELIA - University of Bordeaux - CNRS - CEA (UMR5107), France; 3. ISM - CNRS - University of Bordeaux - Bordeaux INP (UMR5255), France; 4. LP2N - IOGS - University of Bordeaux - CNRS (UMR5298), France; 5. ICMCB - CNRS (UPR9048), France; 6. ICMCB-CNRS (UPR9048) and CELIA - University of Bordeaux - CNRS - CEA (UMR5107), France

Recent developments in femtosecond DLW allow for realizing a vast range of photonic devices which is a driving force behind industrial interest for rapid prototyping of optical components. Thus, it is necessary to have a better understanding and approach for qualitative and quantitative characterizations in both waveguide and luminescence properties of modified transparent materials. Yttrium aluminium garnet (YAG) is a common crystal widely used as gain medium in solid state lasers. Owing to its crystallographic structure, optical specificities and restrictions of low-symmetry crystal optics are neglected which enables a more simplified systematic approach to study modifications due to femtosecond DLW inside crystals along the damaged tracks In this contribution, we discuss the different types of waveguides and show some first results of characterization approaches of femtosecond DLW induced modifications in these cubic crystals. Mainly, we will focus on refractive index changes along and in the vicinity of the damage tracks for different writing parameters. Furthermore, we will investigate the qualitative and / or quantitative relationship of the spatial map of these changes with micro-Raman and micro-luminescence spectroscopy.

2:30 PM

(ICACC-S14-033-2017) Phosphor nanoparticles prepared by laser ablation in liquid and their optical applications (Invited)

H. Wada*[1]; A. V. Gubarevich[1]; O. Odawara[1]; 1. Tokyo Institute of Technology, Japan

Functionalized ceramics nanoparticles such as phosphor are one of promising materials due to the unique properties. The nanoparticles are widely used in various fields such as biomedical and energy research. In this talk, our previous studies about preparations and applications of the nanoparticles are summarized. Upconversion (UC) nanoparticles Y_2O_3:Er,Yb were prepared by laser ablation in liquid. In the case of UC phosphor, visible emission is observed by excitation of infrared (IR) light. UC nanoparticles are useful for biomedical application such as bioimaging because of low absorption of IR light for a living body. Prepared nanoparticles were highly crystalline and existence of dopants in a nanoparticle was verified by transmission electron microscopy/energy dispersive X-ray analysis (TEM/EDS). In the case of afterglow phosphor, emission is observed after blocking excitation. Afterglow nanoparticles $Sr_2MgSi_2O_7$:Eu,Dy were prepared by same method. Capping nanoparticle with polyethylene glycol (PEG) was investigated to improve the optical properties. Yellow phosphor YAG:Ce is used for white LED. Size reduction of phosphor is studied to reduce scattering loss of blue LED light. Single phase YAG:Ce nanoparticles without YAP and YAM as byproduct were prepared by same method. Photoluminescence properties were changed by nanosizing.

3:20 PM

(ICACC-S14-034-2017) Red Emission of Eu^{2+} Ions doped in Oxides and Zirconates for future LEDs (Invited)

C. Wickleder*[1]; 1. University of Siegen, Germany

Recently, the development of new red phosphors became very important due to designing novel LEDs. In general a blue light emitting GaN-Chip is coated with a yellow phosphor (YAG:Ce^{3+}), but additional phosphors are necessary to obtain satisfying light quality. The current aim is to find host lattices for Eu^{2+} in which the d-f-transition can be excited by the blue GaN-Chip followed by a low energy emission, which is a real challenge! For this purpose, oxides are a promising choice because of their thermal stability, nontoxicity and general low-energy emission of Eu^{2+} ions in these hosts. The respective materials are, however, not well investigated due to difficult preparation. SrO and CaO doped with Eu^{2+} are already described, but with low quality of the samples. For the first time, however, the preparation without reduction in H_2 atmosphere but by direct doping with EuO in Ta ampoules is reported here. Moreover, $CaZrO_3$ and $SrZrO_3$ doped with Eu^{2+} will be presented for the first time, which are extremely advantageous because they are air and long-time stable and excitable even in the blue-green region. All these phosphors show band emission in the red range, which can be assigned to the typical 5d-4f transitions of Eu^{2+}. In this context $CaZrO_3$:Eu^{2+} and CaO:Eu^{2+} emitting at 654 nm and at 734 nm, respectively, are "low energy emitting champions" and are thus extremely promising as future LED phosphors.

3:50 PM

(ICACC-S14-035-2017) Single crystal phosphors for high-brightness white lighting applications (Invited)

K. Shimamura*[1]; E. Víllora[1]; D. Inomata[2]; K. Iizuka[2]; 1. National Institute for Materials Science, Japan; 2. Tamura Corporation, Japan

Conventional white lighting packages consist of blue-LEDs and the yellow Ce^{3+}:$Y_3Al_5O_{12}$ (Ce:YAG) ceramic powder phosphors (CPPs). However, in applications that require high-brightness (HB), a conventional packaging possesses several drawbacks. That is why, a new concept of high-brightness white LEDs based on yellow Ce:YAG single crystal phosphors (SCPs), which can overcome the conventional temperature- and photo-degradation problems of CPPs, is proposed. SCPs demonstrated high internal quantum efficiency (QE_{int}) (over 95%), outstanding thermal stability of QE_{int} in the temperature range 25-300°C, and quit low temperature increase under high blue irradiation, which contrast with the performance of CPPs. Ce^{3+}:$Lu_3Al_5O_{12}$ (Ce:LuAG) SCP is also proposed as an efficient and clear green source. SCP powders obtained by crashing SCPs have shown the same performances as SCPs. These natures favor the binder free HB packaging based on LD.

4:20 PM

(ICACC-S14-036-2017) Quenching growth of single- and poly-crystal phosphors using melt synthesis method

K. Toda*[1]; S. Kim[1]; M. Sato[1]; 1. Niigata University, Japan

In this study, we report the melt synthesis of single- and sintered poly-crystal phosphors using an arc-imaging furnace. The melt reactions are very fast (10 - 60 s) and result in homogeneous compounds owing to rapid diffusion and mixing in the liquid phase. Therefore, melt synthesis techniques are suitable for preparing multi component homogeneous phosphor compounds. Interestingly, single crystal of yellow emitting Ba3Al(PO4)3:Eu and red emitting NaMgPO4:Eu can be synthesized by the melt quenching technique. Both phosphors were thermodynamically metastable materials and have never been synthesized by a conventional solid state reactions.

4:40 PM

(ICACC-S14-037-2017) Synthesis of V^{5+}–rich CaV_2O_6 Nanoparticle by Water Assisted Solid State Reaction (WASSR) Method

M. Watanabe*[1]; M. Muto[1]; S. Kim[2]; T. Kaneko[1]; K. Uematsu[1]; T. Ishigaki[3]; K. Toda[1]; M. Sato[1]; Y. Kudo[4]; T. Masaki[5]; D. Yoon[5]; 1. Niigata University, Japan; 2. Sejong University, Republic of Korea; 3. Tottori University, Japan; 4. N–Luminescence Corporation, Japan; 5. Sungkyunkwan University, Republic of Korea

CaV_2O_6 has been actively investigated because of their cheapness, lower toxicity, and interesting properties such as microwave dielectric properties [1], photocatalytic activity, and red broadband emission [2]. Their nanoparticles have been required because it has been expected to improve dielectric property, and photocatalytic activity. In addition, the nanoparticle with luminescent property has been expected to be used in solar energy converters and bio-detectors due to their permeability. CaV_2O_6 has been usually prepared by a conventional solid state reaction method. This method exhibits little multiplicity in term of morphological and size controls of the particle, because almost particles synthesized by the method have the only microscale with granular morphology. In this study, we proposed a novel soft chemical synthetic method, water assisted solid state reaction (WASSR) method which is very simple method, synthesizing many ceramic nanomaterials just by storing the mixture of raw materials added a small amount of water in closed containers at low temperature. CaV_2O_6 was synthesized by the WASSR method at 297–393 K using $CaCO_3$ or $Ca(OH)_2$ and V_2O_5. The morphology and optical properties of CaV_2O_6 prepared by the WASSR method were compared.

Poster Session B

Room: Ocean Center Arena

5:00 PM

(ICACC-FS1-P069-2017) Biomass ashes as activators in the synthesis of inorganic polymers: From a model system to a blend of industrial residues

A. Peys*[1]; A. Mobili[2]; A. Katsiki[3]; L. Arnout[1]; H. Rahier[3]; B. Blanpain[1]; Y. Pontikes[1]; 1. KULeuven, Belgium; 2. Università Politecnica delle Marche, Italy; 3. Vrije Universiteit Brussel, Belgium

Recent literature suggests inorganic polymers as more environmentally friendly alternatives to ordinary Portland cement. Although a large decrease in CO_2 emissions is observed, there is no general consensus on the full spectrum of environmental "friendliness", including other impacts like eutrophication of the soil or depletion of resources. In addition, the high alkalinity of the solutions that are used in the synthesis of inorganic polymers is criticized for its shortcomings with respect to operation friendliness. The present work investigates the synthesis of one-part ("add water only") inorganic polymers, using biomass ashes as an alkaline activator. The drive of this work is to eliminate the concerns on the alkalinity and to lower environmental impact of the activating solutions. A model system of metakaolin and maize ashes is discussed, after which a transition is made to industrial residues, both for the precursor (an iron-silicate slag) and for the alkaline activator (ashes from a biomass incineration power plant). The compressive strength of the metakaolin – maize ash system reached up to 40 MPa, while the industrial residue system had a maximum of 10 MPa. The latter leaves room for optimization, e.g. in the use of a more reactive slag or pretreatments of the ash.

(ICACC-FS1-P070-2017) Development of novel Aluminosilicate Phosphate Cement for Structural Applications

A. Katsiki*[1]; A. Peys[2]; T. Tysmans[3]; J. Wastiels[3]; H. Rahier[1]; 1. Vrije Universiteit Brussel, Belgium; 2. KULeuven, Belgium; 3. Vrije Universiteit Brussel, Belgium

In this study, the prospects of using acidic activation of metakaolinite (MK) as the main binder in cement production were investigated. The setting reaction of metakaolinite/phosphate cement (MKPC) was investigated by means of isothermal calorimetry (TAM Air), differential scanning calorimetry (DSC), thermogravimetry (TGA), scanning electron microscopy (SEM) and X-ray diffraction (XRD). The reaction of metakaolinite powder with the aqueous phosphoric acid solution led to the formation of a phospho-aluminosilicate cement. The reaction kinetics were studied by varying the Si/P molar ratio from 0.52 to 2.94 and by keeping the quantities of metakaolinite and water in the system constant. The results revealed that the optimum stoichiometric value for the reaction of metakaolinite with the phosphoric acid was close to 1.1 Si/P molar ratio. The compressive strength was found to be strongly dependent on the $PO_{2.5}$ concentration. In fact, the decrease in the Si/P molar ratio led to an increase in strength up to a maximum around a Si/P ratio of 1.1. Thereafter, by further decreasing the Si/P molar ratio, a decrease in strength occurred again due to the excess of phosphates and even self-degradation of the specimens after initial hardening. The investigated MKPC mortar achieved strengths up to 70MPa in compression after 7 days, which render it a promising candidate for construction purposes.

(ICACC-FS1-P071-2017) Adsorption of Mn(II) and Co(II) Ions from Aqueous Solution by Using Geopolymers

I. Kara*[2]; D. Yilmazer[1]; S. Tunali Akar[1]; 1. Eskisehir Osmangazi University, Turkey; 2. Anadolu University, Turkey

Geopolymers are low cost materials as they are produced without the application of temperatures by using metakaolin and/or waste materials such as fly ash. They intrinsically contain substantial amount of nano porosity, and thus, are considered to be alternative materials for the removal of hazardous metal ions from waste waters by adsorption. This study addresses the effectiveness of metakaolin based geopolymer to adsorb Mn(II) and Co(II) ions from aqueous solution in order to assess their suitability for wastewater treatment. Adsorption conditions (pH, concentration, temperature, amount of adsorbent) were systematically varied and the best conditions for adsorptions were established. It was found that Mn(II) and Co(II) ions can be removed from aqueous solutions by metakaolin based geoplymer with almost complete efficiency.

(ICACC-FS1-P072-2017) Processing of Porous Geopolymers

F. Kara*[1]; B. Yazirli[3]; C. Demir[2]; 1. Anadolu University, Turkey; 2. Afyon Kocatepe University, Turkey; 3. Kaleseramik, Turkey

Heat insulation of buildings relies on using porous materials such as polystyrene and mineral wool. Polystyrene is rather good for insulation but it is not fire resistant. On the other hand, mineral wool is a fire resistant alternative but with less insulating performance, particularly in humid conditions due to its open porous structure. There are also cement based porous materials emerging in the market as an alternative fire resistant insulating materials. Like cement based materials, geopolymers could also be an environmentally viable insulating material. Therefore, in this study, low density porous geopolymers by foaming were attempted to be developed by using metakaolin-sodium silcate-sodium hydroxide mixtures. Peroxide was used as foaming agent. It was observed that rather low density (down to 0.2 g/cm^3) geopolymer foams with homogeneous pore size distriution can be produced by using suitable additives that prevent pore coalesence.

(ICACC-FS1-P073-2017) Biomorphous potassium-based geopolymer (KGP)/C-ceramic composites derived from wood template

M. Yu*[1]; E. Bernardo[2]; P. Colombo[2]; A. R. Romero[2]; P. Tatarko[1]; V. K. Kannuchamy[3]; M. Titirici[3]; M. J. Reece[1]; 1. Nanoforce Technology Ltd, United Kingdom; 2. Dipartimento di Ingegneria Industriale, Italy; 3. School of Engineering and Material Science, United Kingdom

Anisotropic, biomorphic (wood derived) potassium-based geopolymer (KGP)/C-ceramic composites with net shape were manufactured by infiltrating KGP slurry into carbonized porous scaffolds derived from native beech wood. The infiltration process is pumping out the air from porous scaffolds and then followed by infiltrating geopolymer slurry into scaffolds under high vacuum. 70% of pores in scaffolds were fully infiltrated by KGP slurry. Compared to pure geopolymer, the work of fracture of KGP/C-ceramic composites was increased by 300%. After firing, the compressive strength of KGP-infiltrated scaffolds increased to 25MPa, due to the transformation from geopolymer to Leucite (K_2O Al_2O_3 $4SiO_2$) in KGP/C-ceramic composites. Effects of temperature and heating atmosphere (air, argon and nitrogen) on the phase transformation in KGP/C ceramic composites have been investigated. FTIR, TGA and SEM analysis were applied to monitor changes in microstructures and phase formation processes during thermal treatment.

(ICACC-FS1-P135-2017) Particle and Fiber Reinforced Metakaolin-Based Geopolymer Composites: A Review

R. A. Sa Ribeiro*[1]; M. G. Sa Ribeiro[1]; W. M. Kriven[2]; 1. INPA-National Institute for Amazonian Research, Brazil; 2. University of Illinois at Urbana-Champaign, USA

Research on reinforced metakaolin-based geopolymer for structural applications is reviewed. Geopolymer has been synthesized using metakaolin produced from kaolinite extracted from several regional soils. Kaolin is converted into metakaolin by calcination from 650°C up to 800°C. For higher strength and stiffness achievements, the geopolymer matrix is reinforced with particles and fibers. In addition, synthetic and natural particles and fibers have been used to enhance durability, thermal properties and shrinkage ratio of lighter geopolymer composites. Due to the unavailability of a standard for processing and testing geopolymer composites, different laboratories use differing procedures, making data comparison very difficult. The promising market of geopolymer composites for the sustainable construction industry would benefit from a conformed standard for laboratory processing and testing. This would contribute for the creation of a large and reliable data bank, towards the manufacture and certification of geopolymeric sustainable construction materials.

(ICACC-FS1-P136-2017) Carbbottle Methodology for Evaluation of Carbonate Cement Produced by Hydrothermal Vapor Synthesis

R. Anderson*[1]; D. Kopp[1]; R. Riman[1]; 1. Rutgers University, USA

Carbonate cement ($CaSiO_3$) concrete is a disruptive, innovative technology that has the potential to replace hydraulic cement for infrastructure development and drastically reduce the CO_2 emissions of the cement industry. Both mineral and synthetic $CaSiO_3$ can be used for concrete solidification, but $CaSiO_3$ produced by hydrothermal vapor synthesis (HVS) at low temperatures (<350°C) generates particles with smaller sizes and higher surface areas than natural or kiln-synthesized materials that require milling. Carbonation of $CaSiO_3$ pastes can take a few of hours to a couple of days depending on the sample properties and reaction environment. “Carbbottle” methodology is a simple measurement technique to monitor cement carbonation kinetics using inexpensive equipment. Optimization of processing variables such as the water/cement ratio (0.1-1.0) and green relative density (40-60%) of $CaSiO_3$ powder compacts was enabled by this technique. By combining this comparative carbonation method with compressive strength data it is possible to rapidly analyze and optimize properties such as strength as well as establish specifications for sample processing and compositional formulation for both laboratory and industrial applications.

(ICACC-FS1-P141-2017) Dolomite ($CaMg(CO_3)_2$) Particulate-reinforced Geopolymer Composite

W. M. Kriven[1]; P. F. Keane*[1]; 1. University of Illinois at Urbana-Champaign, USA

Dolomite powder ($CaMg(CO_3)_2$ or $CaCO_3$●$MgCO_3$) is an industrial waste material obtained as a sediment resulting from grinding of dolomite rocks. Representative microstructures, particle size and size distributions were measured. Increasing amounts of dolomite powder were dispersed in sodium and potassium-based geopolymer. The mechanical compressive and flexure properties were measured in three-point bending at room temperature, according to ASTM standard C-78, as well as four-point bending at high temperatures to 1000°C according to ASTM standard C-1341. The thermal conductivities were also measured as a function of solids loading. The microstructures were examined by XRD and SEM/EDS.

(ICACC-S1-P096-2017) Processing and properties of an inexpensive, low-fire, basalt-fiber reinforced ceramic composite material

G. P. Kutyla[2]; P. F. Keane*[1]; C. Marsh[1]; W. M. Kriven[2]; 1. Engineering Research and Development Center, USA; 2. University of Illinois at Urbana-Champaign, USA

Clay-based ceramics are used in a wide variety of applications and could benefit from the addition of a reinforcing fiber. Previous work showed that strength could be improved by 20-40% with the addition of only 10 wt% of milled basalt fibers. However, it was found that temperatures greater than 1050°C would result in the melting and degradation of the basalt fiber, which limited the achievable density to less than 85% of theoretical density. To decrease the temperatures necessary for densification, sintering aids were added to the matrix clay. These included nepheline, feldspar, sodium borate, and ammonium borate. The nepheline and feldspar additives did not improve density or mechanical properties at the temperatures compatible with basalt reinforcement (1050°C or below). While the sodium borate additive did not improve densification at these temperatures, it did improve the mechanical properties. It also resulted in some glassy behavior during firing at 1050°C.

(ICACC-S1-P074-2017) Effect of Ion Irradiation on Bioactivity of Hydroxyapatite Ceramics

S. Kobayashi*[1]; T. Izawa[1]; Y. Teranishi[2]; 1. Tokyo Metropolitan University, Japan; 2. Tokyo Metropolitan Industrial Research Institute, Japan

Effect of ion irradiation conditions on bioactivity of HA was investigated. Ca or Mg ion was irradiated on HA surface at different ion dose and in depth. Surface potential of ion irradiated specimens was measured using Atomic Force Microscopy (AFM). The bioactivity was investigated by soaking in simulated body fluid (SBF) followed by surface observations using a scanning electronic microscope (SEM). The HA irradiated with Ca ion at different ion dose and in depth showed acicular apatite formation on the surface after soaking in SBF at all conditions. However, apatite formation rate was different at each ion irradiation condition. The rate increased with the ion dose and depth. Apatite formation on the HA irradiated with Mg ion became lower with increasing Mg ion dose. It was found that Ca ion irradiation increased the bioactivity of HA. On the other hand, it was also found that Mg ion irradiation decreased it. It suggested that these results were due to surface potential changes by ion irradiation. The surface irradiated with Ca ion had surfaces positively charged and the surface irradiated with Mg ion had negative potential. Thus, the apatite formation of HA surface can be controlled by changing ion species, dose and depth. This means that the change in microstructure of HA can control the bioactivity appropriate for use site.

(ICACC-S1-P075-2017) Effect of Thermal Shocks on the Residual Compressive Strength of Concrete

J. K. Mann*[1]; 1. Government, India

The main objective of this investigation was to study the effect of thermal shock during cooling on the compressive strength of concrete exposed to varying temperature. The tests were carried out on cylindrical specimens 150mm in diameter & 300 mm in height made of Ordinary Portland Cement of grade 43. The design mix used was M20. The specimens were heated up to 300°, 500°, 700°& 900° C &then cooled down to temperature in two different ways. A first group of specimens was cooled in air (naturally), while second group was immersed in water for 5 minutes. After the rest period in water the specimens were cooled in air. The next day, after the heating cooling process, the residual compressive strength was measured. An analysis of tests shows that exposure temperature and cooling method have a significant influence on the residential compressive strength of the specimen. The study reveals that air cooled concrete specimen maintained high residual compressive strength than water cooled specimens.

(ICACC-S1-P076-2017) Influence of Free Carbon Elimination on Microstructure of ZrC-SiC composite ceramic fibers

X. Lv*[2]; M. Ge[1]; S. Yu[1]; Y. Tian[2]; W. Zhang[1]; 1. Institute of Process Engineering, Chinese Academy of Sciences, China; 2. University of Chinese Academy of Sciences, China

EB-cured in N_2 polyzirconocenecarbosilanes (PZCS, a novel polymeric precursor) fibers were pyrolyzed at 1100°C under controlled hydrogen/nitrogen atmosphere to eliminate free carbon in ZrC-SiC fibers, and subsequently heat treated at 1500°C under argon atmosphere. The elemental composition of fibers during pyrolysis was investigated using Elemental analyzer, EDS and XPS analysis. The microstructure were examined by SEM, TEM and XRD. The results show that the carbon content in ZrC-SiC fibers decreases with H_2 concentration increasing. The hydrogen atmosphere suppresses H_2 evolution and helps to remove excess carbon. The microstructures of these fibers exhibited nanoscale ZrC particles dispersed in continuous SiC matrix containing different free carbon contents.

(ICACC-S1-P077-2017) Synthesis and characterization in praseodymium doped $BiFeO_3$ multiferroic ceramics

C. Tu[1]; Y. Hsieh*[1]; Y. Huang[1]; 1. Fu Jen Catholic University, Taiwan

A-site rare-earth substitution in $BiFeO_3$ has been used to enhance ferroelectric and magnetic properties. This work reports structures, electronic states, phonons, magnetizations, and polarizations of single-phase praseodymium (Pr)-doped $BiFeO_3$ $(Bi_{1-x}Pr_x)FeO_3$ (BFO100xPr; x=0.0, 0.05, 0.10) prepared by solid-state-reaction method with sintering temperature of 850 C (3 hrs). Characterizations have been performed by using synchrotron X-ray diffraction (XRD), scanning electron microscopy (SEM), synchrotron X-ray absorption spectroscopy (XAS), and high-resolution transmission electron microscopy (HRTEM), back-scattering Raman spectroscopy, and magnetization/polarization hysteresis loops. The SEM morphologies suggest that average grain size decreases with increasing Pr substitution. A rhombohedral phase with R3c space group was identified for BFO5Pr and BFO10Pr at room temperature. Raman-active modes exhibit a linear shift toward lower frequencies with increasing temperature. The Fe K edge XAS reveals that the valence of Fe ion remains 3+ in BFO5Pr and BFO10Pr. The oxygen K edge absorptions (from the O 1s→O 2p transition) suggest that the hybridization between O 2p and Fe 3d orbitals is stronger in BFO5Pr than in BFO10Pr. A linear antiferromagnetic hysteresis loops were observed in BFO5Pr and BFO10Pr.

(ICACC-S1-P078-2017) Porous Graphite from Pitches: Manufacturing and Mechanical Characterization

H. Hosseini*[1]; S. Ghaffarian[2]; M. Teymouri[3]; A. Moeini[3]; 1. University of Missouri, Columbia, USA; 2. Amirkabir University of Technology, Islamic Republic of Iran; 3. Research Institute of Petroleum Industry, Islamic Republic of Iran

Porous graphite has been synthesized by two step polymerization of coal tar pitch under heat and pressure. First step is attributed to the meso carbon micro bead (MCMB) formation in the pitch matrix via heating to the maximum fluidity temperature and applying pressure to maintain stability in MCMB production. XRD and optical microscopy results showed the formation of stacking aromatic carbon sp2 layers and enhanced graphitization degree. MCMB formation is essential for the next step due to its graphite transformation in the matrix. Second step belongs to foam production based on internal bubble formation, as a result of pyrolysis reactions and releasing of remaining volatile matters restricted by external pressure. XRD presented magnified peaks of graphite crystals formed on the ligaments of pore walls. SEM and OM depicted the formation of pores throughout the mesophase pitch media. Mechanical properties proved the capability of synthesized porous structure to use as a core in sandwich composite construction (Compressive modulus of 0.175 GP and ultimate compressive strength of 12.5 MPa), furthermore, low density and light weight features ($\rho < 1g/cm3$ and higher specific weight) are another brilliant characteristics of these noble materials make them favorable to utilize in light weight material constructions.

(ICACC-S1-P079-2017) Domain structure and phase transition in samarium doped $BiFeO_3$ near morphotropic phase boundary

C. Yu*[1]; C. Tu[1]; W. Wu[1]; 1. Fu Jen Catholic University, Taiwan

Domain structures and phase transitions across a ferroelectric rhombohedral to antiferroelectric orthorhombic boundary in polycrystalline $(Bi_{1-x}Sm_x)FeO_3$ (BFO100xSm) solid solutions have been investigated for x=0.12, 0.14, and 0.16 by high-resolution synchrotron X-ray diffraction, TEM and back-scattering Raman spectroscopy. For x=0.12, a rhombohedral R3c space group was identified by the 1/2(000) superlattice diffraction. At the critical composition of x=0.14, appearance of ½(000) and ¼(100) diffractions reveals a coexistence of R3c and orthorhombic Pbam space groups. For x=0.16, a mixture of R3c, Pbam, and Pbnm (or Pnma) space groups was observed with ½{100}, ½{110}, ½{111}, ¼{100}, ¼{110}, and ¼{112} diffractions. The synchrotron Fe L_3 and oxygen K-edge X-ray absorption suggest that the hybridization effect of the O 2p and Fe 3d orbitals was decreased by the A-site Sm^{3+} substitution. BFO12Sm exhibits a structural transition from rhombohedral R3c to another rhombohedral phase at ~573-623 K. BFO14Sm shows a transition from a coexistence of coexistence of R3c and orthorhombic Pbam space groups (R3c+Pbam) to another rhombohedral structure at ~473-523 K. A transition from a R3c+Pbam+Pbnm to another rhombohedral structure takes place at ~373-423 K in BFO16Sm.

(ICACC-S1-P080-2017) Torsion tests on SiC/SiC joined by "Mo-wrap" method

P. Gianchandani*[1]; V. Casalegno[1]; M. Ferraris[1]; 1. Politecnico di Torino, Italy

SiC/SiC, sintered- and CVD-SiC joined by a MoSi2/Si composite obtained in-situ by reaction of silicon and molybdenum at 1450°C in Ar, the "Mo-wrap" method, have been characterized by three different mechanical tests (single lap, single lap off-set and torsion). Fracture surfaces were studied in order to find the pros and cons of each test method, with the focus on measurement of true or apparent shear strength of the composite (MoSi2/Si) joining material. Single lap was unsuitable to test the strength of these joints, due to interlaminar failure of SiC/SiC or fracture propagating inside the sintered SiC

(ICACC-S1-P081-2017) SiC foam sandwich structures obtained by Mo-Si2 joining

P. Gianchandani*[1]; V. Casalegno[1]; M. Ferraris[1]; G. Bianchi[2]; A. Ortona[2]; 1. Politecnico di Torino, Italy; 2. The University of Applied Sciences and Arts of Southern Switzerland (SUPSI), Switzerland

The combination of lightweight and high temperature stability is of interest for several advanced applications ranging from aerospace to energy production, thermal protection systems and shielding of electromagnetic waves. This work presents the development of several silicon carbide foam sandwiched between two ceramic matrix composites skins (C/SiC or SiC/SiC) by joining the SiC foam to composites with the "Mo-wrap" technique; the "Mo-wrap" technique is based on a $MoSi_2$/Si joining material obtained by in-situ reaction of silicon and molybdenum at 1450°C in Ar flow. The "Mo-wrap" technique, already successfully employed to join SiC-based materials, was found effective also to control joining material infiltration in SiC foams. The sandwich structures is tested in compression and simulated working environment

(ICACC-S1-P082-2017) Joining of C/SiC to Ti-6Al-4V by Zirconium-based brazing alloys

M. Bangash*[1]; V. Casalegno[1]; M. Ferraris[1]; 1. Politecnico di Torino, Italy

C/SiC are promising candidates for several advanced applications due to their high specific strength, low density and high thermal stability. They are valuable for applications in aerospace industry with demanding thermal and mechanical requirements. Ti6Al4V is a high strength, formable, lightweight and corrosion resistant Ti alloy. It accounts for more than 50% of total Ti usage and is a world standard in aerospace applications. Most such applications requires joining of Ti alloys to C/SiC composites. Zr based amorphous alloys, chosen as brazing alloys, have high corrosion resistance, mechanical strength and fracture toughness. This work presents the activity on joining of C/SiC to Ti6Al4V by brazing, by using TiB-590 (Zr-21Ti-16Ni-1Hf wt %) braze at 900°C for 10 minutes in Argon atmosphere. The joints microstructure is characterized by Optical Microscopy, Scanning Electron Microscopy (SEM) and Energy Dispersion Spectroscopy (EDS). The joint strength is evaluated by Single Lap (SL) and Single Lap Offset (SLO) mechanical testing. The microscopic results show a uniform joining seam and reaction layers are formed in the joint area near both Ti6Al4V and C/SiC interface. Failure was due to composite delamination and not at the composite/metal interface.

(ICACC-S1-P083-2017) Joining of Nextel ™610/YAG-ZrO_2 ceramic composites using brazing alloys

M. Akram*[1]; V. Casalegno[1]; M. Ferraris[1]; T. Wamser[2]; W. Krenkel[2]; 1. Politecnico di Torino, Italy; 2. University of Bayreuth, Germany

Nextel™610/YAG-ZrO_2 oxide fiber composites were successfully joined using commercial and modified Cu-based and Ti-based brazing alloys in Argon atmosphere. All brazing materials showed good wetting properties with the composites. Scanning electron microscopy (SEM) showed that the joint interfaces were sound, continuous and free of defects. The energy dispersive X-ray spectroscopy (EDS) was used to investigate the elemental distribution and formation of different phases at the interfaces. Lap shear tests were performed at room temperature to check joint reliability in working conditions.

(ICACC-S1-P085-2017) Spark Plasma Sintering (SPS) of Silicon Carbide Ceramic Composites

S. Subhash*[2]; A. Trachet[1]; 1. University of Florida, USA; 2. Buchholz High School, USA

Silicon carbide (SiC) is lightweight and strong but brittle. Thus, the objective of this research was to develop SiC ceramics that were not only hard but also tough. SiC powder was mixed with varying amounts of metal and ceramic powders (e.g., cobalt and silicon) in order to improve the toughness of the finished ceramic. The mixed powders were sintered using spark plasma sintering (SPS) at temperatures between 1700 and 1800°C and pressures ranging from 30 to 35 MPa. The elastic modulus (stiffness) and Vickers hardness of the sintered ceramics were measured using ultrasonic methods and indentation hardness testing, respectively. Fracture toughness values were estimated from indentation crack length measurements and were employed to compare relative fracture toughness values between ceramics produced in this study. The majority of the ceramic composites exhibited hardness values on par with typical SiC (21 to 24 GPa), and it was noted that ceramics containing silicon as a secondary phase displayed a promising combination of hardness and toughness. The details of processing and properties of these ceramic composites and the relevant mechanisms contributing to these properties will be presented. In addition, challenges particular to the SPS of SiC will be discussed.

(ICACC-S1-P086-2017) Evaluation of mechanical properties of a carbon fiber using molecular dynamics and Mori-Tanaka theory

T. Niuchi*[1]; J. Koyanagi[1]; R. Inoue[1]; Y. Kogo[1]; 1. Tokyo University of Science, Japan

Carbon fiber is produced by heat treatment of Polyacrylonitrile (PAN) in several steps. With the increase of heat treatment temperature (HTT), crystallization of carbon fiber typically increases. Based on its structure, PAN-based carbon fiber can be divided into two regions, one that has a crystal structure (similar to that of graphite) and the other, whose structure is amorphous. While the mechanical properties of crystal carbon fibers are already well know, those of amorphous structured fibers are not yet fully understood. Perhaps one of the reasons for that is their difficult experimental examination. In order to improve and design mechanical characteristics of PAN-based carbon fiber, their evaluation must be made. In this study, several patterns of amorphous region in carbon fibers are modeled based on following assumptions: its molecular structure is simple, molecules consist of hydrogen and oxygen atoms, and the molecular structure is dependent on HTT. Moreover, these models are adopted to perform a simulation on the elastic modulus and the interfacial stress between the crystal and amorphous model using molecular dynamics simulation technique.

(ICACC-S1-P087-2017) Mechanical properties and microstructure characterization of SiC fiber reinforced SiC-ZrC nanocomposite matrix composites

X. Wei*[1]; H. Zhang[1]; W. Zhang[1]; 1. Institute of Process Engineering, Chinese Academy of Sciences, China

SiC ceramic matrix composites (SiC CMC) have been paid considerable attention due to their superior performances. SiC CMCs are suitable to be used in the oxidation atmosphere below 1650°C theoretically. While it is beyond the limit of SiC matrix under the environment of higher temperature, high speed and complicated flow. Many efforts have been reported to introduce UHTCs into SiC CMCs. It is generally accepted that small size and uniform distribution of UHTCs in SiC are greatly beneficial to improve their performance. It was reported that the micro-sized and sub-micro-sized UHTCs particles had been introduced into the SiC CMCs. In this work, we prepared CMCs with SiC-ZrC nanocomposite matrix by chemical vapor infiltration combined with precursor impregnation & pyrolysis method using novel polyzirconiumcarbosilane (PZCS) as precursors. 2.5D SiC textile preforms were employed as reinforcement. The mechanical properties and microstructure of the composites were investigated. The flexural strength, fracture toughness and work of fracture of the composites were 436.0 MPa, 13.7 MPa $m^{1/2}$ and 14.6 kJ/m^2 respectively. The composites presented a typical nonlinear fracture behavior and pseudo-plastic failure mode. The SiC-ZrC nanocomposite matrix was characterized with about 10 nm ZrC particles uniformly distributed in SiC matrix.

(ICACC-S1-P088-2017) SPS apparatus used as a testing tool for studying compression creep of polycrystalline magnesium aluminate spinel

B. Ratzker*[1]; M. Sokol[1]; S. Kalabukhov[1]; N. Frage[1]; 1. Ben-Gurion University of the Negev, Israel

Compression creep experiments on a fine-grained (250 nm) polycrystalline magnesium aluminate spinel were successfully performed using a spark plasma sintering (SPS) apparatus. The experiments were conducted in the 1100–1200°C temperature range, under an applied stress of 120–200 MPa. It was found that the stress exponent and activation energy depended on temperature and applied stress, respectively. The deformed samples were characterized by high resolution scanning electron microscope (HRSEM) and high resolution transmission electron microscope (HRTEM). The results indicate that the creep mechanism was related to grain boundary sliding, accommodated by dislocation slip and climb. The experimental results, extrapolated to higher temperatures and lower stresses, were in good agreement with data reported in the literature. It has to be pointed out that in the present study SPS apparatus used for the first time as an analytical testing tool for investigation of creep in ceramics at elevated temperatures.

(ICACC-S1-P089-2017) Cataloging Anomalous Nanoindentation Behavior and Mechanical Properties in Rare-Earth Orthophosphate Ceramics

T. Wilkinson[1]; D. Wu[1]; M. Musselman[1]; C. Packard*[1]; 1. Colorado School of Mines, USA

Rare-earth orthophosphates ($REPO_4$) exhibit incredible compositional flexibility as well as excellent thermal- and chemical-resistance, which makes them candidates for a wide array of applications including proton conductors, nuclear waste storage, and fiber coatings in ceramic matrix composites. $REPO_4$s exist in either a monazite (Z: 57 – 64) or xenotime structure (Z: 65 – 71) in equilibrium at ambient conditions. Xenotime compositions near the monazite/xenotime border are known to undergo pressure-induced phase transformations and some reportedly have lower indentation modulus and hardness values than expected, showing anomalous unloading behavior in the form of 'elbows' where the slope in the data changes distinctly. We catalog the indentation behaviour of $EuPO_4$, $GdPO_4$, $TbPO_4$, and $DyPO_4$, identifying the frequency of anomalous behaviour and measuring the mechanical properties of the materials over a range of conditions. It is shown that elbow-type behavior is apparent in all of the examined materials surrounding the monazite/xenotime boundary, including those that are non-transforming; thus we conclude that the presence of an elbow is not a unique identifier of phase transformation in $REPO_4$s. It is also shown that the mechanical properties of all these compositions approach the modulus values predicted in simulations, provided that the samples are nearly fully dense.

(ICACC-S1-P090-2017) Phonon Anomalies and Magnetization in A-Site Doping $(Bi_{1-x}A_x)FeO_3$ Ceramics

W. Wu*[1]; C. Tu[1]; 1. Fu Jen Catholic University, Taiwan

Phonon vibrations and magnetizations have been characterized in polycrystalline $(Bi_{1-x}A_x)FeO_{3-d}$ (A=Ca, Sr, Ba, Ho, Lu) solid solutions for x=0-0.15 as functions of temperature (123-300 K). The $(Bi_{1-x}A_x)FeO_{3-d}$ (A=Ca, Sr, Ba, Ho, Lu) (BFO100xA) ceramics. Enhanced ferromagnetic magnetization loops were observed in Ba and Ho-doped compounds. The Raman modes of the R3c rhombohedral $BiFeO_3$ are $4A_1$+9E. Four major Raman modes appear below 250 cm^{-1}; E(1) at ~60-80 cm^{-1}, E(2) at ~130-140 cm^{-1}, $A_1(1)$ at ~160-170 cm^{-1}, and $A_1(2)$ at ~210-220 cm^{-1}. The Raman modes in the lower frequencies (below 170 cm^{-1}), middle frequencies (~150-270 cm^{-1}), and higher frequencies (>260 cm^{-1}) correspond to atomic vibrations associated with (Bi,Ca, Sr, Ba, Ho, Lu), Fe, and O atoms, respectively. Raman spectra (60-600 cm^{-1}) reveal broadening profiles due to randomness of A-site ion distribution. The Raman vibrations illustrate frequency shifts due to A-site substitution. To quantitatively analyze frequency shift and full-width-at-half-maximum (FWHM), the Raman spectra were fitted with a Lorentzian function. Temperature-dependent Raman frequency shifts of the E(2) and $A_1(1)$ modes exhibit an frequency anomaly in the region of 150-200 K in (Ca, Sr, Ba)-doped compounds. The frequency anomalies in the Fe-sensitive E(2) and $A_1(1)$ modes in the region of 150-200 K likely result from the coupling between phonons and low-lying magnons.

(ICACC-S1-P091-2017) Dispersion and reinforcing mechanisms of multi-walled carbon nanotubes in a ceramic material

M. Estili*[1]; Y. Sakka[1]; 1. National Institute for Materials Science (NIMS), Japan

Our recent achievements in the processing of defect-free multi-walled carbon nanotube (MWCNT) – ceramic matrix composites and a direct characterization of interfacial shear resistance and mechanical response of individual MWCNTs while embedded in the ceramic matrix will be briefly presented. We also highlight a new concept, which led to the fabrication of the first and most CNT–concentrated ceramic material ever (20 vol% MWCNT) with full density (~99%), which shows unprecedented electrical transport (~5000 S/m at RT) and doubled strain tolerance characteristics suitable for various functional and structural applications. Furthermore, we discuss how a recently discovered nanoscale in-MWCNT load-transfer process, at an optimized, high loading of MWCNTs (10 vol%) and in a pore-free and uniform matrix structure, could lead to unprecedented, simultaneous enhancement in strain tolerance (81%; average 0.0019), fracture toughness (52.2%; average 6.71 MPa.m0.5), and flexural strength (22%; average 483.19 MPa) of the Al2O3 ceramic matrix.

(ICACC-S1-P092-2017) Mechanical and tribological properties of boron carbide/graphene platelets ceramic composites

A. Kovalcikova*[1]; R. Sedlak[1]; P. Rutkowski[3]; J. Balko[1]; E. Mudra[1]; V. Girman[2]; J. Dusza[1]; 1. Institute of Materials Research Slovak Academy of Sciences, Slovakia; 2. Pavol Jozef Šafárik University in Košice, Faculty of Science, Slovakia; 3. AGH University of Science and Technology in Krakow, Poland

Boron carbide based composites containing up to 6 wt. % of graphene platelets (GPLs) were prepared by hot pressing at 2100°C for 1h. The influence of GPLs addition on the microstructure development, mechanical and tribological properties of B_4C/ GPLs composites has been investigated. The friction and wear behaviour using ball-on-flat technique with SiC and WC-Co balls as friction counterpart under an unlubricated condition at room temperature have been determined. The GPLs were relatively well distributed in the B_4C matrix, located at the B_4C/B_4C grain boundaries, however the platelets were often not in single form but stucked together two or more. All investigated mechanical properties were the highest in the case of B_4C + 0.5 wt. % GPLs composite. Higher GPLs content resulted in lower mechanical properties except fracture toughness. Processing flaws in the form of GPLs agglomerates and un-sintered areas were identified as fracture origins. The main toughening mechanisms, responsible for the increased fracture toughness were crack deflection, bridging through GPLs necking, and GPLs pull-out. With increased GPLs addition the coefficient of friction was stable, but significant improvement of wear rate has been observed. The wear rate significantly decreased ~77% in the case of B_4C+6 wt. % GPLs when compared to reference material at load of 5 N, and ~ 60% at load of 50 N.

(ICACC-S1-P093-2017) Influence of addition and holding times on the mechanical properties of in-situ Al/TiB_2 composites

A. Vivekananda[2]; S. Balasivanandha Prabu[2]; R. Paskaramoorthy*[1]; 1. University of the Witwatersrand, South Africa; 2. Anna University, India

In recent years, TiB_2 reinforced Al metal matrix composites (MMC) have been used in various applications. This paper reports the influence of two process parameters, namely holding time and addition time, on the formation and distribution of TiB_2 particles within Al/TiB_2 composites. The TiB_2 particles were formed by the in-situ reaction of potassium hexafluoro titanate (K_2TiF_6) and potassium tetrafluoroborate (KBF_4) with molten aluminium at various addition and holding times. The formation of TiB_2 was confirmed by XRD examination. The microstructure and size of the TiB_2 particles formed are found to be strongly affected by both addition and holding times. The microstructures displayed significantly different degrees of particle agglomeration. Hardness and ultimate tensile strength (UTS) are strongly affected by both addition time and holding time. The variations of hardness and UTS are mostly consistent with those of the microstructure of the composites. 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 51% and 44%, respectively, and this was for the composite sample fabricated with 20 minutes of addition time and 30 minutes of holding time. It is worthy to note that this composite displayed near-uniform particle distribution.

(ICACC-S1-P094-2017) Oxidative Exposure of Ceramic Matrix Composites: Post Flex and Acoustic Emission Analysis

G. Ojard*[1]; D. Goberman[1]; J. Cardinale[1]; J. Holowczak[1]; 1. United Technologies Research Center, USA

Ceramic matrix composites are materials with ceramic fibers in a high temperature matrix of ceramic or glass-ceramic. This emerging class of materials is viewed as enabling for efficiency improvements in many energy conversion systems. The key controlling property of ceramic matrix composites is a relatively weak interface between the matrix and the fiber that aids crack deflection and fiber pullout resulting in greatly increased toughness over monolithic ceramics. It is well known that degradation of the interface will change the mechanical toughness of the material. For this study, two different sets of ceramic matrix composites fabricated via a polymer infiltration pyrolysis approach were exposed to an intermediate temperature oxidation condition at 815C for times up to 200 hours. The material was then mechanically tested in bending to failure while monitored with acoustic emission transducers. The mechanical and acoustic emission data will be reviewed and trends discussed. The effort showed strong changes in the toughness of the material while subtle changes in other properties were observed.

(ICACC-S1-P095-2017) Deposition of Silicon Carbide Intergranular Phase on Boron Carbide and its Effect on the Bulk Mechanical Properties

Q. Yang*[1]; C. Hwang[1]; A. Khan[1]; V. Domnich[1]; S. DiPietro[3]; K. Xie[2]; K. Hemker[2]; R. A. Haber[1]; 1. Rutgers University, USA; 2. Johns Hopkins University, USA; 3. Exothermics Inc, USA

Due to its attractive mechanical properties, boron carbide is widely used in the abrasives, nuclear, and armor industries. Despite its high hardness, boron carbide has relatively low fracture toughness (3-4 MPa.m$^{1/2}$) and exhibits catastrophic failure when subjected to pressure exceeding its Hugoniot elastic limit (20 GPa). One possible way to improve its fracture toughness is by introducing a second phase such as silicon carbide on the boron carbide grain boundaries. The second phase is formed by first depositing silicon dioxide and carbon mixture through sol-gel process onto the boron carbide powder. The coated powder is then sintered at high temperature and simultaneously silicon carbide is formed. The effect of having a silicon carbide phase on the intergranular boundaries will be reported and characterization results such as X-ray analysis, TEM, SEM micrograph, Raman spectroscopy, hardness, and mechanical properties will be presented.

(ICACC-S1-P097-2017) Molecular Dynamics Study on Tribochemical Reaction of Amorphous Silica in Humidity Environment

N. Takahashi*[1]; J. Chida[1]; Y. Wang[1]; J. Xu[1]; Y. Ootani[1]; T. Nishimatsu[1]; Y. Higuchi[1]; N. Ozawa[1]; M. Kubo[1]; 1. Institute for Materials Research, Japan

Friction of seismic faults is important to understand an earthquake mechanism. Thus friction of the rocks are widely studied. Silicon dioxide is one of the most abundant compounds in the rocks. It is reported that the friction coefficient of the silicon dioxide decreases as the slip velocity increases. A silica gel layer formed in the friction interface through the chemical reaction with the water is suggested to be the main cause. However the detailed mechanism is still not clear. In this work, we performed the friction simulation of the amorphous silicon dioxide (a-SiO_2) by using the molecular dynamics. The structural change in the friction interface and chemical reaction with water molecules were investigated. When the water molecules were absent, successive formation and dissociation of Si-O-Si bonds between two slabs occurred. Then, the atoms in the slabs transferred to the opposite side slabs. In the presence of the water molecules, the formation and dissociation of the Si-O-Si bond were suppressed, because the water molecules saturated dangling bonds generated on the Si and O atoms. This reaction prevented the transfer of the atoms in the two slabs and hydroxylated the surface. We also found that the water molecules penetrated in the slab.

These results indicate that the water molecule affect to the structural change in the friction interface of the a-SiO$_2$.

(ICACC-S1-P098-2017) High temperature, high velocity flame rig with damage monitoring for turbine engine materials

M. Kannan*[1]; R. Panakarajupally[1]; M. J. Presby[1]; G. N. Morscher[1];
1. The University of Akron, USA

Significant improvements are still needed in the development of materials/coatings for such applications where the environment is a mixture of high temperature, pressure and corrosive particles (eg. Salt, sand or ash). Part of that process is the development of facilities to qualitatively and quantitatively evaluate these combined effects on new material/coating approaches. A facility has been constructed that would enable the evaluation of materials of interest such as ceramic matrix composites or superalloys with environmental coatings in an environment closer to that of a jet engine. The facility comprises of a high velocity oxygen fuel (HVOF) vertical flame with an impinging velocity up to Mach 2 onto a horizontally oriented specimen thus providing good uniform local heating and the possibility for thermal gradients. The setup also includes an Aramis 3D DIC system which can be used to record the change in strain as the sample is loaded in tension and thermal imaging. Health monitoring techniques such as Acoustic Emission and Electrical Resistance are also incorporated to see the effect of temperature and environment on the properties of the material or study the life of a coating on a given sample.

(ICACC-S1-P099-2017) Structural and mechanical proprieties of calcined nano-bioceramic, alumina alpha

M. Fellah*[1]; L. Aissani[2]; 1. tribology, materiamll surface and interface group, laboratory og metallurgy and materials science, Annaba university, Algeria;
2. department of physics, abbes laghrour university, Algeria

The structural evolution and morphological changes of the nano-structured α-Al2O3 powder, formed by calcinations at different temperatures of gibbsite (300- 1400K) were reported using XR diffraction, XPS. MEB and oscilating tribometer. It is observed that the crystallite size of the particles reduced to 8 nm after calcinations at 1400 C. Morphological studies of powder particles indicated that the powder particle size continuously decreases with increasing temperature of calcinations. The sliding wear rate and wear coefficient of friction were lower in the nanocrystalline samples calcined at 1400 K at same applied load (3, 6 or 10N). The improved friction and wear resistance is attributed to the finer microstructure of the sample calcined at 1400°C.

(ICACC-S1-P101-2017) Comparison of Mechanical Strength of Reaction-bonded Silicon Carbides with Different Sample Size

S. Kim*[1]; Y. Oh[1]; S. Lee[1]; Y. Han[1]; H. Shin[2]; Y. Kim[2]; 1. Korea Institute of Ceramic Engineering and Technology (KICET), Republic of Korea;
2. Inocera Inc., Republic of Korea

Reaction-bonded silicon carbide (RBSC) represents a family of composite ceramics processed by infiltrating with molten silicon into a skeleton of SiC particles and carbon, in order to fabricate a fully dense body. RBSC has been commercially used and widely studied for many years, due to its advantages such as relatively low temperature for fabrication and easier to form components with near-net shape and high density, compared with other sintering method. In this study, RBSC is fabricated with a forming process of casting for radiant tube applications. Mechanical strengths with different size and shape are examined with the reaction-sintered samples. The relation between microstructures and physical properties is also discussed.

(ICACC-S1-P102-2017) Deformation and Damage Evolution Process of Laminated SiC/SiC Composite

Y. Kagawa*[1]; S. Arai[1]; K. Yonekura[1]; T. Takagi[2]; M. Ukai[3]; M. Uchihashi[3];
S. Suyama[3]; 1. The University of Tokyo, Japan; 2. Ibiden Co. Ltd, Japan;
3. Toshiba Corporation, Japan

Deformation and micro-damage evolution behavior of laminate structure SiC/SiC composite under tensile load, both notched and unnotched, have been studied. Laminated $(\pm\theta°)_n$ plate type composite fabricated by CVD process is used. Change of surface strain distribution during tensile loading is monitored using DIC technique. Damage evolution under low level loading is observed and effects of artificial stress concentration on the strain distribution and local damage evolution are discussed. Observed behaviors are discussed in terms of fiber architectures and laminate fiber structure. Special attention has focused on bundle unit fiber failure and micro damage evolution process of composite.

(ICACC-S1-P103-2017) The effect of SiO$_2$/Al$_2$O$_3$ ratio on properties of glass-ceramic composite materials from SiO$_2$-Al$_2$O$_3$-Na$_2$O-K$_2$O-CaO-MgO system with variable content of barium

J. Partyka*[1]; K. Pasiut[1]; M. Lesniak[2]; M. Gajek[1]; M. Sitarz[2]; J. Lis[1]; 1. AGH University of Science and Technology, Poland; 2. AGH University of Science and Technology, Poland

Glass-ceramics are polycrystalline materials of fine microstructure produced through controlled crystallization of base glass. Crystallization usually can be induced by appropriate selection of the oxide molar composition and/or firing curve and/or by the addition of crystallization nuclei(s). This publication is focused on effect of the silica to alumina ratios and content of barium oxide on microstructure and some selected properties of the glass–ceramic materials based on the system SiO2– Al2O3–Na2O–K2O–CaO-MgO. The following characterization techniques have been used: X-ray diffraction (XRD), scanning electron microscopy with micro-analyser (SEM–EDS), mid-infrared analysis (MIR), far-infrared analysis (FIR) and Raman Spectroscopy. Significant differences were observed in microstructure of silica–alumina network of glassy phase and phase composition. Discussed results are part of a larger project implemented under the PBS Applied Research Programme, in order to determine the compositions of glass–ceramic materials with potential application as a chemically resistant hard coatings or/and resistant to thermal shock or as construction materials.

(ICACC-S1-P104-2017) Thermo-mechanical properties of Metal-supported Solid Oxide Fuel Cells components

F. Teocoli*[1]; J. Nielsen[1]; Å. H. Persson[1]; K. Brodersen[1]; 1. DTU, Denmark

Metal-supported solid oxide fuel cells provide substantial enhancements compared to conventional ceramic cells, such as increased resistance against mechanical and thermal stresses and a reduction in materials cost. However, sintering of multilayer is a critical process, and microstructural changes and stresses take place, leading to shape distortion, delamination and cracks. Furthermore, the implementation of a metallic support introduces new challenges with respect to cell fabrication, since the usual sintering in an oxidizing atmosphere needs to be avoided. Half-cell tape-casted structure was studied during the thermal processing under reduced oxygen partial pressure. Different sintering kinetics of the single layers give rise to localized tensile stresses, which result in a poor densification of the electrolyte as well as a low porosity of both metal support and anode functional layer (AFL). To analyze the phenomena, a thoughtful investigation of shrinkage and viscoelastic properties of single layers under reduced oxygen partial pressure was carried out by thermo-mechanical analysis (TMA). The characterization of thermo-mechanical properties of the components can enables the prediction of microstructural stability of the cell.

(ICACC-S1-P105-2017) Spark-Plasma-Sintered Si_3N_4-SiC Nanocomposites for Self-Healing Applications

P. Jenuš[1]; L. Gruden[2]; T. Kosmac[1]; A. Kocjan*[1]; 1. Jozef Stefan Institute, Slovenia; 2. Jozef Stefan`s International Postgraduate School, Slovenia

Composites of silicon nitride (Si_3N_4) and silicon carbide (SiC) are important structural ceramic materials due to their good combination of mechanical and thermal properties. Moreover, it was shown that a self-healing ability can be achieved in Si_3N_4-SiC composites. In the present work, advanced synthesis and sintering methods were employed in order to prepare Si_3N_4-SiC nanocomposites with improved mechanical properties and self-healing. Namely, the SiC nanoparticles were embedded into the Si_3N_4 powder matrix via a polymer (Starfire) precursor route, whereas the consolidation was performed in an SPS furnace. The phase compositions of the starting materials and the sintered composites were examined with XRD, while their microstructures were analysed with FIB-SEM. The hardness and fracture toughness were determined. The self-healing properties of the composites were evaluated using TGA and SEM. The Si_3N_4-SiC nanocomposites prepared via the polymer precursor route displayed an increased sintering activity, resulting in a more homogeneous distribution of smaller, nanoscaled SiC particles in the Si_3N_4 matrix, yielding nanocomposites with higher fracture toughness and better self-healing ability, as compared to the Si_3N_4-SiC composites prepared from the commercially available Si_3N_4 and SiC powders.

(ICACC-S1-P106-2017) Origin Of "Tough Behavior" in Discontinuous Carbon Fiber Reinforced SiC Matrix Composites

K. Kajihara*[1]; Y. Atsumi[1]; Y. Kagawa[1]; 1. The university of Tokyo, Japan

Tough behavior of discontinuous carbon fiber reinforced SiC matrix composites originates from crack store mechanism. Initially formed microcracks in Si or SiC phase by thermal stress and generated microcracks during loading show completely different behavior. The former microcracks have a tendency to be arrested by carbon fiber minicomposite phase, however the later microcracks propagate and form major long cracks. These crack growth processes are dominated by the interaction between cracks and heterogeneous microstructure in the composite. These processes are explained based on the experimental evidences. Finally, discussions are made on the controlling factors for tough behavior of the composite.

(ICACC-S1-P107-2017) Mechanical properties of YSZ and porous Ni(O)-YSZ with varying Y-content

P. Khajavi*[1]; P. Hendriksen[1]; H. Frandsen[1]; K. Brodersen[1]; 1. Technical University of Denmark, Denmark

Enhancing the cell strength is essential for the robustness of solid oxide cell (SOC) technologies, especially when enlarged footprints and taller stacks are needed for large-scale installations. Yttria stabilized zirconia with different stabilizer contents (3YSZ, 8YZ) forms the structural component in most SOFC/SOEC cell designs. The tetragonal to monoclinic phase transformation that may occur during crack growth in 3YSZ at room temperature strongly improves its fracture toughness compared to fully stabilized cubic 8YSZ. For the SOC technology, however, it is necessary to consider effects of high temperature processing and operating conditions, as this will affect the tetragonal phase stability and transformability. In this work, the possible improvement of the mechanical properties of SOC supports by lowering the stabilizer amount in Y-doped zirconia was studied. Four different Y contents were considered (2, 2.3, 3 and 8) and the phase stability of nanocrystalline powders with temperature and after heat treatment at 1350°C was investigated. The results showed that a significant amount (95%) of tetragonal phase can be maintained in the 2.3Y powder even after 2h calcination at 1350°C, whereas only 28% remains in the 2Y sample. Besides the phase stability results, strength and fracture toughness of porous Ni(O)-YSZ supports prepared using the four powders both at room and high temperature will be reported.

(ICACC-S3-P108-2017) Integration of a Solid Oxide Fuel Cell with an Organic Rankine Cycle and Absorption Chiller for Dynamic Tri-Generation of Power, Hydrogen, and Cooling

M. Asghari*[1]; J. Brouwer[1]; 1. University of California Irvine, USA

It would be interesting to live in a residential complex in which power, cooling, heating and even hydrogen fuel for a Fuel Cell Electric Vehicle are supplied by a Solid Oxide Fuel Cell (SOFC) system. Although SOFC systems exhibit high electrical efficiency, in practical applications almost half of the fuel energy is converted to heat. An Organic Rankine Cycle (ORC) can be used when low temperature waste heat is available making it a candidate as a bottoming cycle for SOFC systems. Pure hydrogen gas can also be produced by the SOFC system as an energy co-product using the tri-generation concept of lower fuel utilization followed by hydrogen separation from the anode off-gas. The current work develops and applies a model to study the dynamic characteristics of an integrated novel tri-generating SOFC system with an ORC bottoming cycle and absorption chiller in Matlab/Simulink. This system is evaluated in terms of efficiency, capacity, dynamic operation and control to meet measured residential power, heating, cooling, and fuel demands. Preliminary results based on steady state conditions show a significant increase in the electrical efficiency by recovering SOFC waste heat through the ORC. The proposed system may offer a high efficiency low emissions conversion solution to overcome both fuel consumption and environmental concerns.

(ICACC-S3-P109-2017) Spray pyrolysis of Mn-Co spinels for protective coatings for interconnect protection

D. Szymczewska[2]; S. Molin*[1]; P. Jasinski[2]; M. Chen[1]; P. Hendriksen[1]; 1. Technical University of Denmark, Denmark; 2. Gdansk University of Technology, Poland

Manganese-cobalt spinels are promising materials for use as protective coatings for steel interconnects for high temperature Solid Oxide Fuel/Electrolysis stacks. Many different coating fabrication methods have been used: screen printing, slurry spray deposition, electrophoretic deposition etc, primarily fabricating ~10 μm thick coatings. It is of interest to evaluate the behavior of thinner protective layers, especially for potential lower temperature applications. In this study spray pyrolysis method is used to fabricate $MnCo_2O_4$ coatings with thickness of 2000 nm on the Crofer 22 APU steel. Spray pyrolysis is a versatile deposition method allowing fabricating dense ceramic layers at low temperatures (~400°C) without the need for high temperature sintering step, where steel can corrode and form and oxide scale. For evaluation of corrosion protective properties, $MnCo_2O_4$ coated steels are oxidized at 750°C in air atmosphere and are subsequently analyzed by x-ray diffractometry and scanning electron microscopy in order to determine oxide phase composition and thickness.

(ICACC-S3-P110-2017) Thermo-mechanical behaviour of multilayered ceramic systems for SOFCs

A. Masini*[1]; F. Šiška[1]; O. Ševeček[2]; Z. Chlup[1]; I. Dlouhý[1]; 1. Institute of Physics of Materials, AS CR, Italy; 2. Brno University of Technology, Czech Republic

Solid Oxide Fuel Cells have to withstand considerable mechanical and thermal stresses during production and especially during operation. Mechanical failure of one cell is enough to damage the whole stack, threatening the lifetime and efficiency of the entire system. Thus, it is of high importance to gain knowledge on the mechanical properties of the cell at the relevant operational conditions, improving the reliability and durability of SOFC technology. In this study, the overall behaviour of an Yttria-based electrolyte supported cell has been investigated, focusing on the interaction between layers and co-sintering effects. For this purpose, the effects of layers sintered to the electrolyte were analysed through destructive and non-destructive techniques. The layers were added to the electrolyte one by one in order to be able to distinguish the individual interactions. The relationship between Young's modulus and the presence of various combinations of layers was measured from room temperature up to a temperature lying above the service one. Results presented in this contribution show that the elastic moduli of the cell continuously decreases with the increasing number of layers. The experimental results are supported by simulations.

(ICACC-S3-P111-2017) New glass-ceramic sealants for SOEC applications

H. Javed*[1]; K. Herbrig[2]; D. Schimanke[2]; C. Walter[2]; M. Salvo[1]; F. Smeacetto[1]; 1. Politecnico di Torino, Italy; 2. Sunfire GmbH, Gasanstaltstraße 2, Germany

New glass-ceramic compositions were designed and characterised as sealant materials for solid oxide electrolysis cell (SOEC) applications, having working temperature at 850°C. The crystallization and the sintering behaviour were investigated by using differential thermal analysis (DTA) and heating stage microscopy (HSM), respectively. The thermo-mechanical and thermo-chemical compatibility of the glass-ceramic sealants with both 3YSZ and Crofer22APU substrates was examined. Glass transition temperatures (T_g) in the range of 700°C - 750°C were obtained and coefficients of thermal expansion (CTE) of 9.6 - 10.8 × 10^{-6} K^{-1} (200°C - 500°C) were measured for the obtained glass ceramics, closely matching with CTEs of other cell components. Thermal treatments at 930-950°C for 1 h in air were chosen for the joining process. The compatibility between the glass-ceramic sealants and the Crofer stainless steel (bare, pre-oxidised and electropherotically coated Mn-Co spinel) is reviewed and discussed by means of SEM and EDS analysis before and after the tests.

(ICACC-S3-P112-2017) Sintering behavior of ceria-based nanopowders

M. Biesuz*[2]; L. Spiridigliozzi[3]; G. Dell'Agli[3]; V. M. Sglavo[1]; 1. University of Trento, Italy; 2. University of Trento, Italy; 3. University of Cassino, Italy

One of the most important component of a SOFC is the electrolyte, which consists in a thin and dense ceramic ionic conductor layer. Ceria-based ceramics, doped with trivalent rare earths (Gd^{3+}, Sm^{3+}…), have been identified as optimal choice for such application, due to their high ionic conductivity in the intermediate temperature range (500-800°C). Despite these advantages, ceria-based materials present some drawbacks like the high temperature required for sintering. This limits the use of many anode materials with low melting point. So, the sintering temperature reduction of these materials represents a fundamental issue. Many works have been focused on the addition of different sintering aids to ceria although in most cases the ionic conductivity is reduced. Alternatively, attempts have been made to reduce the size of the powder. In the present work we studied the sintering behavior of nanograined ceria-based materials produced by co-precipitation method. In particular, the effect of different precipitating agents used during the synthesis on the densification process was pointed out. The produced powders were shaped by uniaxial pressing and subjected to "conventional" sintering cycles, analyzed by dilatometric tests. Also fast firing treatments were carried out for comparison. The results show that the densification properties are strongly related to the chemicals used during the precipitation step and to the sintering schedule.

(ICACC-S3-P113-2017) Mono/polycrystalline 8%mol yttria-stabilized zirconia composites for increased electrical conductivity

F. Antunes[1]; C. A. Goulart*[1]; M. Andreeta[1]; D. Souza[1]; 1. Federal University of Sao Carlos, Brazil

Solid Oxide Fuel Cells (SOFCs) are regarded as one of the enabling key technologies for clean and sustainable power generation. The state of the art consists of Yttria-Stabilized Zirconia (YSZ) as electrolyte, since this material offers great chemical stability under SOFCs operation conditions. However, its conductivity is only viable at high temperatures (T>800°C), which implies higher costs for large-scale production of SOFCs and hinders its dissemination. The objective of this work was to increase YSZ electrical conductivity in order to allow its operation in intermediate temperatures (IT) (600-800°C). YSZ composites were made by the addition of single crystal YSZ fibers obtained by Laser-Heated Pedestal Growth technique in a polycrystalline YSZ matrix. Samples were formed by isostatic pressing, sintered at 1600°C for 2 hours, and characterized by impedance spectroscopy, X-ray diffraction, scanning and transmission electron microscopy, and electron backscatter diffraction. Electrical conductivity of the composite was up to 18 times higher than regular YSZ in function of fiber volume fraction. Stresses in the matrix and distortions in the crystalline structure around the monocrystalline/polycrystalline interface were found to be the causes for the increased electrical conductivity. These results are promising and can allow the use of YSZ as electrolyte for IT-SOFCs.

(ICACC-S3-P114-2017) Strontium Nickel Oxide Nanocomposites for Chromium Capture on Solid Oxide Fuel Cell

W. A. Rodriguez*[1]; B. Hu[1]; A. Aphale[1]; C. Liang[1]; P. Singh[1]; 1. University of Connecticut, USA

Strontium nickel complex oxide (SrNiOx) for Cr-capture has been synthesized via sol-gel method using metal-nitrate salt precursors and ethylenediaminetetraacetic acid (EDTA) as chelating agent and characterized by XRD, SEM, EDS and BET. The powder was subsequently used to coat and fabricate a surface layer above the lanthanum strontium manganite (LSM) cathode of a half-cell by a number of techniques. Electrical performance of the cell was measured at 850°C with a reverse bias of 0.5V. Using electrochemical impedance spectroscopy (EIS), the polarization and current density of the half-cell was monitored for a span of 100h. Pre- and post-test XRD and SEM characterization of the coated cathode were carried to compare the composition, phase and morphology changes, as well as Cr migration and infiltration between the cathode and SrNiOx interface and surface. Pre- and post-test characterization results of the SrNiOx-coated LSM cathode will be presented. Electrochemical results show that the electrode performance degradation is mitigated in the presence of Cr vapors under the conditions tested.

(ICACC-S3-P115-2017) Development of LCFCN System Perovskites as Interconnect and Cathode Materials for Solid Oxide Fuel Cells

A. Kolisetty[1]; S. Gajjala[1]; Z. Fu*[1]; R. Koc[1]; 1. Southern Illinois University Carbondale, USA

The purpose of this research is to develop interconnect and cathode materials for use in SOFCs which demonstrates properties of high electrical conductivity, excellent chemical stability at high temperatures, desirable thermal expansion characteristics and which can be easily manufactured by sintering in conditions acceptable with other cell components. Five perovskite oxides comprising of lanthanum with chromium, iron, cobalt and nickel oxides powders $LaCr_{0.7}Co_{0.1}Fe_{0.1}Ni_{0.1}O_3$, $LaCo_{0.7}Cr_{0.1}Fe_{0.1}Ni_{0.1}O_3$, $LaFe_{0.7}Cr_{0.1}Co_{0.1}Ni_{0.1}O_3$, $LaNi_{0.7}Cr_{0.1}Co_{0.1}Fe_{0.1}O_3$, $LaCr_{0.25}Co_{0.25}Fe_{0.25}Ni_{0.25}O_3$, were synthesized through Pechini method. Obtained powders were characterized by XRD to observe crystal structure. XRD results show that all materials are single phase few with rhombohedral or orthorhombic crystal structure. The powders were then sintered at a temperature of 1400°C in air. Properties of sintered samples like relative density, mechanical properties and electrical conductivity from room temperature to 800°C were studied and evaluated. The material with the desired properties and highest electrical conductivity of 88 S/cm was then doped on the A-site with Ca at different concentrations and the properties were studied. A significant drop in the electrical conductivity was observed when the A-site was doped along with the B-site.

(ICACC-S3-P116-2017) Synthesis and Characterization of Cu-doped $Sr_2MgMoO_{6-\delta}$ for Use as Solid Oxide Fuel Cell Anode Materials

T. Chung[1]; C. Liu[1]; J. Lin[1]; H. Tseng[1]; R. Lee[1]; T. Lin*[1]; 1. Institute of Nuclear Energy Research, Taiwan

The $Sr_2Mg_{1-x}Cu_xMoO_{6-\delta}$ (Cu-doped SMMO) powders, which can act as a promising anode material for solid oxide fuel cell (SOFC), were synthesized successfully by using sol-gel thermolysis method with citric acid as the chelating agent. A high purity (>98%) Cu-doped SMMO was obtained from as-synthesized powders after calcination at 1200°C for 48 hours in flowing air. Crystalline structure, thermal expansion coefficient, and electrical resistance of Cu-doped SMMO were measured by using X-ray diffraction, thermomechanical analyzer, and AC impedance analyzer, respectively. It was found that when the value of x was larger than 0.4, the main diffraction peak (112) of Cu-doped SMMO shifted gradually toward a low angle due to the lattice distortion and the unit-cell volume increased with increasing Cu-doping level. In addition, Cu-doping enhances the electrical conductivity of SMMO. The electrical conductivity of $Sr_2Mg_{0.6}Cu_{0.4}MoO_{6-\delta}$ is 0.74 S/cm at 800°C in $H_{2(g)}$, which is 106% higher than that of Cu-free SMMO. Furthermore, the SMMO powders possess a superior structural stability. It was not found any secondary phases formed in the powders after aging at room temperature over eighteen months in vacuum. In the present study, we provided and discussed the doping mechanism for $Sr_2Mg_{0.6}Cu_{0.4}MoO_{6-\delta}$ with respect to the defect chemistry and lattice geometric structure.

(ICACC-S3-P117-2017) Effect of Ni diffusion during co-sintering process in proton-conducting solid oxide fuel cells

H. Shimada*[1]; T. Yamaguchi[1]; H. Sumi[1]; Y. Yamaguchi[1]; K. Nomura[1]; 1. National Institute of Advanced Industrial Sience and Technology (AIST), Japan

Solid oxide fuel cells using perovskite-type proton-conducting ceramics for electrolyte material were investigated, focusing on BaZr0.1Ce0.7Y0.1Yb0.1O3–δ (BZCYYb) due to its high protonic conductivity and chemical stability. BZCYYb and NiO-added BZCYYb were evaluated using electrolyte-supported and anode-supported cells. The addition of NiO, however, led to decrease in electrochemical performance. Although power density of the anode-supported cell was much higher than the electrolyte-supported cell, the OCV of the anode-supported cell was lower value. This is due to Ni diffusion from the NiO-BZCYYb anode into the BZCYYb electrolyte during high temperature co-sintering. We concluded that controlling Ni diffusion during co-sintering process is essential to achieve higher performance anode-supported cells using BZCYYb.

(ICACC-S3-P118-2017) Yttrium Strontium Titanate Based Anode Materials for Intermediate Temperature Solid Oxide Fuel Cells

S. Singh*[1]; P. A. Jha[1]; P. Singh[1]; 1. Indian Institute of Technology(BHU), India

Solid Oxide Fuel Cells (SOFCs) are one of the most attractive electrochemical energy conversion systems, as these devices present a clean energy production, thus promising high efficiencies and low environmental impact. The electrodes are the main components that decisively control the performance of a SOFC. Conventional, anode materials (like Ni-YSZ) are operates at very high temperature. Therefore, cost-effective materials which operate at relatively lower temperatures are still required. In present study, we have synthesized Y-doped Strontium Titanate via sol-gel combustion route. The structural, compositional, microstructural and density of the pellet have been investigated employing XRD, XPS, SEM and Archimedes Principle, respectively. Surface area and porosity of the compositions have been determined via BET Surface area analyzer. The electrical conductivity of the systems has been determined by impedance spectroscopy techniques. The electrical conductivity of the various mole % doped Yttrium Strontium Titanate (YST) has been found to be higher than the Ni-YSZ cermet system at 660°C.

(ICACC-S3-P119-2017) Processing of Graded SOFC Electrode Using Centrifuge Deposition

I. Bantounas[2]; S. P. Shafi[3]; U. Schwingenschlogl[4]; E. Traversa[5]; S. Boulfrad*[1]; 1. Hamad Bin Khalifa University – Qatar Foundation, Qatar; 2. Imperial College, United Kingdom; 3. Indian Institute of Science Education and Research, India; 4. King Abdullah University of Science and Technology, Saudi Arabia; 5. Xi'an Jiaotong University, China

The optimization of electrode microstructures is critical to increase both the output and life time of solid oxide fuel cells (SOFC). Many microstructure-performance simulations have predicted better performance with continuously graded electrodes, due to lower activation and concentration overpotentials. In addition, the resistance to thermal cycling is expected to be improved by this configuration. The goal of this work was to develop graded SOFC cathodes, for the first time, via centrifugal deposition. A stable LSM-YSZ suspension was prepared using a recipe derived from the usual tape casting slurry preparation routines and compositions, including organic dispersants, plasticizers and binders. After the centrifugation process, flexible, tape casting like, self-sustained films were obtained. These films were then laminated on YSZ pallets and sintered to form symmetrical cells. The cross-sectional investigation of the obtained electrodes, using electronic microscopy, showed a quasi-linear increase of the porosity from 13% in the vicinity of the electrolyte to 65% next to the air side of the electrode. In parallel, the weight fraction of Zr decreased from 54% to 43%. The polarization resistance was equal to 0.6 Ωcm^2 at 850°C, suggesting that further optimization of the particle size distribution of the starting powders might be necessary to improve the electrode performance.

(ICACC-S3-P120-2017) Performance of New Catalyst Carriers Made from Conducting Ceramics for PEM Fuel Cell

L. Li*[1]; W. Wang[1]; H. Wang[1]; J. Zhang[1]; F. Zhang[1]; 1. Wuhan University of Technology, 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.

(ICACC-S3-P121-2017) Influence of gallium oxide addition on the ionic conductivity of Sr- and Mg-doped lanthanum gallate

E. N. Muccillo*[1]; S. L. Reis[1]; 1. Energy and Nuclear Research Institute, Brazil

Lanthanum gallate doped with strontium and magnesium has been extensively studied for application in solid oxide fuel cells operating at intermediate temperatures (~500-700°C), due to its high ionic conductivity compared to the conventional yttria-stabilized zirconia solid electrolyte. Moreover, doped lanthanum gallate exhibits negligible electronic conductivity and high chemical stability over a broad range of oxygen partial pressures. Sintering of this solid electrolyte is usually carried out at high temperatures, yielding loss of gallium and consequent formation of secondary phases. In this work, the compound $La_{0.9}Sr_{0.1}Ga_{0.8}Mg_{0.2}O_{3-\delta}$ with small additions of gallium oxide (up to 1.5 mol%) was prepared by solid-state reaction and the influence of the additive on phase composition and ionic conductivity was investigated. The sintered solid electrolytes achieved densities higher than 95% of the theoretical value after sintering at 1350°C, with average grain size of about 3.1 μm. The contents of secondary phases were found to be dependent on the additive content. The total ionic conductivity reached 18.8 mS.cm^{-1} at 600°C for 1.5 mol% gallium oxide addition.

(ICACC-S3-P122-2017) Development of Ammonia-fueled Solid Oxide Fuel Cell

S. Suzuki*[1]; T. Koide[1]; H. Iwai[1]; Y. Takahashi[1]; K. Eguchi[2]; 1. Noritake Co. Limited, Japan; 2. Kyoto University, Japan

Ammonia has been studied as an energy carrier because of carbon-free and high hydrogen capacity. We have investigated performance of ammonia-fueled SOFC (Solid Oxide Fuel Cell) to study the possibility of ammonia as an energy carrier. In SOFC generation system, stack, which is made by cells, sealing glass and other materials, is main parts. These parts need ammonia resistance at 700-800 degree. So we developed sealing glass which has an ammonia resistance at 700-800 degree and fabricated 200W class stack. Ammonia was directly supplied to the anode chamber. The performance of ammonia-fueled SOFC was same as well as that of hydrogen-fueled SOFC. This work was supported by the Council for Science, Technology and Innovation (CSTI) Cross-ministerial Strategic Innovation Promotion Program (SIP) "energy carrier" (Funding agency: JST).

(ICACC-S5-P123-2017) A Novel Approach for Synthesis of Monticellite ($CaMgSiO_4$) based Bioactive Ceramic Powders Obtained from Boron Derived Waste

L. Koroglu*[1]; C. Peksen[2]; E. Ayas[1]; 1. Anadolu University, Turkey; 2. Ondokuz Mayis University, Turkey

Over the last decade, Ca-, Mg- and Si-containing bioactive ceramics have received much attention as bone graft substitutes due to high bioactivity and osteoconductivity, leading formation of a bone-like apatite layer and then strong implant-bone bond. Among the bioactive ceramics belong CaO-MgO-SiO_2 triple oxide system, monticellite ($CaMgSiO_4$) bioactive ceramics have a high usage potential for bone void filling and coating on Ti-6Al-4V alloy due to higher fracture toughness and much closer Young's modulus to that of cortical bone than hydroxyapatite (HAp) and excellent bioactivity resulting of Ca, Si and Mg ion dissolutions. Turkey has 73 % of the boron reserves in the worldwide and annualy 600 thousand tons boron derived wastes are emerged during the production of boron products. Increment of amount of waste causes waste storage problems and environmental pollution. In order to find a solution to mentioned problems, the eco-friendly and cost-effective monticellite ($CaMgSiO_4$) based bioactive ceramic powders were synthesized by thermal process using boron derived waste. Monticellite phase which is stable at 1450°C and above at normal conditions was able to obtain at too low temperature as 650°C during experiments. In addition, the bioactivity assessment of monticellite based bioactive ceramic powders was carried out by in vitro tests.

(ICACC-S5-P124-2017) Evaluation of bioactivity and hydroxyapatite formation of novel 45S5 sol-gel derived bioactive glass formulations

C. M. Goldbach*[1]; M. Demir[1]; G. Pomrink[1]; L. Howell[2]; 1. NovaBone Products, USA; 2. Particle Solutions, USA

Novel treatment methods (stepwise heating, ultraviolet (UV) light exposure, and hydrogen peroxide soaking) were previously investigated in order to produce solgel-derived 45S5 glass formulations with high surface areas (surface areas from 20 to >100m^2/g); these treatments remove organics without collapsing the pore structure. In this study, the bioactivity of these novel solgel-derived glass formulations was evaluated by determining whether or not hydroxyapatite (HAp) was present following immersion in simulated body fluid (SBF). To evaluate bioactivity, the solgel-derived 45S5 bioactive glasses were immersed in SBF for a periods of 1, 7, and 14 days to initiate hydroxyapatite formation; melt-derived 45S5 bioactive glass was used as a positive control and borosilicate spheres were used as a negative control material. SBF was prepared in accordance with ISO 23317. FTIR and SEM micrographs were used to detect HAp presence. Conclusions: Immersing novel solgel-derived 45s5 glasses in SBF resulted in HAp formation as detected using FTIR and SEM; the positive control materials exhibited HAp formation and the negative controls did not express HAp, as expected. The formation of HAp indicates that the bone void filler is a bioactive device. A next step in the study will be to further investigate the hemostatic properties of these novel 45s5 glasses.

(ICACC-S5-P125-2017) Hemostatic effects of novel 45S5 solgel-derived bioactive glass formulations

C. M. Goldbach*[1]; M. Demir[1]; G. Pomrink[1]; L. Howell[2]; 1. NovaBone Products, USA; 2. Particle Solutions, USA

Novel treatment methods (stepwise heating, ultraviolet (UV) light exposure, and hydrogen peroxide soaking) were previously investigated in order to produce solgel-derived 45S5 bioactive glass formulations with high surface areas (surface areas from 20 to >100m^2/g). In this study, the novel 45S5 formulations were evaluated in a rat kidney bleeding animal model to investigate hemostatic properties; the novel 45S5 formulations in this study have high surface areas (>80 m^2/g) and it has been shown that high surface area contributes to bioactive glasses having effective hemostatic activity. Sodium containing solgel glasses have a lower melting point, typically experiencing pore structure collapse at temperatures >500C. Because of this, solgel-derived 45S5 glasses have historically had low surface areas (0.1 to <12m2/g) while 58S solgel-derived glasses typically have surface areas >100m^2/g but the novel treatments in this study have led to solgel-derived 45S5 glasses with high surface areas. In order to investigate hemostatic activity of the novel solgel-derived 45S5 bioactive glass samples, the samples were placed in a rat kidney bleeding model. Samples previously treated with hydrogen peroxide, UV light, and/or stepwise heating treatments were compared to melt-derived 45S5 bioactive glass and a hemostatic product already on the market.

(ICACC-S5-P126-2017) Antibacterial Gold-Coated Iron Nanoparticle Drug Delivery Optimization via Increased Relative Surface Area

D. J. Banner[1]; E. Firlar[1]; R. Shahbazian-Yassar*[2]; T. Shokuhfar[1]; 1. University of Illinois at Chicago, USA; 2. University of Illinois at Chicago, USA

In gold-coated iron nanoparticle (AuFeNP) mediated drug delivery, the iron core functions as a paramagnetic material at diameters below 20nm. The gold coating serves a dual purpose: to prevent oxidation of the iron core, and as an ideal drug binding surface due to gold's easily functionalized thiol groups. Minimizing the AuFeNP diameter and increases relative drug loading as the delivered bioactive agent volume is directly determined by the total gold surface area. However, AuFeNPs below 2nm are not desired, and decreasing the diameter of the iron core compromises the AuFeNPs paramagnetic property. This study aimed to minimize the AuFeNP gold coating thickness in order to maintain the AuFeNPs paramagnetism, while increasing the total drug dose that could be delivered from a constrained volume in applications such as AuFeNP implant drug loading or an AuFeNP injection. By altering a well-established reverse emulsion protocol, AuFeNPs were synthesized with gold coatings between 1Å to 10Å with total diameters between 7nm to 20nm. Streptomycin was bound to the surface of the AuFeNPs by reduction with sodium borohydride. The AuFeNPs were analyzed by High Resolution Transmission Electron Microscopy, Selected Area Electron Diffraction in TEM, and Electron Dispersive X-Ray Spectroscopy mapping in Scanning TEM. Streptomycin induced E.Coli inhibition was determined by zone testing.

(ICACC-S5-P139-2017) Effect of Drawing Condition on Initial Strength and Molecular Orientation of β-TCP/PLA Composite Bone Fixation Screw

M. Sakaguchi*[1]; S. Kobayashi[2]; S. Ogihara[1]; 1. Tokyo University of Science, Japan; 2. Tokyo Metropolitan University, Japan

Poly(lactic acid) (PLA) attracts much attention as material of bone fixation device which is used for fracture treatment, because it degrades to nontoxic lactic acid through non-enzymatic hydrolytic degradation. However, the material has drawbacks, such as lower elastic modulus and no osteoconductivity. Therefore, composite material of poly(lactic acid) (PLA) and bioactive ceramics powder which has higher stiffness and osteoconductivity has investigated. Composite bone fixation device with hydroxyapatite (HA) which is included in bone has been developed. However, degradation ratio of HA/PLA composite is too slow. It was reported that this composite remained for 5 years in vivo. The strength in 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 and β-TCP/PLA composite was drawn to improve the strength. Past studies about self-reinforce by drawing has been conducted to polymer. In this study, effect of ceramic particles on mechanical properties and higher order structure of β-TCP/PLA composite was investigated. Screw was focused as the most general bone fixation device and formed from drawn β-TCP/PLA composite. The shear strength and orientation function were measured as mechanical properties and higher order structure, respectively.

(ICACC-S6-P128-2017) Thermal stability and brazing of thermoelectric tellurides

D. Ben Ayoun*[2]; Y. Sadia[1]; Y. Gelbstein[1]; 1. Ben-Gurion University of the Negev, Israel; 2. Ben-Gurion University of the Negev, Israel

One of the biggest obstacles in generating a working thermoelectric generator is turning materials into functional devices. Challenges that must be overcome include thermal stability at the material level and reliable contacts at the device level. In this research we extend from basic materials research, toward testing and improving some of the challenges to broad applications. For approaching practical thermoelectric power generation devices, highly reliable telluride materials were implemented. Thermal cycling tests were conducted in order to evaluate their thermal stability in long term usage. Brazing compositions based on Mn-Sn and Ag-Cu-In were considered for bonding of thermoelectric material into Co and Fe bridges. The final goal of the research is achieving a generator prototype which will work in a lab testing facility.

(ICACC-S6-P129-2017) Dielectric, Structural and Spectroscopic Properties of $Na_xBi_{(2-x)/3}Cu_3Ti_4O_{12}$ Ceramics by Solid State Reaction Method

E. Izci*[1]; 1. Anadolu University, Turkey

$Na_xBi_{(2-x)/3}Cu_3Ti_4O_{12}$ () samples were prepared by conventional solid-state reaction method. The influences of Na^+ substitution for Bi^{3+} on phase structure, microstructure, and dielectric properties were investigated using X-ray diffraction (XRD), Raman spectroscopy, impedance spectrometer. X-ray diffractions reveal that phase-pure samples can be synthesized by this method with no impurity is found. In the Raman spectra of x=0 sample, the first peak at 110 cm^{-1} was attributed to the Bi– O vibrations. The second peak at 276 cm^{-1} is associated with the vibration of the TiO_6 group and is observed in many perovskite materials. The peaks in the 400- 550 cm^{-1} region are contributed to the Cu- O stretching in CuO_4 tetrahedra and the peaks in the 550- 700 cm^{-1} region are ascribable to the Ti (Cu)-O stretching in TiO_6 or CuO_6) octahedral. In the Raman spectrum of x=0.52, x= 0,76, and x= 0.96 samples, a new peak at about 771 $cm^{-1}$is shown. This peak appears from x = 0.52 with weak intensity and then increases in intensity to x= 0.76, and x= 0.96. It was found that the low-frequency dielectric constant increased with the increase of Na^+ content. $Na_xBi_{(2-x)/3}Cu_3Ti_4O_{12}$ sample with x = 0.30 showed the lowest dielectric loss (0.018) at 1 kHz with a relatively high dielectric permittivity value (7330) among the prepared samples.

(ICACC-S6-P130-2017) Sintering Characteristics and Dielectric Properties of $Na_{1/2}Bi_{1/2}Cu_3Ti_4O_{12}$ Ceramics by Solid State Reaction Method

E. Izci*[1]; 1. Anadolu University, Turkey

$Na_{1/2}Bi_{1/2}Cu_3Ti_4O_{12}$ samples were prepared by conventional solid-state reaction method, which were sintered at temperatures of 880°C, 920°C, 960°C and 1000°C for 20 hours. The samples were characterized using XRD, and influence of sintering temperature was shown to affect the dielectric properties. As is shown in the XRD patterns, weak impurity peaks from CuO phase were identified for sample sintered at 860°C; however, no impurity phases are detected for samples sintered at higher temperatures (Ts≥920°C). In addition, with the increase of sintering temperature, there is a gradual intensity increasing and narrowing of the diffraction peaks, indicative of better crystallization and increase of crystalline size. For the sample sintered at 880°C, it exhibits the largest dielectric constant at 100 Hz and the smallest value in the frequency range of 10^4–10^6 Hz among the samples. Besides, with the sintering temperature increases, the dielectric constant value increases in the frequency range of 10^4–10^6 Hz and the tanδ decreases in the whole frequency range. In addition to the samples sintered at higher temperatures (Ts≥920°C) show a flatter dielectric constant curve related to frequency.

(ICACC-S6-P131-2017) Effect of graphene oxide as reinforcement phase in LATP

M. C. Ramírez*[1]; R. Kumar[1]; B. W. Sheldon[1]; 1. Brown University, USA

Improving the mechanical properties of ceramic solid electrolytes in Li ion batteries is an important goal for enhancing performance and extending their life, particularly in rechargeables where repeated charge/discharge cycles are needed. Graphene oxide and reduced graphene oxide have been successfully used as reinforcements in a variety of polymers and engineering ceramics, with important effects on elastic modulus and toughness with relatively low additions. Extending this to modify mechanical properties of ceramic electrolytes, though attractive, is challenging due to the risks of reducing ionic conductivity and/or increasing electrical conductivity. In the present work we explore the reinforcement capability of small amounts of graphene oxide added to LATP electrolytes that were consolidated with spark plasma sintering. We have combined the techniques of focused ion beam (FIB) machined microcantilever bending tests, and electrochemical impedance to obtain information on the mechanical response and the ionic conductivity of these composites.

(ICACC-S13-P132-2017) Damage monitoring of silicon carbide composites by electrical and acoustic methods

T. Nozawa*[1]; H. Tanigawa[1]; 1. National Institutes for Quantum and Radiological Science and Technology, Japan

A silicon carbide fiber reinforced silicon carbide matrix (SiC/SiC) composite has been developed as a candidate structural material for various types of nuclear components such as a functional material for fusion DEMO blanket and an accident tolerant fuel cladding for light water reactor. To ensure its structural performance throughout its operation, developing damage monitoring techniques by non-destructive and destructive methods should be more emphasized. This study therefore aims to identify the effects of macro/micro damages, e.g., matrix cracking, micro-cracking at the fiber/matrix interface, fiber break, etc., on electrical properties of the composites to develop the damage monitoring techniques. Various experimental techniques such as DC resistivity measurement and AC impedance measurement will be addressed as a primary option. For comparison, acoustic emission as well as microstructural observation will be utilized to discuss detailed damage accumulation process.

(ICACC-S13-P133-2017) In-situ observation of SiC oxidation under environmentally-controlled electron irradiation

N. Hashimoto*[1]; H. Nagakura[1]; F. Kano[2]; K. Okonogi[2]; 1. Hokkaido University, Japan; 2. Toshiba Corporation, Japan

Silicon carbide (SiC) ceramic has been developed as one of promising material for accident- t-tolerant-fuel (ATF) due to less active characteristics in the presence of high-temperature H_2O. SiC has also a smaller neutron absorption cross-section, which is advantageous feature in terms of neutron economy. In this study, electron beam irradiation test was conducted by utilizing a high voltage transmission electron microscope with an environmentally-controlled specimen holder in order to observe oxidation process of SiC under irradiation. Analysis of chemical composition by soft X-ray emission spectroscopy (SXES) exhibited the formation of SiO_2 in the area of irradiated surface, especially in higher oxygen pressure condition. Relationship between irradiation induced defect cluster and SiO_2 will be discussed.

(ICACC-S13-P134-2017) Characterization of compatibility between U-Zr melts containing RE elements and coating layer of the graphite and ceramic crucible

S. Oh*[1]; 1. Korea Atomic Energy Research Institute, Republic of Korea

Compatibility between various coating layers on graphite pellets and Uranium-Zirconium alloys containing RE elements were investigated via induction heating furnace. A large issue with Y2O3 coating is the adhesion between the graphite and coating layers. Due to the coefficient of thermal expansion (CTE) mismatch of the components the coatings often lack mechanical integrity. KAERI investigated using Nb, Yttria Stabilized Zirconia (YSZ), TaC, and porous Y2O3 as intermediate or adhesion layers against either Y2O3 or Y2O3 + YSZ. In addition solid YSZ and MgO stabilized ZrO2 (MSZ) ceramic crucibles were investigated. Thermal cycling tests were first conducted heating to 1600°C at 40°/min for 30 minute. Each coating layer was excessively cracked or shattered after thermal cycling tests. In the case of the solid ceramic crucibles, the Y2O3 crucible cracked but showed no reaction while the MSZ showed substantial reaction.

Thursday, January 26, 2017

3rd Pacific Rim Engineering Ceramics Summit

Challenges and Opportunities II

Room: Coquina Salon C
Session Chairs: Joerg Adler, Fraunhofer Institute for Ceramic Technologies and Systems; Manabu Fukushima, National Institute of Advanced Industrial Science and Technology (AIST)

8:30 AM

(ICACC-PACRIM-033-2017) Porous LPS-SiC ceramics: Variability and applicability of a versatile material (Invited)

J. Adler*[1]; H. Heymer[1]; U. Petasch[1]; 1. Fraunhofer Institute for Ceramic Technologies and Systems, Germany

Porous silicon carbide ceramics have been state-of-the-art for applications in hot gas (dust) filtration, diesel particulate filters (DPF) and recently in liquid filtration of processed water, but in refractories and kiln furniture as well. In detail, very different types of porous SiC ceramics are known, which are made by evaporation/sublimation, sintering, reaction bonding or by bonding SiC particles with secondary phases like crystalline or glassy silicates. But besides RSiC ("Recrystallised" SiC), there have not been any common standards of porous SiC materials yet. Usually, the term "LPS-SiC" refers to a special kind of sintering SiC using additives in the system Al-RE-(Si)-Oxides. LPS-SiC is mostly known as structural ceramics with high density, strength and fracture toughness. But LPS additives and sintering conditions can be used favorably to design porous SiC ceramics, too. Starting with a short overview about the different types of porous ceramics made of silicon carbide and their properties, the presentation describes the preparation and properties of some porous LPS-SiC ceramics. Examples of applications in exhaust treatment, liquid filtration or as diaphragm in light metal melts explains the versatility of this material.

9:00 AM

(ICACC-PACRIM-034-2017) Processing methodologies of porous ceramics with tailored pore configurations (Invited)

M. Fukushima*[1]; A. Shimamura[1]; Y. Yoshizawa[1]; M. Hotta[1]; T. Ohji[1]; N. Kondo[1]; 1. National Institute of Advanced Industrial Science and Technology (AIST), Japan

This presentation reviews recent our processing strategies for macroporous ceramic components and different ways for achieving control of their pore morphology at different length scales. Two advanced processing methodologies to produce precisely controlled pore configuration have been discussed in terms of: direct blowing of a phenoric resin included in powder compacts, to form an array of interconnected spherical pores with various unique surfaces comprised of relatively denser and/or zebra-type (denser and porous) layer created by using one-pot blowing process, and gelation-freezing to provide elongated anisotropic and/or completely closed pore channels by templating ice crystals in gel bodies. These simple and versatile approaches can be used to tailor pore configurations, and fabricate macroporous monoliths with various distinct characteristics.

9:30 AM

(ICACC-PACRIM-035-2017) Current Status and Prospects of Ceramic Membrane for Water Treatment (Invited)

I. Song*[1]; J. Ha[1]; J. Lee[1]; 1. Korea Institute of Materials Science, Republic of Korea

Ceramic membranes can be applied under extreme operating conditions such as low pH, high pressure and high temperature. Especially, the inherent limitations of conventional polymer membranes can be overcome by adopting ceramic membranes simply to the existing water treatment systems. Moreover, there are a lot of potential applications of ceramic membranes such as distillation, adsorption and extraction in various industrial areas. Therefore, the ceramic membrane technology is not a mere ceramic processing technology, but a highly influential technology to the overall environment technology. In this study, we focused the current status and prospects of ceramic membrane for water treatment by adopting various kinds of ceramic materials such as alumina, silicon carbide and natural materials. Especially, the ceramic membrane of flat-sheet type fabricated by extrusion process was introduced in various applications such as membrane bioreactor (MBR), industrial wastewater, and pretreatment in desalination. This presentation consists of three parts: (1) Tailoring pore structures of the substrate of ceramic membrane, (2) Functionalizing the coating layer of ceramic membrane, and (3) Extruding the substrate of ceramic membrane.

10:20 AM

(ICACC-PACRIM-036-2017) Influence of carbon on mechanical and tribological properties of metal-ceramics friction materials (Invited)

J. Yang*[1]; H. Guo[1]; B. Wang[1]; 1. Xi'an Jiaotong University, China

In this study, the influence of carbon contents on Cu-Fe matrix composites containing flake graphite (CFC-FG) and mesocarbon microbead (CFC-MM) are fabricated using vacuum hot-pressing sintering. Physical and mechanical properties are analyzed and tribological behavior based on a ball-on-disk tribometer s studied for varying the extent of doping (5–25 vol. %). The results indicate that all the samples can be densified, and microstructures of all the composites reveal that the carbons are uniformly dispersed in the samples. The hardness and flexural strength decrease with increasing carbon content from 0 to 25 vol. %, and that of the CFC-MM are higher than the CFC-FG, due to the diffusion of carbon into Fe and the isotactic morphology of the mesocarbon microbead (MCMB). The tribological performance of CFC-MM is excellent for the lower friction coefficient and less wear rate because of the formation of solid lubricant-rich films on the surface of friction materials and isotactic morphology of the MCMB. The observed mechanical and wear properties have been correlated with microstructures and worn micrographs. MCMB can stabilize friction and wear properties of Cu-Fe matrix composites.

10:50 AM

(ICACC-PACRIM-037-2017) Efforts of Improving Fracture Behavior in Flexible Inorganic Thin Films with Modulated Electrical and Optical Properties (Invited)

S. Lee[1]; H. Choi[1]; H. Choi[1]; Y. Cho*[1]; 1. Yonsei University, Republic of Korea

Flexible thin films-based devices need to be survived with various bending operations without mechanical failure. The evolution of cracks upon bending operations is a major challenge to be overcome. Here, we report several novel ways to improve substantially the fracture behavior of films, which include (1) passivation approach with graphene and (2) in situ stress-driven processes. For example, combined with the stabilization stage, the sputtered ZnO thin films on a stretched polymer substrate demonstrate near double enhancement of crack initiation strain, which means that we can bend more without the occurrence of cracks. The critical strain of the optimal films exhibited the highest value of ~1.83 %, which is a significant increment compared to the current tolerance value of ~1 %. It further results in ~300 % improvement in fracture energy. The fracture behavior and improved mechanical behavior is understood with associated electrical, optical and photovoltaic properties under the external bending conditions for some selected examples of flexible devices.

FS1: Geopolymers, Chemically Bonded Ceramics, Eco-friendly and Sustainable Materials

Coatings and Mechanical Properties

Room: Coquina Salon E

Session Chair: Claus Rüscher, Leibniz University Hannover

8:30 AM

(ICACC-FS1-008-2017) Coating of unreactive and reactive surfaces by a geopolymer binder (Invited)

S. Rossignol[1]; L. Vidal[1]; A. Gharzouni*[1]; 1. SPCTS, France

Nowadays, a lot of applications require the assembly of different materials to improve their properties in use. This work focuses on the production of a geopolymer binder coating on metallic or agglomerated sand substrates. To do this, various sodium- or potassium-based compositions and different metakaolin reactivities and their interactions in presence of various types of substrates (metallic, organic or inorganic materials) were studied. The interactions between the binder and substrates were analysed by wetting angles measurements. The influence of the binder composition and the drying and deposition parameters was observed through coating tests conducted over tin-plated copper and bonded sand. Scanning electronic and optical microscopy observations confirm the chemical adhesion between the various components. FTIR spectroscopic analyses have also identified the parameters for obtaining a geopolymer network such as the reactive aluminium concentration (5 mol / L) and the molar Si / Al and M / Al (M = K or Na) ratios (2 and 1.2, respectively). It is therefore possible, by determining the wetting angle, to control the deposition on either a metal or silica sand.

9:00 AM

(ICACC-FS1-009-2017) Preparation of coatings and characterization of adhesion between metakaolin-based geopolymers and mild steel (Invited)

G. P. Kutyla[1]; P. Stynoski*[2]; T. Carlson[2]; C. Marsh[2]; W. M. Kriven[1]; 1. University of Illinois at Urbana-Champaign, USA; 2. US Army ERDC-CERL, USA

Materials which adhere strongly to metals have many applications, ranging from protective coatings to construction and metal-metal joints. Geopolymers may offer improvements in these areas as they tend to bond well with oxides, such as the native oxide layer on metal surfaces, but the fundamental mechanisms of adhesion between geopolymers and metals are not fully understood. We applied metakaolin-based sodium and potassium geopolymers to well-polished mild steel substrates, both as coatings and as adhesives between bar faces. Steel tensile samples bonded together using unreinforced sodium geopolymer showed bond strengths between 6 and 17 MPa, with typical failures occurring cohesively within the geopolymer. When applying geopolymer as a coating, care must be taken to prevent dehydration, as the thin material can lose water to the air rapidly, which prevents full curing. Coatings which were sealed and fully cured (7 days at room temperature) developed map cracking and spalled off within seconds of unsealing due to drying shrinkage. Mica reinforcement addition was required to toughen the coating and prevent shrinkage cracking. Considering our experimental results, geopolymers may act as effective coatings and bonding agents for steel substrates, provided adequate water control and addition of mechanical reinforcement.

9:30 AM

(ICACC-FS1-010-2017) Long-term mechanical and structural properties of metakaolin-based geopolymers (Invited)

L. M. Matulova*[1]; M. Steinerova[2]; 1. University of Chemical Technology Prague, Czech Republic; 2. Institute of Rock Structure and Mechanics, Academy of Sciences of the Czech Republic, Czech Republic

Geopolymers are alumosilicate materials that have long been studied. Despite this, little is known about the long-term stability of their mechanical and structural properties, so crucial for their successful application. To improve understanding, we investigated the effect of four different types of environments on the mechanical and structural properties of a metakaolin-based geopolymer (MK GP). The MK GP samples were stored in laboratory conditions (control samples), in water at 20°C, in water at 80°C, and outside exposed to the weather. Compressive and tensile strengths were measured after 28, 56, 90 and 360 days. In parallel, structural properties were analyzed using XRD, SEM, MAS NMR spectroscopy and mercury intrusion porosimetry. Whereas the mechanical properties of the samples in laboratory conditions and in 20°C water were stable, the mechanical properties of the other samples differed. Structural analyses were focused on changes in sample microstructure (developing microcrack network, porosity) and identifying zeolites, the presence of which would indicate detrimental processes in the structure that can change it from amorphous to crystalline. No zeolites were found during the 360-day period in MK GP samples, but the reduction in mechanical properties coincided with a developing network of microcracks and changes in pore size distribution.

10:20 AM

(ICACC-FS1-011-2017) Low Temperature Joining of Borosilicate Glass

E. Muskovin*[1]; W. Fahrenholtz[1]; J. Buckner[2]; R. Brow[1]; 1. Missouri University of Science & Technology, USA; 2. Air Force Research Lab, USA

Deep eutectic solvents (DESs) were used to join borosilicate glass at temperatures below 200°C. DESs are non-aqueous ionic liquids comprising of a hydrogen bond donor and a halide salt; the mixture of the two constituents forms an ionic solvent near or below room temperature. Due to the DESs ionic character, metal salts exhibit high solubility within the solvent, which can be exploited to form bonds with high strength that are stable at high temperatures. Solidification of the eutectic solvent occurred at temperatures below 200°C in inert environments. The chemical reaction of the joining process resulted in a solid layer with a higher melting temperature than the original eutectic solvent (60°C). The joining layer was examined using thin film x-ray diffraction, x-ray photoelectron spectroscopy, scanning electron microscopy and energy dispersive spectroscopy.

10:40 AM

(ICACC-FS1-012-2017) Fracture Behavior of Geopolymer Concretes at the Microscopic Length-scale (Invited)

J. Chen[1]; C. Johnson[1]; A. Akono*[1]; 1. University of Illinois at Urbana-Champaign, USA

Due to their extraordinary strength-to-weight ratio and low carbon footprint, geo-polymer concretes represent environment-friendly construction material alternatives. However, they have been confined to niche applications, partly due to the lack of advanced mechanical characterization methods that would facilitate their insertion into building codes. An open question is how to assess the fracture properties directly at the level of individual micro-constituents. Thus, the research objective is to measure the fracture energy of geopolymer concretes at the micron level via scratch testing. Commonly employed for quality control of thin films and hard coatings, the scratch test consists in pushing a diamond stylus across the surface of a polished specimen under a linearly increasing vertical force. We devised a rigorous specimen preparation routine so as to yield a low surface roughness, which is essential for microstructural observation and high-accuracy testing. We carried out microscopic scratch tests on metakaolin-based geopolymer/granite composites, investigating the resistance in the matrix, the reinforcement and at the interface. Newly scratch-induced fracture surfaces were observed. The fracture toughness was calculated using nonlinear fracture mechanics modeling. In sum, a novel experimental method was formulated for fracture assessment based on scratch tests that can be scaled to smaller levels.

Sustainable Materials and Novel Applications I

Room: Coquina Salon E
Session Chair: Henry Colorado, Universidad de Antioquia

11:00 AM

(ICACC-FS1-013-2017) Geopolymer formation versus zeolite crystallisation (Invited)

C. Rüscher*[1]; 1. Leibniz University Hannover, Germany

Very early investigations of synthesis routes of zeolite X and Y are reviewed in comparison to some more recent efforts to increase the size of single crystals of zeolite Y. Within this routes the formation of geopolymer structural units could well be detected. The contribution and formation of geopolymer fragments as a possible glue between zeolite nanocrystals and as special water resevoirs and conducting path in NaBH4-sodalites for hydrogen release is discussed.

11:30 AM

(ICACC-FS1-014-2017) Silver Ion Exchange Capacity and Release Kinetics of Geopolymer-Based Nanostructured Aluminosilicates for Antimicrobial Applications (Invited)

S. Chen*[1]; J. Popovich[2]; S. Haydel[2]; D. Seo[1]; 1. Arizona State University, USA; 2. Arizona State University, USA

Due to the presence of labile alkali metal ions in the structure, geopolymers have shown ion exchange properties. As the properties are governed by effective ion diffusion in the material, nanostructurization has been demonstrated as a suitable way to minimize the particle size effect on diffusion time by reducing the diffusion path length. Therefore, it is highly interesting to prepare nanostructured geopolymers with different degrees of crystallinity and to study their ion exchange properties without the diffusion limitation of bulk materials. Herein we report controlled syntheses of various geopolymer-based nanostructured aluminosilicates including amorphous geopolymer, partially zeolitic geopolymer and highly zeolitic products, as well as their characteristics from N_2 sorption, X-ray diffraction, FT-IR and SEM/TEM studies. We will also present the silver ion exchange capacity and ion release kinetics of the materials which are relevant to antimicrobial application of the materials. Superb ion release kinetics and antibacterial efficacy on MRSA (methicillin-resistant Staphylococcus aureus) have been observed for the highly zeolitic material. Together with the submicron sizes of the aggregates, the materials' high efficacy may allow production of antimicrobial polymer-geopolymer nanocomposites such as medical plastics, textile and apparel.

FS2: Advanced Ceramic Materials and Processing for Photonics and Energy

Advanced Applications

Room: Halifax A/B
Session Chairs: Safa Kasap, University of Saskatchewan;
Mauro Epifani, CNR-IMM

8:30 AM

(ICACC-FS2- 033- 2017) Electronic Band Structure in Semiconductor Quantum Dots: A Scanning Probe Microscopy Study

D. Benetti*[1]; H. Zhao[1]; A. Vomiero[2]; F. Rosei[1]; 1. Institut national de la recherche scientifique, Canada; 2. Luleå University of Technology, LTU, Sweden

Nanostructured materials have been among the most promising objects in the past two decades due to their unique properties. One of the most interesting nanostructures is the family of quantum dots (QDs). They are semiconducting nanocrystals with size ranging from 1 to 20 nm, in which quantum confinement of the electronic band structures occurs, due to their limited dimensions. In many applications, the QDs are linked to semiconducting metal oxides, e.g. TiO_2, ZnO, etc. to form composite systems with peculiar optoelectronic properties. Critical applications based on such heterostructures are LEDs, photo-catalysis, bio-probes and solar cells. In this work, we investigate the electrical proprieties of the QDs material that forerun their integration in a metal oxide. We study composite QDs formed by lead and cadmium chalcogenides directly grafted on highly oriented pyrolytic graphite (HOPG) surface through the successive ionic layer absorption and reaction (SILAR) technique. We apply Atomic Force Microscopy (AFM) and Kelvin Probe Force Microscopy (KPFM) to investigate SILAR-produced pure PbS, pure CdS and PbS/CdS composite systems. We were able to follow for the first time the variation of the contact potential distribution (CPD) of the different QDs via KPFM, investigating the evolution of their electronic band structure as a function of QD size and composition.

8:50 AM

(ICACC-FS2- 034- 2017) Comprehensive structural study on Nd^{3+}/Yb^{3+} co-doped La_2O_3-TiO_2-ZrO_2 glass-ceramics

M. Zhang*[1]; 1. Shanghai Institute of Ceramics, China

Nd^{3+}/Yb^{3+} co-doped La_2O_3-TiO_2-ZrO_2 glasses have been prepared by aerodynamic levitation method. The glasses show high refractive index of 2.28 and Abbe number of 18.3. Glass-ceramics heated at 880°C for 50 min perform the strongest upconversion luminescence. XRD patterns of glass-ceramics with different depths indicate that rare earth ions restrain the crystallization. Body crystallization mechanism is confirmed in the heat treatment. Surface crystals achieve the priority to grow, resulting in important effects on upconversion luminescence. Crystal particles with uniform size distribute densely and homogenously on the surface and large amount of glass matrix exists in the glass ceramics heated at 880°C for 50 min by AFM and SEM. SAXS results indicate well grown crystals in glass-ceramics heated at 880°C for 50 min. Based on micro-structural study, the mechanism that upconversion luminescence can be improved by heat treatment has been revealed. The results of micro-structural analysis agree well with the spectra.

9:10 AM

(ICACC-FS2- 035- 2017) 3D printing of biomaterials (Invited)

R. Narayan*[1]; 1. NCSU, USA

Laser-based techniques have been used to create structures with microscale and nanoscale features for medical applications. As an example, two photon polymerization involves selective polymerization and hardening of photosensitive resins with a femtosecond laser. Polymerization occurs at site in the photosensitive resin where the excitation threshold of the photoinitiator is exceeded. 3d printing of structures containing sub-micrometer features is possible due to the fact that two photon absorption has a nonlinear relationship with incident light intensity. Two photon polymerization has been used to create devices out of several types of materials, such as photosensitive polymers, zirconium oxide hybrid materials, and organically-modified ceramic materials. For example, tissue engineering scaffolds, sensors, and drug delivery devices, have been prepared using two photon polymerization. In this talk, biological and functional characterization of 3d-printed structures will be considered.

9:40 AM

(ICACC-FS2- 036- 2017) Enhanced surface chemistry of metal oxide nanocrystals by inorganic surface modification (Invited)

M. Epifani*[1]; 1. CNR-IMM, Italy

The properties of nanocrystalline materials related to environmental, catalytic and energy storage applications are strongly influenced by the chemical and structural properties of the surface. Hence the surface modification of nanocrystalline structures attracts intense applicative and fundamental interest. For instance, an oxide nanocrystal surface covered with a layer of another oxide may constitute the nanosized counterpart of bulk catalytic systems. This initial concept was successfully developed for enhancing the sensing properties of anatase nanocrystals by deposition of V_2O_5 and WO_3-like species. Then, the investigation was extended to other systems. In this work the main results related to SnO_2-V_2O_5 and TiO_2-MoO_3 nanocrystals will be reviewed. First the chemical principles underlying the employed sol-gel/solvothermal colloidal synthesis will be exposed. Then, a review of the gas-sensing and photocatalytic properties of these systems will be discussed. It will be shown that deposition of MoO_3 is able of boosting the sensing of reducing gases like acetone and ethanol with respect to very inert pure anatase. As concerns the deposition of V_2O_5 onto SnO_2 nanocrystals, the photocatalytic degradation of rhodamine B showed remarkable enhancement with respect to pure SnO_2. The SnO_2-V_2O_5 nanocrystals were able to completely degrade the dye in about 1 h at 10^{-7} M concentration.

10:30 AM

(ICACC-FS2- 037- 2017) Samarium-Doped Fluoroaluminate Glasses for High Resolution, High-Dose Dosimetry in Microbeam Radiation Therapy (Invited)

F. Chicilo[4]; G. Okada[3]; A. Edgar[5]; G. Belev[2]; T. Wysokinski[2]; D. Chapman[2]; S. Kasap*[1]; 1. University of Saskatchewan, Canada; 2. Canadian Light Source, Canada; 3. Nara Institute of Science and Technology, Japan; 4. University of Saskatchewan, Canada; 5. Victoria University of Wellington, New Zealand

Microbeam radiation therapy (MRT) is a promising cancer therapy technique based on the irradiation of a tumour by high-dose narrow microbeams. The dose at the centre of a microbeam can be as high as 1000 Gy or more, while the valley dose between microbeams is 1 - 20 Gy. Certain Sm-doped glasses and glass-ceramics show a Sm^{3+} to Sm^{2+} conversion induced by x-ray irradiation, which serves as a measure of dose delivered. The two ions have distinctly different photoluminescence (PL) signatures, which means that we can separate the Sm^{3+} and Sm^{2+} PL signals and hence use the PL signal from converted Sm^{2+} ions, through suitable calibration, to measure the delivered dose. Using the BMIT beamline at the CLS, we have investigated the energy dependence and the dose rate dependence of the valency conversion in Sm-doped fluoroaluminate (FA) glasses. We demonstrate that the conversion in these Sm-doped FA glasses is an excellent method for recording both high and low doses with high resolution. The present work continues to characterize this promising new material further for MRT applications by examining the dependence of the sensor responsivity on dose, dose rate and the average x-ray photon energy. We discuss the results in terms of radiation induced defects in the glass structure and the role they play in the conversion efficiency of Sm^{3+} to Sm^{2+}.

11:00 AM

(ICACC-FS2- 038- 2017) Epitaxial strain on metal-insulator transition characteristics of $SmNiO_3$ thin films (Invited)

M. Chaker*[1]; B. Torriss[1]; 1. INRS, Canada

Rare earth nickelates $RNiO_3$ display a first-order metal-insulator transition (MIT) at a critical temperature (T_{MI}). This transition is accompanied by dramatic variations of electrical resistivity, infrared and terahertz transmission, which make them interesting candidates for technological applications, such as sensors, electronic switching and thermochromic coatings. However, the synthesis of these materials remains a bottleneck to research due to their thermodynamic instability. In this presentation, we overview our recent studies on the synthesis of $SmNiO_3$ thin films by Pulsed Laser Deposition (PLD). We report the influence of epitaxial strain on the structure, electronic transport and optical properties of epitaxial $SmNiO_3$ thin films grown on $LaAlO_3$ (LAO, compressive) and $SrTiO_3$ (STO, tensile). As the strain changes from tensile to compressive, the transition temperature of the $SmNiO_3$ samples shifts to lower temperatures. The optical conductivity reveals the strong dependence of the Drude spectral weight on the strain relaxation.

11:30 AM

(ICACC-FS2- 039- 2017) Design of Materials for Energy Storage (Invited)

C. S. Ozkan*[1]; 1. University of California Riverside, USA

I will describe innovative approaches for the design and synthesis of hierarchical three dimensional hybrid materials which possess characteristics including ultra large surface area, tunability, and high conductivity which are appealing to diverse energy storage systems. Supercapacitors are promising alternative energy storage systems for applications in portable electronics and electric vehicles. Integration of pseudocapacitive metal oxides with structured nanomaterials has 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) composite systems. The RGM electrode system shows outstanding gravimetric and per-area capacitive performance, and the extended operational voltage window led to an exceptionally high energy density and power density. Next, I will talk about three-dimensional cone-shape nanocarbon composites comprising carbon nanotube clusters (CCC) decorated with amorphous silicon (SCCC) for lithium ion battery anodes. The SCCC composite system is prepared by depositing amorphous silicon onto CCC via magnetron sputtering. The seamless connection between silicon decorated CNT cones and graphene facilitates charge transfer in the system and provides a binder-free technique for fabricating lithium ion batteries.

S1: Mechanical Behavior and Performance of Ceramics & Composites

Tribological Performance

Room: Coquina Salon D
Session Chair: Anil Kumar, Indian Institute of Technology Delhi

8:30 AM

(ICACC-S1-052-2017) Tribological characterization of micro and nano size solid lubricants during grinding of silicon nitride ceramics (Invited)

A. Kumar*[1]; S. Ghosh[1]; S. Aravindan[1]; 1. Indian Institute of Technology Delhi, India

Silicon nitride ceramics are widely used as advanced structural components because of its excellent thermal and mechanical properties at ambient and elevated temperatures. However, high specific energy, high wheel-workpiece friction and the extreme hardness associated with silicon nitride cause high heat generation during grinding. The heat produced during grinding impairs the workpiece quality by inducing surface and sub-surface damages, tensile residual stresses, phase transformation etc. The damages can critically limit the applications of ground ceramic components. This article deals with the investigation of tribological properties of micro and nano solid lubricants like graphite, MoS_2 and WS_2 during grinding of silicon nitride ceramics. Experiments have been conducted to study the effect of solid lubricants on grinding forces and surface roughness during silicon nitride grinding. Results indicate that the use of solid lubricants considerably improves the process performance. The ground surface is characterized by optical microscopy, SEM and XRD.

9:00 AM

(ICACC-S1-053-2017) Tribological behaviour of CC –SiC composites with variation of Laminate orientation and surface conformity

P. Kumar*[1]; V. K. Srivastava[1]; 1. Indian Institute of Technology(BHU), India

Carbon Carbon (C C) composites have high wear rate and poor oxidation resistance which limit its application. The introduction of silicon carbide (SiC) to the matrix of CC composites leads to the application at high temperature and the improvement in tribological properties due to the chemical stability and hardness of SiC. The mechanical properties of carbon–carbon silicon carbide (CC–SiC) composites are not affected at high temperature. The tribological behaviour of CC–SiC composites had been investigated to a greater extent. But most of the investigation deals with the parallel orientation of the laminates and fully conformal surfaces. The effect of perpendicular laminate orientation and surfaces with low conformity are not investigated much. This article deals with the investigation of tribological behaviour of CC–SiC composites with different orientation of laminates and surface conformity. The results conclude that the coefficient of friction (COF) of CC–SiC composites with parallel orientation of laminates was less than COF of CC–SiC composites with perpendicular orientation of laminates at lower sliding speeds and higher at higher sliding speeds. COF for low conformity contacts was lower than that of COF for high conformity contacts. Wear rate also varied with laminate orientation and surface conformity.

9:20 AM

(ICACC-S1-054-2017) Dry Sliding Wear and Friction Behavior of Hybrid ZA-27 Alloy Composites reinforced with Silicon Carbide and Stone Dust Particulates

S. S. Owoeye*[1]; 1. The Federal Polytechnic Ado Ekiti, Nigeria, Nigeria

In this research, the dry sliding wear and friction behavior of ZA-27 alloy based composites reinforced with hybrid silicon carbide and stone dust particulates of 30μm and <38μm respectively was investigated. Stir casting process was used to produce the composites containing 8 wt% and 10 wt% SiC_p- SD particles containing 0, 25, 50, 75 and 100% stone dust respectively. The wear and friction behavior was carried out using Rotopol-V tribometer according to ASTM G99-05 to determine the wear rate, wear volume, wear resistance, wear loss and coefficient of friction of the produced composites. The results show that for 8 wt% reinforcement, hybrid composites with designation A_2 (50%SiC_p: 50%SD) displayed superior wear behavior while for 10 wt% reinforcement, hybrid composites with designation B_1 (75%SiC_p: 25% SD), B_2 (50% SiC_p: 50% SD) and B_3 (25%SiC_p: 75% SD) displayed superior wear behavior over the unreinforced alloy and single reinforced SiC_p composites. However, all the composites fall within the standard value of 0.01-0.3 coefficient of friction.

9:40 AM

(ICACC-S1-055-2017) Effect of Boron Carbide Particle Size on the Abrasive Wear Behavior of Al-B_4C Composites

A. Nieto*[1]; H. C. Yang[1]; J. M. Schoenung[2]; 1. University of California, Davis, USA; 2. University of California, Irvine, USA

The abrasive wear behavior of aluminum composites with different sizes of boron carbide (B_4C) reinforcement is investigated. The B_4C particle reinforcement sizes range from nanometric (~10-50 nm) to micrometric (~5-10 μm). B_4C particles are incorporated into an ultra-fine grained Al5083 matrix via cryogenic mechanical milling, and hot consolidated by dual mode dynamic forging. Abrasive wear performance is assessed using dry SiO_2 (sand) on a rubber wheel with an applied normal load of 133 N for 2000 revolutions, which corresponds to 1435 m of linear distance. Wear resistance is characterized by measuring mass loss during the wear test, and corroborating this to volume loss based on the known densities of the composites. The effect of B_4C particle size on abrasive wear resistance is elucidated through microhardness testing and wear track analysis. Wear tracks are characterized using scanning electron microscopy, energy dispersive spectroscopy, and X-ray diffraction.

10:20 AM

(ICACC-S1-056-2017) Low wear rate of AlN ceramics based on tribo-chemical reactions

A. Matsugami*[1]; J. Tatami[1]; M. Iijima[1]; H. Ohguni[2]; 1. Yokohama National University, Japan; 2. Sumitomo Electric Industries, Ltd., Japan

Wear behavior of material depends on the properties of the material. According to the wear map proposed by Adachi, it suggests that a threshold of the mild wear region and the severe wear region be lower if soft compounds exist on a worn surface. Moreover, the tribo-chemical reaction occurs on the surface of materials by friction or wear depending on surrounding atmosphere. For example, it has been reported that aluminum nitride, AlN, forms oxide and hydroxide layer during ball milling, resulting from tribo-chemical reactions. Therefore, in this study, we aim to achieve low wear rate of AlN ceramics based on tribo-chemical reactions. Their wear properties of AlN ceramics were measured with a ball-on-disk test in low- and high-humidity air. The intergranular fracture was found to be dominant in low-humidity air. On the other hand, the worn surface was very smooth after the test in high-humidity air because of tribo-chemical reactions, which means that the wear behavior of AlN ceramics strongly depends on the humidity. The FT-IR spectra indicated the formation of $Al(OH)_3$ and/or $NH_4Al(OH)_2CO_3$. In order to promote the tribo-chemical reaction, the ball on disk test was also performed in CO_2 flow. In this case, the wear rate was 2.6×10^{-10} mm^2/N, which was lower than that in high-humidity air. Such low wear rate resulted from the soft layer formed by tribo-chemical reactions on the worn surface.

10:40 AM

(ICACC-S1-007-2017) Compositional Analysis of State of the Art Commercial SiC Fibers

A. W. Ross*[3]; P. Mogilevsky[2]; I. Wolford[1]; T. Key[2]; M. Cinibulk[1]; 1. Air Force Research Lab, USA; 2. UES, Inc., USA; 3. SOCHE, USA

Compositional analysis was performed on both as-received and heat treated Hi-Nicalon-S, Tyranno SA3, and Cefnite SiC fibers. Heat treatments were performed in Ar at temperatures ranging from 1400°C to 1800°C. EDS analysis was performed in the TEM to assess changes in the composition of the amorphous content. In a SEM, standard based EDS analysis of polished fiber cross-sections was done to spatially resolve core to edge variations in the composition. Changes between the average composition of the fiber and changes in the composition and quantity of the amorphous content were compared.

11:00 AM

(ICACC-S1-058-2017) Layered-structure and surface-engineering design of ceramic lubricating materials

Y. Zhang*[1]; L. Hu[1]; 1. Lanzhou Institute of Chemical Physics of the Chinese Academy of Sciences, China

As a new lubricating technology, ceramic lubricating materials can realize the efficient lubrication and wear protection under special conditions such as high temperature and highly corrosive. In the present study, a design method that solves the contradiction between the mechanical and tribological properties of the traditional ceramic lubricating materials is proposed. Taking the alumina as the research object, and using the approaches of the experiment research and theoretical simulation, the studies on the relation between macro-micro structure of the prepared materials and their properties are carried out by XRD, SEM, EDS, 3D profile, universaltesting machine and friction and wear tester. The results show that the alumina composites with layered structure and surface-3D-lubricating layer have much higher bending strength, fracture toughness and reliability, also have good self-lubricating ability.

11:20 AM

(ICACC-S1-059-2017) On the Design of MAX-Polymer (MAXPOL) Multifunctional Composites

S. Ghosh[1]; M. Fuka*[1]; F. Al-Anazi[1]; S. Gupta[1]; 1. University of North Dakota, USA

$M_{n+1}AX_n$ (MAX) phases (over 60+ phases) are ternary carbides and nitrides which possesses unique properties like damage tolerance, thermal shock resistance, machinability, among other. It is also possible to fabricates unique materials by forming composites with other additives. The composites of MAX phases with polymers (MAXPOLs) are important from both fundamental and applied perspective. In this paper, we report for the first time the synthesis and characterization of novel MAXPOL composites. It is expected that these novel composites can be used for multifunctional applications.

11:40 AM

(ICACC-S1-060-2017) Hertzian Indentation Damage in TiC-316L Stainless Steel Cermets

C. Jin[1]; K. P. Plucknett*[1]; 1. Dalhousie University, Canada

Titanium carbide (TiC) based cermets are commonly used in the mining and tooling industries because of their high hardness, strength and wear resistance. The present work is focused on the Hertzian indentation damage of TiC-316L stainless steel cermets. Samples were fabricated using a vacuum melt-infiltration procedure, at 1550°C for either 60 or 240 minutes, with steel binder contents ranging from 10 to 30 vol.%. Various diameter WC-Co spheres, ranging from 1.19 mm to 3.97 mm, were used for indentation, with applied loads ranging from 250 N to 2000 N. A standard 4140-4142 grade steel was also prepared and tested for comparison. Indentation stress-strain curves were plotted to study the effects of differences in the cermet microstructure and the loading condition. 'Ideal' Hertzian elastic responses were also calculated, to demonstrate the deviation of the curves. It is shown that the materials perform in a 'quasi-plastic' manner, with evidence of 'strain-hardening' phenomena.

S3: 14th International Symposium on Solid Oxide Fuel Cells (SOFC): Materials, Science and Technology

Sealing Materials

Room: Crystal
Session Chair: Federico Smeacetto, Politecnico di Torino

8:30 AM

(ICACC-S3-044-2017) Tensile strength tests for the investigation of adhesion effects between glass-ceramics and interconnect steels used for SOFCs (Invited)

S. M. Gross-Barsnick*[1]; D. Federmann[1]; C. Babelot[1]; U. Pabst[1]; R. Peters[2]; 1. Forschungszentrum Juelich, Germany; 2. Forschungszentrum Juelich, Germany

Glass-ceramic sealants are commonly used as joining materials for planar solid oxide fuel cell stacks. Several requirements need to be fulfilled by these materials. Besides the electrical insulation and adequate thermal expansion, a good adhesion on the ceramic and metallic components of a stack is necessary to form a gas-tight joint. A key requirement is the mechanical strength of the sealant to maintain the integrity of the stacks under operation conditions. Due to the lack of standardized methods, tensile tests were developed in-house. Computer simulations using FEA have been performed in order to improve the characterization methods of mechanical strength tests. Nowadays, new stack designs are necessary for optimal power output and cheaper materials need to be considered to increase the cost efficiency. Preliminary results have shown that the interaction between interconnect and glass sealant can differ for different hot-rolled steel coils. The goal of our recent study is to analyze the adhesion effects of different interconnect sheets. Beside of the composition the thickness of investigated metal layers are varied in an approved experimental setup for tensile tests. SEM analysis was carried out on cross sections, which allow evaluating the microstructure of the composite and its interfacial reactions.

9:00 AM

(ICACC-S3-045-2017) New glass-ceramic sealants for SOFCs application: Characterization and compatibility with interconnects

A. Sabato*[1]; M. Salvo[1]; M. Kusnezoff[2]; J. C. Schilm[2]; A. Rost[2]; A. Chrysanthou[3]; G. Cempura[4]; M. Ferraris[1]; F. Smeacetto[1]; 1. Politecnico di Torino, Italy; 2. Fraunhofer Institute for Ceramic Technologies and Systems, Germany; 3. University of Hertfordshire, United Kingdom; 4. AGH University of Science and Technology, Poland

New barium-free glass-ceramic sealants were designed, characterised and tested in SOFC relevant conditions. The crystallization and the sintering behaviour were investigated using Differential Thermal Analysis (DTA) and Heating Stage Microscopy (HSM). On the basis of these analyses, a temperature of 850°C was selected to provide an optimum joining termal threatment. Thermal analyses were carried out in order to evaluate the activation energy for crystallization. Dilatometry (DIL) and X-ray diffraction (XRD) analyses were conducted for the glass-ceramic composition, following the joining process and after aging at 800°C in air for various periods of time. The sealing behavior of the glass-ceramics on bare, pre-oxidised and Mn-Co spinel-based coated stainless steels have been examined by morphological and chemical analyses. Joined samples were also submitted to dual atmosphere tests (air and Ar/H_2) with and without the effect of electrical potential. Leakage tests were performed before and after the dual atmosphere tests. SEM-EDS post-mortem analyses were carried out to understand the thermal behaviour and to investigate the degradation processes at the glass-ceramic/steel interface. The compatibility between the glass-ceramic sealants and the stainless steel (bare, pre-oxidised and coated) following tests in air and dual atmosphere is reviewed and discussed.

9:20 AM

(ICACC-S3-046-2017) Development of protective coatings for SOFC/SOEC interconnects at DTU Energy (Invited)

S. Molin*[1]; M. Chen[1]; P. Hendriksen[1]; 1. Technical University of Denmark, Denmark

High temperature corrosion of stainless steel interconnects (IC) in Solid Oxide Fuel Cell and Electrolysis Cell stacks is one of the major degradation cause during their long term operation. Protective coatings for interconnects are thus required to hinder high temperature corrosion and possible evaporation of reactive chromium species from steel interconnects. At DTU Energy, different protective coating solutions are evaluated both for the hydrogen and the oxygen side. In this presentation recent results will be presented. For the oxygen side of the IC, dual layer coatings based on reactive element and spinel layers are being developed. For the hydrogen side, reactive element coatings are studied. The project ForskEL 2015-1-12276 “Towards solid oxide electrolysis plants in 2020” funded by Energinet.dk is gratefully acknowledged.

Protective Coatings

Room: Crystal
Session Chair: Anil Virkar, University of Utah

10:10 AM

(ICACC-S3-047-2017) Mn-Co spinel protective coatings deposited by electrophoretic deposition for solid oxide cell interconnects

F. Smeacetto*[1]; S. Molin[2]; A. Sabato[1]; M. Salvo[1]; A. Chrysanthou[3]; A. R. Boccaccini[4]; 1. Politecnico di Torino, Italy; 2. Technical University of Denmark, Denmark; 3. University of Hertfordshire, United Kingdom; 4. University of Erlangen-Nuremberg, Germany

$(Mn,Co)_3O_4$ spinel is considered as one of the best materials to limit chromium evaporation on the oxygen side of the interconnect in solid oxide cells stacks. This coating has been widely studied and it has been demonstrated to be highly effective in reducing corrosion. In this study, electrophoretic deposition was successfully used for the preparation and deposition of the Mn-Co based coatings on Crofer22APU substrate, with an optimized thickness around 10 μm. New suspensions with different amounts of Cu-doped MnCo spinels were optimized to further improve the MnCo-based coating properties. The effect of Cu doping on the MnCo spinel densification, electrical behavior and the correlation with the preparation method and the protective properties is reviewed and discussed. The Crofer22APU-MnCo Cu-doped EPD coated samples were electrically tested for 5000 hours at 800°C under a current load to determine their area specific resistance. Oxidation kinetics were also evaluated after periods of 1000 hrs. Post-mortem samples were morphologically analyzed by SEM and EDS, to determine the oxide scale thickness and examine for evidence of chromium diffusion and possible new phase formation at the interface.

10:30 AM

(ICACC-S3-048-2017) Characteristics of protective spinel manganese cobaltite coatings produced by APS for Cr-contained SOFC interconnects

C. Chang*[1]; 1. Institute of Nuclear Energy Research, Taiwan

The chromium-contained ferritic stainless steels are widely employed as metallic interconnects in intermediated temperature solid oxide fuel cells. However, the chromium content of these steels would cause obvious degradation phenomena such as the growth of Cr_2O_3, chromium poisoning and delamination of oxide scales. Due to the high electrical conductivity of about 60 S/cm at 800°C in air, the manganese cobaltite with spinel structures are employed as protective coatings on Cr-contained steel interconnects. In this study, Ce-doped manganese cobaltite (CMC) protective coatings are produced by the promising atmospheric plasma spraying technique on the pre-oxidized Crofer22H, Crofer22APU and SS441 substrates. The obtained CMC layers reveal relatively dense microstructure due to the employed process parameters. The ASR values of the coated interconnects were measured by a four-probe dc technique at 800°C in air. After about 4,343 hours ageing at 800°C in air, the initial and final ASR values of the coated Crofer22APU sample with pre-oxidation treatment are 1.50 and 1.86 mWcm^2, respectively. The smallest increasing rate of ASR in this study is only about 0.08 μWcm^2/hr. XRD analysis was adopted to identify the crystal structures of the obtained coatings. The morphology and cross-sectional observations of the coatings were characterized by SEM equipped with EDS.

10:50 AM

(ICACC-S3-049-2017) Study of Cr-Gettering Material with LSCF-based Cell in a Generic Stack Test Fixture: Cell Performance and Microstructure

Y. Chou*[1]; J. Choi[1]; J. W. Stevenson[1]; C. Liang[2]; B. Hu[2]; W. A. Rodriguez[2]; A. Aphale[2]; P. Singh[2]; 1. Pacific Northwest National Lab, USA; 2. U Connecticut, USA

Cr poisoning is proven the leading cause for solid oxide fuel cell degradation. Mitigation was vigorously sought by protective coating as aluminization on perimeter areas as well as conducting coating of Mn-Co spinel on active area of metallic candidate materials and has been successfully demonstrated in short and medium term of operations. Nevertheless, there is the need for Cr mitigation should the coatings start to fail in long-term operation. PNNL has teamed up with U. Conn. to evaluate a novel Cr-getter material developed at the university. Two areas will be tested: one at air inlet and one on cell in a PNNL’s generic stack test fixture using commercial 2”x2” cells at 800°C. Commercial LSCF-based YSZ/NiO anode-supported YSZ cell will be used with standard sealing system and diluted fuel (H_2:N_2=1:1 + ~5% H_2O) and humidified air. Cr sources will be provided using pre-oxidized AISI 441 metal stripes. Two forms of Cr-getter will be evaluated: one by impregnated ceramic foam provided by U. Conn. and one by in-house solid-state reaction made pellets. For comparison, plain cell with and without Cr sources will also be tested for baseline data. All cells will be tested at constant current mode for ~1000h. At the end cell electrochemical performance and microstructure analysis will be conducted to assess the effect of inlet and on-cell Cr gettering.

11:10 AM

(ICACC-S3-050-2017) Influence of temperature and steam content on degradation of metallic interconnects in reducing atmosphere

C. Folgner*[1]; V. Sauchuk[1]; M. Kusnezoff[1]; A. Michaelis[2]; 1. Fraunhofer IKTS, Germany; 2. Fraunhofer IKTS, Germany

Three ferritic steels (Crofer 22 APU,Crofer 22 H,AISI441) were tested in SOFC anode gas environment in a temperature range between 725–875°C. The experiments were carried out under variation of water vapor content in the gas mixture for different exposure times in order to create accelerated degradation testing conditions for metallic interconnectors (MICs) and to investigate the behavior of these materials caused by the formation of growing chromium oxide based scales. Both gravimetric measurements and FESEM/EDX data was analyzed to characterize the oxidation kinetics and the microstructure of the oxide scales. A clear correlation between increasing temperatures and increasing oxide growth rate constants k_w can be demonstrated in all materials. This interrelation results in thicker surface oxide scales. The structures of the oxide layers are specific for each material and consist of Cr_2O_3 and $(Cr,Mn)_3O_4$

with different element distribution and thickness ratio. Beside this, the zone of an inner oxidation with Al-,Si- and Ti-rich oxide inclusions can be seen in ferritic samples, whose microstructures differ depending on analyzed materials and temperatures. Electrical measurements in dual gas atmosphere reveal also an increase of resistance within 1000 h material exposure. The results of oxide growth in tested samples are compared with data derived from real stacks.

11:30 AM

(ICACC-S3-051-2017) Oxidation Resistance of Nickel Coated SS430 steel

M. K. King*[1]; M. K. Mahapatra[1]; P. Singh[2]; 1. University of Alabama at Birmingham, USA; 2. University of Connecticut, USA

Corrosion and chromium evaporation resistant and electrically conductive coating is required for metallic cell to cell interconnect used in high temperature electrochemical systems such as solid oxide fuel cells and solid oxide electrolysis cells. Nickel coating has been deposited on SS430 steel substrates by electroless and electroplating methods. The nickel coated SS430 substrates were subjected to oxidation in oxidizing and reducing atmospheres at 700-800°C for 10-100 h. XRD, SEM, and SEM-EDS were used to analyze the coatings before and after oxidation. XPS study was carried out for selected samples to determine the coating performance for mitigating chromium evaporation from the SS430 substrate. A comparative study of the nickel coating processing methods will be discussed based on the experimental results.

S5: Next Generation Bioceramics and Biocomposites

Bioceramics II

Room: Coquina Salon F

Session Chairs: Leena Hupa, Åbo Akademi University; Leif Hermansson, Applied Research Sweden AB

8:30 AM

(ICACC-S5-009-2017) Bioactive glass-ceramics from novel 'inorganic gel casting' and sinter-crystallization (Invited)

H. Elsayed[1]; A. Rincon Romero[1]; C. Vitale-Brovarone[2]; E. Bernardo*[1]; 1. University of Padova, Italy; 2. Politecnico di Torino, Italy

Highly porous wollastonite-diopside glass-ceramics have been successfully obtained by a new gel-casting technique. The gelation of an aqueous slurry of glass powders was not achieved according to the polymerisation of an organic monomer, but as the result of alkali activation. The alkali activation of a Ca-Mg silicate glass (with a composition close to 50 mol% wollastonite - 50 mol% diopside, with minor amounts of Na_2O and P_2O_5) allowed for the obtainment of well-dispersed concentrated suspensions, undergoing progressive hardening by treatment at low temperature (80°C), owing to the formation of C-S-H (calcium silicate hydrate) and M-S-H (magnesium silicate hydrate) gels. An extensive direct foaming was achieved by vigorous mechanical stirring of partially gelified suspensions, comprising also a surfactant. The open-celled structure resulting from mechanical foaming could be 'freezed' by the subsequent sintering treatment, at 800-900°C, causing a substantial crystallization. A total porosity exceeding 85%, comprising both well-interconnected macro-pores and micro-pores on cell walls, was accompanied by an excellent compressive strength, above 3 MPa. An extension of the approach to another Ca-Mg bioglass (CEL2, richer in Na_2O and P_2O_5), will be presented as well.

8:50 AM

(ICACC-S5-010-2017) On the Potential of Total Chemically Bonded Ceramic System within Odontology (Invited)

L. Hermansson*[1]; 1. Applied Research Sweden AB, Sweden

The presentation deals with the chemically bonded bioceramic systems and their potential within odontology. The materials can be produced from chemically bonded ceramics of the CaO-Al2O3-SiO2-P2O5-H2O system, the CASPH-system. Materials from this main system can be used for products as dental cement, inlay, onlay, restorative and base, as well as coatings. In this way a total chemically bonded bioceramic system can be formed from the tooth structure via the dental cement to inlay and capping material including a base in the case a waist appear in the tooth cavity to be repaired. Additive manufacturing is a great possibility in the system. Nanostructures including nanocrystals and nano-porosity are easily formed in the CASPH-system due to a low solubility of the phases formed. Requirements according to ISO Standards related to mechanical properties including among others flexural strength, compressive strength, fracture toughness and elasticity, and to biological properties such as biocompatibility, bioactivity, tissue integration and antibacterial properties, are easily fulfilled.

9:10 AM

(ICACC-S5-011-2017) Biomimetic remineralization using dental calcium silicate cements (Invited)

S. S. Bhasin*[1]; V. Aggarwal[2]; 1. Faculty of Dentistry, Jamia millia Islamia, India; 2. Faculty of Dentiatry, India

The direct dental composite restorations are commonly used to restore the cavitated teeth. The resin composites are inherently hydrophobic and pose a challenge to bond with hydrophilic dentin. To improve the bonding, the tooth surface is etched with phosphoric acid. This creates micro-mechanical tags between the composite restoration and the dentinal surface. However acid etching also opens up the dentinal tubules and increases the fluid movement inside the tubules. As a result the hydrophobic resins are not able to penetrate the full depth of hydrophilic demineralized dentin. Thus an uninfiltrated demineralized layer of dentin remains beneath the hybrid layer. This uninfiltrated layer is prone to hydrolytic degradation by Matrix Metallo Proteins (MMPs). To prevent this degradation, a new method of remineralizing the uninfiltrated dentin is proposed and evaluated. It combines the use of calcium silicates with casein phosphopeptides. It is hypothesized that calcium silicates will remineralize the deep layers of dentin and the casein phosphopeptides will stabilize this mineralization. The study involved an in vitro experiment on the extracted human teeth. The results of the study will be presented in the scientific presentation

9:30 AM

(ICACC-S5-012-2017) Near-Infrared Excited Nanoparticles for Biomedical Applications (Invited)

F. Vetrone*[1]; 1. Institut National de la Recherche Scientifique, Canada

Nanoparticles excited in the near-infrared (NIR) are quickly emerging as useful tools in diagnostic and therapeutic medicine. In particular, the usefulness of these nanomaterials 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 (approximately BW-1, 700-990; BW-II, 1050-1350; and BW-III, 1550-1870 nm). The ability to stimulate luminescent inorganic nanoparticles with NIR light has made possible their use in a plethora of biomedical applications. In fact, the biggest impact of such materials would be in the field of disease diagnostics and therapeutics, now commonly referred to as theranostics. Here, we present the synthesis of various NIR excited nanoparticles and demonstrate how they can be used in the development of a multifunctional hybrid nanoplatform for the potential diagnostics and therapeutics of disease including bioimaging, controlled drug delivery, nanothermometry, photodynamic therapy, photothermal therapy and thermal ablation.

10:10 AM

(ICACC-S5-013-2017) Glass-infiltrated ceramic composites for dental restorations: Effects of processing parameters to achieve high translucency (Invited)

H. N. Yoshimura*[1]; A. Chimanski[1]; P. F. Cesar[2]; 1. Federal University of ABC, Brazil; 2. University of Sao Paulo, Brazil

Glass-infiltrated ceramic composites have been used in Dentistry due to their biocompatibility and high potential to mimic natural dentition. These composites have shown high strength and toughness, which favors for long lifetime, but their translucency is relatively low, which limits aesthetic qualities. In this work, the effects of glass composition of the system SiO_2-B_2O_3-Al_2O_3-La_2O_3-TiO_2-CaO-CeO_2, designed using SciGlass, as well the infiltration parameters on optical properties of alumina-based composites were evaluated. The prepared glasses were infiltrated into porous alumina preforms between 1000 and 1200°C, showing that the increase in B_2O_3 content enhances the infiltration process, due to the decrease in glass viscosity. The addition of CaO or CeO_2 further improved the infiltration distance due to the increase in wettability. The addition of CaO was also benefic to increase the light transmittance of the composite, but the addition of CeO_2 decreased it significantly, because of the coloring effect. It was also observed that the multi-cycle infiltration and pressure-assisted infiltration are effectives in reducing residual porosity and improving light transmittance. The results were discussed on the basis of measured refractive indexes of glasses, and coefficients of absorption and scattering by Kubelka-Munk model from reflectance measurements.

10:30 AM

(ICACC-S5-014-2017) Porous scaffolds of bioactive silicate glasses using different fabrication techniques (Invited)

L. Hupa*[1]; L. Aalto-Setälä[1]; S. Fagerlund[2]; S. Eqtesadi[1]; 1. Åbo Akademi University, Finland; 2. Paroc Group Oy, Finland

Currently, extensive research efforts are paid to fabricate porous tissue engineering scaffolds from bioactive glasses. The main requirements for the scaffolds can be summarized as a controlled degradation combined with a suitable porous 3D architecture and an appropriate mechanical strength. This presentation discusses the properties of scaffolds manufactured from bioactive glasses 45S5, S53P4 and 13-93 through three methods: i) direct sintering of particles, ii) foam replication method, and iii) additive manufacture. In the scaffold manufacture, thermal treatment above the glass transition temperature is required to achieve the desired 3D structure and mechanical strength. However, the strong tendency of the bioactive glasses to crystallize challenges the degradation and thus also the release of the inorganic ions affecting the cellular processes. The scaffolds can be divided into three main types based on their final phase composition: glass-ceramic scaffolds which easily crystallize in all thermal treatments, biphasic scaffolds which can be sintered to a limited degree without extensive crystallization, and amorphous scaffolds. The glass composition, the particle size utilized, and the thermal history control the final phase composition and thus the degradation kinetics of the scaffolds. The results give guidelines for the choice of glasses to be used in various scaffold structures.

10:50 AM

(ICACC-S5-015-2017) The phase-partitioning-dependent ageing and fracture mechanics in 3Y-TZP bioceramics

A. Kocjan*[1]; T. Kosmac[1]; D. Bucevac[2]; 1. Jozef Stefan Institute, Slovenia; 2. Vinca Institute of Nuclear Sciences, University of Belgrade, Serbia

Yttria-stabilized tetragonal zirconia (3Y-TZP) is a popular bioceramic structural material due to its excellent biocompatibility and mechanical properties. However, it is prone to moisture-induced, tetragonal-to-monoclinic (t–m) transformation, a detrimental degradation process known as ageing, mainly governed by the amount of yttria in the solid solution and the grain size, but it is also sensitive to the presence of internal strains and residual stresses. We have investigated the influence of phase partitioning of 3Y-TZP on the stability and structure of transformable yttria-lean tetragonal phase (YLZ) and un-transformable yttria-rich phases (YRZ; t`` and t`) and on the formation of residual stresses in YRZ, further affecting the ageing kinetics and fracture mechanics. An attempt was made to differentiate between the effect of phase partitioning from the effect of grain size and grain-boundary strengthening by sintering alumina-free 3Y-TZP at different sintering temperatures and times to yield ceramics with identical grain size, but differing in phase composition. The results indicate that the formed YRZ phases are under compressive stresses in the YLZ matrix, since a systematic relaxation after ageing was observed. The observed relaxation puts additional perspective on the understanding of the t–m transformation mechanism governing both ageing and fracture mechanics in 3Y-TZP.

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

Thermal Energy Conversion and Energy Storage XI

Room: Tomoka A
Session Chair: Holger Kleinke, University of Waterloo

8:30 AM

(ICACC-S6-038-2017) Atomistic manipulation of Phonon Thermal Conduction in Layered Thermoelectric Oxides (Invited)

M. Yoshiya*[1]; S. Fujii[1]; 1. Osaka University, Japan

To reduce thermal conductivity without deteriorating electronic properties responsible for thermoelectricity is one of the challenging issue to tackle with. Conventional theories of thermal conductivity often fails to predict thermal conductivity of thermoelectric oxides, thereby disabling us to further control thermal conductivity. It is primarily due to wider spectrum of interatomic bonding ranging from ionic one to covalent one in oxides compared with metallic counterparts. In addition, more complicated crystal structures disable us to use the conventional theory, especially when crystals has layered structure, unlike macroscopic layered matter. To overcome this difficulty, we have performed atomistic simulation of layered thermoelectric oxides, not only to obtain ideal value of thermal conductivity, but also to reveal underlying mechanisms that govern thermal conduction, thereby enabling us to tailor thermal conduction in the layered thermoelectric oxides. In this talk, recent progress in revealing the mechanism behind microscopic heat transport in the layered oxides and attempts to manipulate thermal conduction on the atomic level based on the understanding will be presented.

9:00 AM

(ICACC-S6-040-2017) Durability and improvement of power generation for oxide thermoelectric module (Invited)

R. Funahashi*[1]; Y. Matsumura[1]; T. Urata[1]; M. Suzuki[1]; H. Ikenishi[1]; 1. National Institute of Advanced Industrial Science and Technology (AIST), Japan

Long life time test has been carried out for oxide thermoelectric modules up to 1073 K for the hot-side temperature in the air atmosphere. The module fabricated in this study is composed of $Ca_{2.7}Bi_{0.3}Co_4O_9$ (Co-349) and $CaMn_{0.98}Mo_{0.02}O_3$ (Mn-113) for the p-type and n-type legs, respectively. The Co-349 bulks were prepared using a hot-pressing technique to make the bulk density and the grain alignment high. The Mn-113 pellets were sintered at 1473 K in air. Both bulks were cut to provide thermoelectric legs with a cross section of 3.5×3.5 mm and length of 5 mm. The 14 pairs of p and n-type devices were used to prepare the modules. Ag paste including Co-349 powder and Ag sheets. An alumina plate with 0.38 mm thick was used for one side of the module as a substrate. The module was put on a plate shape furnace to heat at 873-1073 K (T_H) in air. A cooling jacket composed of Cu was put on the other side the module and cooled at 293 K by water circulation (T_C). Measurement of the power generation (P_{max}) was carried out in air by scanning the load resistance using a DC power supply device for one month. The load resistance was connectedto generate 1 A continuously from the module, and the generated voltage (P_{1A}) was measured for one month. Although the P_{max} and P_{1A} change due to the changes of temperature difference between T_H and T_C, no degradations in both generated power are observed up to 1073 K of T_H.

9:30 AM

(ICACC-S6-036-2017) Thermoelectric properties of plasma spray synthesized sub-stoichiometry TiO_{2-x} and associated energy harvesting devices

H. Lee*[1]; S. Han[1]; R. Chidambaram Seshadri[1]; S. Sampath[1]; 1. Stony Brook University, USA

The thermoelectric properties of sub-stoichiometric TiO_{2-x} deposits prepared by plasma spray deposition process are investigated from room temperature to 750 K. The deposits resultant from interaction with the reducing hydrogen laden plasma and subsequent re-oxidation in-flight through interaction with surrounding atmospheric oxygen leads to a complex interplay among process conditions, stoichiometry and phases. The deposit comprises of both Rutile and Magneli phases contributing to process dependence thermoelectric properties. Under optimal conditions, Seebeck coefficients of -230 $\mu V\ K^{-1}$ and electrical conductivity of $5.48\times10^3\ S\ m^{-1}$ are obtained resulting in a power factor of 2.9 $\mu W\ cm^{-1}\ K^{-2}$ at temperatures of 750 K. The lamellar deposits resultant provides thermal conductivity of 1.65 $W\ m^{-1}\ K^{-1}$ at 750 K and associated ZT value of 0.132. Multilayer assemblies of patterned p-n junctions allowed for fabrication of series and parallel connected thermoelectric elements allowing for enhancement in current, voltage and in some cases both. The results point to a potential pathway for a large scale fabrication of low-cost oxide based thermoelectric with potential applicability to moderate temperatures.

Thermal Energy Conversion and Energy Storage XII

Room: Tomoka A

Session Chair: Masato Yoshiya, Osaka University

10:20 AM

(ICACC-S6-041-2017) A recommendation engine for suggestion unexpected thermoelectric chemistries: Prediction and validation (Invited)

T. D. Sparks*[1]; A. Mar[2]; A. Oliynyk[2]; M. W. Gaultois[3]; B. Meredig[4]; G. Mulholland[4]; 1. University of Utah, USA; 2. University of Alberta, Edmonton, Canada; 3. University of Cambridge, United Kingdom; 4. Citrine Informatics, USA

Materials with complex crystal structures are being widely used in thermoelectric applications due to the inherent low thermal conductivity in complex, layered, disordered, rattling, and defect structures. The RE12Co5Bi (RE=Gd, Er) compounds, closely related to the structurally complex skutterudite family of materials, have recently been recommended via a first-of-its-kind machine learning algorithm as good candidate thermoelectric materials. Pure, polycrystalline samples have been synthesized and the thermoelectric transport properties have been measured. Although the samples are too metallic to have good thermoelectric performance (~15μV/K) the materials exhibit highly unusual and unexplained thermal conductivity which increases twofold with temperature rather than decreases. In this contribution we describe the materials recommendation engine as well as recent experiments using high temperature synchrotron diffraction to look for systematic changes to the RE12Co5Bi structure such as changes to the thermal displacement parameter which could explain the unusual thermal conductivity observed in these materials.

10:50 AM

(ICACC-S6-042-2017) Investigation of Thermoelectric Properties of Polymer-Derived SiOCN Ceramics

R. Iyer*[1]; S. Pilla[1]; 1. Clemson University, USA

Increasing stringency of environmental standards for automobiles, both within US through CAFÉ standards, and across the globe through the climate change pact at the Conference of Parties (CoP) 2015, has led to enhanced focus on improving fuel economy of automobiles. Among several ongoing efforts in this regard, harvesting heat energy wasted in exhaust pipes through use of thermoelectric devices has gained attention. However, currently available commercial thermoelectrics suffer from issues of high weight, leading to increase in vehicular weight and reduction in potentially achievable fuel savings. This work is an attempt at evaluating polymer-derived ceramics as a possible, low-weight thermoelectric alternative. SiOCN ceramic samples were processed through curing and pyrolysis of polymer precursors at temperatures from 1000 to 1400 C. Thermoelectric properties – thermal conductivity, electrical conductivity and Seebeck coefficient – of SiOCN samples were measured at different temperatures to calculate their corresponding ZT values. FTIR, XRD and elemental analysis were undertaken to measure chemical composition of ceramic samples, while Raman spectroscopy was undertaken to analyze the nature of free carbon present in the ceramic. SEM and HRTEM of ceramic samples were undertaken to analyze their compositional distribution.

11:10 AM

(ICACC-S6-043-2017) Development of a phase change material composite for thermal energy storage for solar power plants

Y. Jiang[2]; Y. Sun*[1]; S. Li[2]; F. Bruno[3]; 1. CSIRO, Australia; 2. Univeristy of New South Wales, Australia; 3. University of South Australia, Australia

Concentrated solar power technology uses focused sunlight to provide heat at high temperature for generation of electricity or applications in energy-intensive industries. Thermal energy storage plays a significant role in increasing its efficiency and reducing its capital cost. The low cost and high heat of fusion of inorganic salts

such as chlorides and carbonates are reported as good thermal energy storage materials. However, these salts are highly corrosive to metal containment construction materials. Ceramics have shown a good corrosion resistance to these molten salts. This work focuses on developing an eutectic NaCl-Na_2CO_3 salt-ceramic composite to prevent the salt from direct interaction with the containment materials. Thermal stability of the eutectic NaCl-Na_2CO_3 salt was first examined in CO_2 and N_2 environments. The results show that the eutectic NaCl-Na_2CO_3 salt has higher thermal stability in CO_2 than in N_2. The high thermal stability results from the presence of CO_2 inhibiting the salt's thermal decomposition. The salt ceramic composite was designed and its thermal stability was tested through a thermal cycling in CO_2. The results indicate that the composite has good chemical stability after 100 thermal cycles.

S7: 11th International Symposium on Nanostructured Materials: Functional Nanomaterials and Thin Films for Sustainable Energy Harvesting, Environmental and Health Applications

New Materials and Processing III

Room: Coquina Salon A
Session Chair: Renata Solarska, University of Warsaw

8:30 AM

(ICACC-S7-040-2017) Scanning near field optical microscopy (SNOM) imaging and its applications in energy harvesting design (Invited)

G. Fanchini*[1]; 1. University of Western Ontario, Canada

In this presentation, we will offer an overview of scanning near field optical techniques, including the development of three dimensional (3D) SNOM methods undertaken by our team to locally image the distribution of the electromagnetic radiation harvested by random and deterministic nanosystems. Metallic nanostructures exhibit unique effects under light illumination, including plasmon resonance and waveguiding properties. We will discuss a few specific applications in which 3D-SNOM was utilized for designing and understanding specific functional nanomaterials and thin films for the harvesting of light at the nanoscale. Specific case studies that will be presented include molecular gold nanoclusters embedded in polyimide, the design of plasmonic photovoltaics optically enhanced by random arrangements of copper nanoparticles, and the use of 3D-SNOM for characterizing evanescent waveguides self-assembled from of copper nanoparticles assembled on thin films of graphene.

9:00 AM

(ICACC-S7-041-2017) Epitaxial strain on metal-insulator transition characteristics of $SmNiO_3$ thin films (Invited)

B. Torriss*[1]; 1. Institut National de la Recherche Scientifique, Canada

Rare earth nickelates $RNiO_3$ display a first-order metal-insulator transition (MIT) at a critical temperature (T_{MI}). This transition is accompanied by dramatic variations of electrical resistivity, infrared and terahertz transmission, which make them interesting candidates for technological applications, such as sensors, electronic switching and thermochromic coatings. However, the synthesis of these materials remains a bottleneck to research due to their thermodynamic instability. In this presentation, we overview our recent studies on the synthesis of $SmNiO_3$ thin films by Pulsed Laser Deposition (PLD). We report the influence of epitaxial strain on the structure, electronic transport and optical properties of epitaxial $SmNiO_3$ thin films grown on $LaAlO_3$ (LAO, compressive) and $SrTiO_3$ (STO, tensile). As the strain changes from tensile to compressive, the transition temperature of the $SmNiO_3$ samples shifts to lower temperatures. The optical conductivity reveals the strong dependence of the Drude spectral weight on the strain relaxation.

9:30 AM

(ICACC-S7-042-2017) Lithium-Sulfur Battery Electrodes: Insights from Mesoscale Modeling and Analysis (Invited)

P. Mukherjee*[1]; 1. Texas A&M University, USA

Recent years have witnessed tremendous interest in energy storage technologies for vehicle electrification and renewable energy integration in the electric grid. The lithium-sulfur (Li-S) battery could be the next promising candidate due to its high theoretical capacity and energy density. Evidently, the Li-S battery is a complex electrochemical system which involves a multitude of physicochemical phenomena occurring at multiple scales. The mesoscale modeling approach aims to answer how to improve battery performance from different perspectives. Introducing defects into discharge products is a promising way to increase the electrical conductivity. The first-principle simulation is a powerful tool to predict the chemical properties of defects in Li_2S. Also, the first-principle simulation can also reveal interaction mechanisms between polysulfides (PSs) and electrode surfaces. The growth kinetics can be used to investigate the influence of Li_2S precipitation on cathode microstructure variation, which affects the species transport and the operating condition on the battery performance. The mesoscale modeling approach provides a fundamental study of physical and electrochemical interplays in Li-S battery electrodes.

10:00 AM

(ICACC-S7-043-2017) Complex porous structures through solution synthesis

G. Westin*[1]; 1. Uppsala University, Sweden

The development of catalysts for solar fuels, solar cells and photo-assisted water and air cleaning require robust, routes capable of producing advanced hetero-metallic and hetero-phasic complex 3D structured materials. Here is discussed solution based processing routes to metal, composite and oxide porous 3D structures of complex composition, including structures coated with ultra-thin complex films. The controllability with respect to composition and structure will be discussed for a range of materials of potential use in catalysis, photo-catalysts, batteries and solar cells. The materials prepared were characterized with a large range of analytical techniques including TEM, SEM, XRD, IR and Raman spectroscopy, DSC and TG, and their structures will be related to their properties.

New Materials and Processing IV

Room: Coquina Salon A
Session Chair: Anja Mudring, Iowa State University

10:30 AM

(ICACC-S7-044-2017) Atomic diffusion and vibrational properties of alumina ceramics by first principles calculations (Invited)

T. Tohe*[1]; 1. The University of Tokyo, Japan

α-Al_2O_3 is one of the most popular oxide ceramics and widely used as high-temperature structural materials due to its high mechanical strength and chemical stability up to elevated temperature. Atomic diffusion or vibrational properties at grain boundaries (GBs) in alumina is of great interest because it is responsible for GB sliding which is an elementary process of creep deformation at high temperatures. However, its atomistic details and mechanisms have still been elusive despite extensive studies devoted so far. In this study, we have made first principles theoretical analysis on atomic migration and vibrational (phonon) properties at alumina GBs to understand elementary process of GB diffusion with atomistic detail. We focused on alumina {101}S11 GB as a model system in the present study. Quantitative atomistic simulations were performed on model GB structures by employing first principles plane wave basis

PAW method, in conjunction with bicrystal fabrication and STEM observation experiment. Based on the S11 GB structure model verified by the STEM observations, we have evaluated elementary migration paths and activation energies for the alumina GB diffusion. We also calculated and discuss about phonon spectrum and thermodynamic functions on the GB containing structure models. The results should add new and interesting information about GB related phenomena in alumina ceramics.

11:00 AM

(ICACC-S7-045-2017) Dispersion/assembled structure control of nanoparticles in non-aqueous multicomponent slurries by surface modification and microstructure control of Si-based green body for post-reaction sintering (Invited)

M. Iijima*[1]; 1. Yokohama National University, Japan

Microstructure control of green bodies as well as particle-based composite materials is quite essential issue to tune and improve the properties of end products which appears in the form of sintered bodies and/or polymer nanocomposites etc. Herein we report a unique assemble process of fine-/nano-particles modified by PEI-OA complex on un-modified core particles in non-aqueous multicomponent slurries. While un-modified core particle itself strongly aggregated in non-aqueous solvents, addition of PEI-OA modified fine-/nano-particles resulted to the assembly of surface modified fine-/nano-particles along un-modified core particles, and simultaneously, the dispersion stability of as-prepared multicomponent slurry with composited particles has drastically improved. The proposed method for dispersion structure control of nano-particles has applied to Si-Y_2O_3-MgO/toluene multicomponent slurry system. Relations among the assembled structure of raw fine particles in the slurries on the microstructure of green compacts prepared from the corresponding slurry, nitrided body and post-reaction sintered body of Si_3N_4 has investigated.

11:30 AM

(ICACC-S7-046-2017) Thin Films and Nano Wires based on Pyrochlore $Nd_2Sn_2O_7$ for Energy Harvesting and Gas Sensing Applications

A. Jamil*[1]; Y. Gönüllü[1]; P. Sekhar[2]; S. Mathur[1]; 1. Inorganic Chemistry, Germany; 2. Engineering and Computer Science, USA

Single-source precursors to rare earth metal pyrochlores, $Ln^{III}M^{II}(O^tBu)_5Py$ (Ln^{III} = Nd; M^{II} = Sn; Py = pyridine), were synthesized by equimolar reaction of in-situ produced individual metal alkoxides. Nanofibers and thin films of rare earth pyrochlores were produced by electrospinning and spin-coating of chemical sols obtained from mixed-metal single-source precursors. Photocatalytic studies demonstrated high activity for $Nd_2Sn_2O_7$ nanofibers when compared to commonly applied TiO_2 nanostructures used as the reference. Moreover $Nd_2Sn_2O_7$ films grown over Fe_2O_3 photoanodes yielded superior efficiency for photoelectrochemical splitting of water in comparison to bare Fe_2O_3 layers, possibly due to a higher absorption cross-section and enhanced charge carrier mobilities in the $Nd_2Sn_2O_7//Fe_2O_3$ heterojunction. Moreover, $Nd_2Sn_2O_7$ films were deposited on the Al_2O_3 sensor platforms, onto which the interdigitated Pt circuits were previously deposited by screen-printing. The gas-sensing characterization was carried out in a specially constructed apparatus by controlling the temperature and total gas flow. The sensor measurements were conducted under different gas exposure (such as H_2, CH_4, CO and NO_2). The results showed that the $Nd_2Sn_2O_7$ presents a response only toward to H_2 with a fast response and recovery time.

11:50 AM

(ICACC-S7-047-2017) Nanostructured Composites Towards Multifunctional Lightweight Conformal Structures (Invited)

M. Jung de Andrade*[1]; 1. University of Texas at Dallas, USA

In the era of the "Internet of Things" (IoT), flexible nanostructured systems have a significant impact towards multifunctional morphing and lightweight technologies. In this work, we discuss some of the main techniques to fabricate conformal nanostructured materials. For instance, core-shell structure of highly conductive, transparent and flexible woven partially aligned structure of indium-tin oxide over poly(acrylonitrile) nanofibers showed good potential for curved thermoacoustic sound generators. Moreover, both infiltration and conformal deposition routes demonstrated the versatility of nanostructured carbon nanotube based composites towards yarn-like sensors and actuators. Finally, we recently reported a conceptual work towards flexible woven-yarn thermoelectric that merged conformal-like deposition method for nanostructured materials with conventional textile technologies.

12:10 PM

(ICACC-S7-048-2017) X-ray Spectroscopy of Energy-Relevant Material in Operando Condition (Invited)

C. Dong*[1]; 1. Tamkang University, Taiwan

A new age of human demand for renewable energy is coming and material scientists are devoted to hunting for new functional materials to create more clean and sustainable energy. To improve the energy conversion/generation/storage efficiency of current energy material is a great challenge. In many important energy material systems such as nanostructured catalysts, artificial photosynthesis, smart materials, and energy storage devices, it is essential to determine the variation of atomic/electronic structures near the interfacial region under the real working environment. Without comprehending the fundamental properties such as chemical states, atomic and electronic structures and how they alter near the interfacial region during the physical/chemical reaction, it is difficult to better design the material for advancing performance in an effective approach. Exploiting the in situ characterization approach, the way to monitor the modification of atomic and electronic structures of the energy material under operational environment now becomes accessible. This presentation will report the in situ characterization on energy relevant materials by synchrotron x-ray spectroscopy, including soft- and hard-x-ray absorption spectroscopy. Emerging characterization tool, recent progress of in situ technique development, and a number of recent studies will be presented in this talk.

S8: 11th International Symposium on Advanced Processing and Manufacturing Technologies for Structural and Multifunctional Materials and Systems (APMT11)

Novel Ceramic Processing IV

Room: Coquina Salon B
Session Chairs: Jerzy Lis, AGH University of Science and Technology; Tohru Suzuki, National Institute for Materials Science

8:30 AM

(ICACC-S8-026-2017) High-hardness Diamond-based Composites by Spark Plasma Sintering (Invited)

T. Goto*[1]; M. Kitiwan[1]; H. Katsui[1]; 1. IMR Tohoku University, Japan

Diamond has been used for cutting tools because of its high hardness. Diamond powder is commonly consolidated at a ultra-high pressure around several GPa. If the diamond can be densified at a moderate pressure less than 100 MPa, it is practically useful for wide-ranged applications. Although spark plasma sintering (SPS) can densify various hard-to-sinter materials, diamond powder cannot be consolidated by SPS. On the other hand, by coating sinterable materials on diamond powder making core-shell powder, diamond powder would be consolidated and dense diamond-based composites can be prepared by SPS at moderate pressure. Diamond powder (2 to 4 μm in dia.) was coated by SiC layer (25 to 30 nm in thickness) using rotary chemical vapor deposition. The diamond (core)/ SiC (shell) powder was densified with SiO_2 powder (35 mass %) by spark plasma sintering (SPS). The diamond powder without SiC layer mixed with SiO_2 was about 80 %, whereas diamond and diamond-SiC powder without SiO_2 cannot densified, Dr= 50 %. No phase transformation from diamond to graphite was observed in diamond/SiC-SiO_2 composite at a sintering temperature of 1873 K. The highest density and Vickers microhardness were 94 % and 39 GPa (4.9 N load), respectively.

9:00 AM

(ICACC-S8-027-2017) Predicting powder densification behavior in large temperature gradients produced by Spark Plasma Sintering

P. Colasuonno*[1]; 1. Colorado State University, USA

In this study we investigate powder sintering behavior inside large temperature gradients to produce functionally graded materials (FGMs) with the Spark Plasma Sintering (SPS) technique. Practical tooling and considerations to create and characterize large steady state temperature gradients with Joule heating is described. Densification and grain growth models of the powder system were created and compared to resulting FGM microstructures at representative sintering conditions. The apparent sintering activation energy was calculated using the Master Sintering Curve (MSC). The empirical data can be used to produce FGMs with tunable porosity and grain size distributions using engineered thermal gradients.

9:20 AM

(ICACC-S8-028-2017) The effect of sintering parameters on the properties of B_4C samples sintered by SPS

M. Asadikiya*[1]; C. Rudolf[1]; C. Zhang[1]; B. Boesl[1]; A. Agarwal[1]; Y. Zhong[1];
1. Florida International University, USA

Boron carbide (B_4C) has various applications such as abrasive powder, blasting nozzles, and grinding wheels. It is due to its interesting properties like high hardness, low density and good chemical stability. However, B_4C is difficult to sinter because of its covalently bonded structure. The sintering parameters play critical role in the density and mechanical properties of a sintered B_4C sample. It is important to identify the effect of each sintering parameter and also the reason for that effect in order to design proper sintering parameters to reach a desired sintered sample. In this study, the effects of sintering temperature, heating rate, holding time and also increasing the number of graphite foil layers are investigated on the density and hardness of the Spark Plasma Sintering (SPS) of B_4C powder. The results are described based on the computational thermodynamics predictions. The results will provide better understanding of the effects of sintering parameters by the support of computational thermodynamics.

9:40 AM

(ICACC-S8-029-2017) Sintering Condition and Microstructure of Translucent Silicon Nitride Ceramics by SPS Process (Invited)

J. Hojo*[1]; 1. Kyushu University, Japan

Translucent Si_3N_4 ceramics are expected to use for a special optical application in high temperature and corrosive environment owing to its high mechanical strength and thermal and chemical stability. To achieve the translucency of Si_3N_4 ceramics, fine-grained microstructure with an appropriate sintering aid is important to reduce light scattering. The Si_3N_4 ceramics were fabricated by spark-plasma sintering (SPS) with AlN-MgO additive. Fine α-Si_3N_4 powder with high purity was useful to obtain translucent Si_3N_4 sintered body. When the additive content was large, α-SiAlON formed, whereas β-Si_3N_4 formed at a small additive content, revealing the high translucency of both phases in visible light region. The AlN/MgO ratio should be on the SiAlON forming line (AlN/MgO=3/1 wt.) in Si_3N_4-AlN-MgO phase diagram. The short-time sintering at high temperature (1850°C) by SPS was effective to obtain the dense and fine-grained microstructure, and the transmittance of 28 % at the wavelength of 900 nm was achieved under the optimal condition.

10:20 AM

(ICACC-S8-030-2017) Coupled electro-thermo-mechanical simulation for multiple pellet fabrication of uranium dioxide in spark plasma sintering

B. Nili*[1]; G. Subhash[1]; J. Tulenko[2]; 1. University of Florida, USA;
2. University of Florida, USA

Spark plasma sintering (SPS) is a powder unification technique where a pulsed current and uniaxial pressure are employed to fabricate near-net-shape components with high microstructural homogeneity at significantly lower processing temperatures and in less time than traditional methods. Also, SPS has the economic benefit of enabling the fabrication of multiple specimens in a single run. In the present study, a comprehensive experimental and three-way coupled electro-thermo-mechanical finite element (FE) modeling approach is undertaken to investigate the temperature field and stress distribution within a specimen and SPS tooling which consists of punches, spacers, and a die. The FE analysis is used to propose methods for reducing the overall power consumption of the manufacturing process by optimizing the dimensions and configurations of SPS tooling used to sinter multiple specimens in parallel. Furthermore, the pellet microstructure and stress distribution are discussed with respect to their dependence upon various parameters, such as heating rates and the presence of graphite foil at interfacial contacts. The current results of the FE method may be used for further SPS optimization as they demonstrated a good agreement compared with experimental data on uranium dioxide fuel pellets.

10:40 AM

(ICACC-S8-031-2017) Liquid Phase SPS Manufacture and Characterization of Mayenite-electride/titanium composite

T. J. Hammann*[1]; P. Colasuonno[1]; T. B. Holland[1]; 1. Colorado State University, USA

A novel titanium matrix composite (TMC) consisting of equal mass% mayenite electride and titanium was synthesized by spark plasma sintering (SPS) in a liquid phase by a single step from simple precursor powders. Mayenite electride is a crystalline ceramic in which electrons replace oxygen atoms in subnanometer sized cages and act as a conductive medium. Mayenite electride has a predicted work function of 0.6 eV, leading to high interest for cold-cathode field emitters, electronic propulsion, and refrigeration devices. Titanium is added to the system to provide structural reinforcement as well as providing means for ohmic conduction in the composite allowing for direct heating by electrical current. Prior to sintering, powders were high energy ball milled before mixing with the titanium. To control a liquid phase in SPS, a novel die design was fabricated to sinter the metal matrix composite. X-ray diffraction (XRD) confirms the presence of mayenite, while scanning electron microscopy (SEM) proved good distribution of the mayenite in the titanium matrix. Microhardness testing was conducted on individual phases of the composite and reported for the first time on sintered mayenite and results agree with previously published titanium hardness values.

11:00 AM

(ICACC-S8-032-2017) BCN Phase Formation via Reaction Synthesis of 2D Graphene and Boron Nitride Nanosheet using Spark Plasma Sintering

A. Loganathan*[1]; A. Sharma[2]; P. Nautiyal[1]; S. Suwas[2]; B. Boesl[1]; A. Agarwal[1]; 1. Florida International University, USA; 2. Indian Institute of Science, India

Boron carbon nitride (BCN) system in the recent past has drawn major research interest due to their promising mechanical, lubrication and electrical properties. Generally the BCN systems were synthesized by solid phase pyrolysis, CVD, PVD, mechanical alloying, high pressure high temperature synthesis or nitrification of solid phase precursors. In the present work, two dimensional (2D) materials namely graphene nanoplatelet (GNP) and boron nitride nanosheet (BNNS) were used as the starting materials. They were mixed uniformly by mechanical alloying. Further the mixture was used to form the BCN phase by reaction synthesis using spark plasma sintering (SPS) technique. The phase formation in the sintered compacts were characterized by X-ray diffraction, FTIR, Raman spectroscopy and transmission electron microscope. Additionally, the nanomechanical properties of the sintered 2D-BCN was studied. High load in-situ indentation studies were conducted to understand the deformation mechanism of reaction synthesized BCN phase.

S10: Virtual Materials (Computational) Design and Ceramic Genome

Multiscale and Interface Modeling

Room: Ponce DeLeon

Session Chairs: Gerard Vignoles, University Bordeaux; Paul Rulis, University of Missouri - Kansas City

8:30 AM

(ICACC-S10-039-2017) Recent Developments in the Orthogonalized Linear Combination of Atomic Orbitals Method: Application to Complex Systems (Invited)

P. Rulis*[1]; 1. University of Missouri - Kansas City, USA

The orthogonalized linear combination of atomic orbitals (OLCAO) method is a density functional theory based method with a localized basis set and periodic boundary conditions. Since the source code for the method was first publicly released in 2011 a number of significant developments and advancements have been made in the newest release of the package. The structure of the basis set has been redeveloped, new density functionals have been included, and among others, significant efficiency improvements have been made that enable the OLCAO package to deal with ever larger and more complex material systems. This talk will first briefly summarize key developments in both the OLCAO method itself and in the associated support tools. Then, examples that illustrate application of the OLCAO method to complex materials will be presented including complex surfaces, nano-polycrystalline materials, and amorphous molecular solids.

9:00 AM

(ICACC-S10-040-2017) Towards the Development of "Grain Boundary Diagrams" as a Useful Materials Science Tool: From Phenomenological Thermodynamic Models to Atomistic Simulations (Invited)

S. Yang[1]; N. Zhou[1]; J. Luo*[1]; 1. UCSD, USA

Grain boundaries (GBs) can undergo phase-like (complexion) transitions with broad impacts on fabrication processing (e.g., sintering), microstructural evolution (grain growth), and various GB-controlled mechanical, chemical (e.g., oxidation), electronic (including superconductive), ionic, magnetic, and thermal properties. In the last a few years, we have made great efforts to develop several phenomenological interfacial thermodynamic models to successfully forecast some useful trends in the formation of 2-D interfacial phases (also called "complexion") at GBs and subsequently construct "GB diagrams". More recently, we utilize a hybrid molecular dynamics (MD) and Monte Carlo (MC) method, in conjunction a modified genetic algorithm (GA), to predict equilibrium GB structures and transitions in a semi-grand canonical ensemble. The results from atomistic simulations are used to construct GB "phase" (complexion) diagrams, which reveal the GB excess, effective interfacial width, structural disorder, and GB diffusivity as functions of both the bulk concentration/chemical potential and equilibrium temperature. Examples of a metallic alloy (Mo-Ni), a metal-nonmetal mixed system (Ni-S), and oxide ceramics (Y_2O_3-stablized ZrO_2 and perovskite oxides) will be presented and discussed.

9:30 AM

(ICACC-S10-041-2017) Solid-solution of dopants to "record" sintering and microstructural behaviors in structural ceramics (Invited)

H. Gu*[1]; 1. Shanghai University, China

Phase relations and microstructure behaviors are usually two separate properties of the structural ceramics, representing the inherent uniformity and the tailorable inhomogeneity of ceramic materials, respectively. Dopants, together with unavoidable impurities, are necessary sintering aides to densify the ceramic bodies for all sintering modes, which often remain at grain boundaries to form minor phase and/or intergranular glassy film (IGF). The later was found to have equilibrium thickness, and in 30 years it develops into a key property of ceramic microstructure, especially in understanding of mode change induced by the roughening transition of IGF. This is also the primary reason to establish the "complexion" scheme, forming effectively the interfacial "phase" diagram for structural ceramics. However, our measurements of dopant segregation to IGFs have pointed all to the transient behaviors in the "model" Si_3N_4 and Al_2O_3 as well as in the "practical" SiC, Sialon and TZP ceramics. Moreover, non-uniform solid-solution of dopants was frequently detected in the primary ceramic phases, correlating with microstructure in one way or another. In this talk, I shall report how the two properties are unified in a same picture to reveal the sintering mechanisms. This forms also the essential work of a key research program on structural ceramics in the science foundation of China.

10:20 AM

(ICACC-S10-042-2017) Evidence for percolation diffusion of cations and material recovery in disordered pyrochlore from accelerated molecular dynamics simulations (Invited)

R. Perriot*[1]; B. Uberuaga[1]; R. Zamora[2]; D. Perez[2]; A. Voter[2]; 1. Los Alamos National Lab, USA; 2. Los Alamos National Lab, USA

Pyrochlores, a class of complex oxides with formula $A_2B_2O_7$, exhibit high tolerance to radiation and fast ionic conductivity. Both properties are intimately linked to the cation structure and evolution of the disordered state, and it is therefore critical to investigate cation transport and defect interaction as a function of cation disorder. However, the inherently slow motion of the cations makes it a challenge to reach relevant timescales via atomistic simulations. We used accelerated molecular dynamics to characterize cation diffusion in $Gd_2Ti_2O_7$ as a function of the disorder on the microsecond timescale. We find that, while cation vacancy diffusion is slow for low levels of disorder, higher levels of disorder allow for fast cation diffusion, which is then also accompanied by fast antisite annihilation and reordering of the cations. The cation diffusivity is therefore not constant, but decreases as the material reorders. We find evidence that fast cation diffusion is triggered by the formation of a percolation network of antisites. These results highlight the dynamic interplay between fast cation diffusion and the recovery of disorder and have important implications for understanding radiation damage evolution, sintering and aging, as well as diffusion in disorder oxides more generally.

10:50 AM

(ICACC-S10-043-2017) Use of ab-initio molecular dynamics and data mining for design and synthesis of novel nanomaterials (Invited)

R. Sakidja*[1]; 1. Missouri State University, USA

There have been remarkable new advancements in the atomistic-based computational modeling and computing power that enable us to design and to aid the synthesis processes of a wide variety of nano-scale materials. In particular the more recent use of ab-initio olecular dynamics (MD), which integrates the electronic structure calculations within the Density Functional Theory approximation and the classical MD simulations, coupled with the data mining approach, has provided us with the capability to tailor materials properties so as to yield novel functionalities. This approach has been demostrated in the design and synthesis of a variety of 2D & core-shell nanomaterials as well as meso-porous structures. There remains however a big challenge ahead in accelerating the computational simulations and several possible paths forward will be discussed.

11:20 AM

(ICACC-S10-044-2017) Polishing Simulation of Gallium Nitride Substrate Assisted by Chemical Reactions with Hydroxyl Radicals

K. Kawaguchi*[1]; T. Igarashi[1]; Y. Higuchi[1]; N. Ozawa[1]; M. Kubo[1]; 1. Institute for Materials Research, Japan

Gallium nitride (GaN) is a promising material for electronics because it has a wide band gap and high saturation electron speed. On the other hand, the GaN substrate is difficult to be polished because it has a high hardness and chemical stability. The high efficient and precise polishing process for planer GaN substrate is required. Some experiments suggest that the GaN substrate is well polished by using the OH radicals, which are generated by an ultraviolet excitation at the friction interface. However, the detailed polishing mechanisms are still unknown because it is composite process using chemical reactions and mechanical polishing. Thus, we performed polishing simulations of a GaN substrate by a SiO_2 abrasive grain with OH radicals in order to reveal effects of chemical reactions on the polishing process. We use our tight-binding quantum chemical molecular dynamics code. The OH radicals react with the GaN substrate and a surface-adsorbed O atom is generated. Then, it is mechanically pushed into the GaN substrate by the abrasive grain at the friction interface. This O atom intrusion induces the dissociation of Ga-N bonds. After the intrusion of surface-adsorbed O atom is repeated, volatile N_2 molecules are generated and desorbed from the substrate. The abrasive grain contacts with the reacted GaN substrate and remove a Ga atom by the generation of Si-O-Ga bridge bonds.

11:40 AM

(ICACC-S10-045-2017) Using Virtual Manufacturing to Simulate the Changing Electrical Properties of Ceramics during Sintering

M. C. Golt*[1]; S. M. Kilczewski[2]; 1. U.S. Army Research Laboratory, USA; 2. Bennett Aerospace, USA

The electrical properties of sintering ceramics change with the evolving microstructure. As such, the major stages of sintering (neck formation, pore removal, grain boundary dewetting, grain growth, etc.) could be detected through changes in a bulk measurement of the electrical impedance. To understand the influence these stages have on the electrical properties of various ceramic systems, a virtual manufacturing approach was adopted that simulates microstructure evolution through the major stages of sintering and computationally determines how these stages affect the transport of electrical charge through the structure. A furnace model was developed to simulate the PID-based control and response of a furnace to develop realistic processing temperature profiles. These thermal profiles are input to a Monte Carlo Potts model which includes pore removal to simulate the sintering microstructure from a powder compact, given the temperature profile. Finally, a finite-element method is used to determine the electrical transport (D/C and A/C currents) through the simulated structure at each time step. Simulated results are compared to electrical characterizations performed on alumina during sintering, however the approach is fully parametric to simulate the relationship between the electrical properties and the sintering characteristics of many ceramic systems.

S13: Advanced Materials for Sustainable Nuclear Fission and Fusion Energy

Standards and Mechanical & Physical Properties

Room: Coquina Salon H

Session Chairs: Weon-Ju Kim, Korea Atomic Energy Research Institute; Michael Jenkins, Bothell Engineering and Science Technologies

8:30 AM

(ICACC-S13-018-2017) Developing Codes and Specifications for Ceramic Composites in Nuclear Reactors within the ASME BPV Framework (Invited)

S. T. Gonczy*[1]; Y. Katoh[2]; M. Mitchell[3]; M. G. Jenkins[4]; 1. Gateway Materials Technology, USA; 2. Oak Ridge National Lab, USA; 3. Eon Consulting, South Africa; 4. California State University Fresno, USA

Ceramic matrix composites are being developed for nuclear fuel cladding and for high temperature reactor components, offering higher temperature capabilities and improved durability compared to metals. The ceramic composite components will have to meet the stringent licensing and operational regulations of the United States Nuclear Regulatory Commission (NRC). The NRC uses provisions of the American Society of Mechanical Engineers (ASME) Boiler and Pressure Vessel (BPV) Code as a major part of the framework to establish the design, fabrication, construction, testing, and performance requirements for structures, systems, and components important to nuclear safety. The ASME codes are extensively detailed documents that address engineering factors (classification, materials specification, life design and qualification, testing and NDE, fabrication and installation, in-service inspection, etc.), as well as management and regulatory issues (legal responsibilities of designers, producers, and owners; certification requirements; quality assurance systems). The ASME BPV Graphite and Composites Subgroup is writing the codes, specifications, and test standards for carbon-carbon and SiC-SiC composites for nuclear fission applications. This presentation will review the progress and work plans for these development efforts.

8:50 AM

(ICACC-S13-019-2017) Composition, Structure, Manufacture, and Properties of SiC-SiC CMCs for Nuclear Applications: Update on Evolving Chapters in the ASME BPV Code Section III, Division 5, Working Group On Graphite & Composites (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 reactors planned by US DOE will use SiC/SiC CMCs to enhance fuel performance and improve accident tolerance. SiC/SiC CMCs are tolerant of the irradiation and chemical environments of current and future nuclear reactors. Because SiC/SiC CMCs are nonconventional materials, a concern with these plans involves the mission of the US Nuclear Regulatory Commission (NRC) which licenses and regulates the civilian use of nuclear materials. NRC regulates nuclear reactors and new reactor design as well as the reactor materials. NRC is legally required to use, consensus codes and standards as an integral part of the regulatory process. Note that ASME Boiler and Pressure Vessel Code (BPVC) Section III "Rules for Construction of Nuclear Components" (which codifies and standardizes acceptable materials for nuclear applications) is included in the NRC regulations. Therefore, for SiC/SiC CMCs to be incorporated into current and future nuclear reactors, they must be included in ASME BPVC as acceptable materials. This presentation provides an update on evolving chapters from ASME BPVC Section III, Division 5 (High Temperature Reactors), Working Group on Graphite and Composites regarding the composition, structure, manufacture, and properties of SiC/SiC CMCs.

9:10 AM

(ICACC-S13-020-2017) Thermal conductivity mapping of SiC-SiC composites with sub-micron spatial resolution

J. Brethauer*[1]; D. Cahill[1]; 1. University of Illinois at Urbana-Champaign, USA

Understanding the thermal conductivity of SiC-SiC composites is critical for their optimal use as fuel cladding in nuclear reactors. In this work, we use time domain thermoreflectance (TDTR) to measure the thermal conductivity of the components of a SiC-SiC composites at temperatures between room temperature and 300C. The composite consists of ≈10 um diameter Hi-Nicalon type S ceramic fibers that are bound to a SiC matrix with a pyrolytic carbon/SiC multilayer interphase. The matrix has an average thermal conductivity of 34 W m-1 K-1, and the fibers have an average thermal conductivity of 19 W m-1 K-1 at room temperature. We map the thermal conductivity of a cross section of a fiber at a resolution of <1 um and find that it changes by up to 60% from its core to its outer edge.

9:30 AM

(ICACC-S13-021-2017) Strength Retention and Microstructure Evolution SiC_f/SiC_m Composites Following Heat Treatment in Argon up to 1800°C

C. P. Shih*[1]; H. Khalifa[1]; G. Jacobsen[1]; C. Deck[1]; 1. General Atomics, USA

Both transient eutectic phase (TEP) and chemical vapor infiltration (CVI) processed continuous silicon carbide (SiC) fiber reinforced SiC matrix (SiC_f/SiC_m) composites are stable under irradiation and have previously been shown to have good strength retention up to 1600°C, making them promising candidate structural materials in nuclear systems. However, even higher temperatures could occur during accident scenarios, and strength retention and microstructure evolution at temperatures up to 1800°C have not been studied in detail. In this work, the room temperature flexural strength and microstructure of nuclear grade TEP and CVI SiC_f/SiC_m composites were investigated following heat treatments up to 1800°C in argon. Both composites showed excellent performance after the high temperature exposure, retaining 80 to 85% of the as-fabricated strength following 1700°C heat treatment. Stress-displacement curves and microstructure evolution observations were used to better understand potential causes of these small changes in mechanical response. These results suggests that TEP and CVI processed SiC_f/SiC_m composites will have much improved strength retention under high temperature accident conditions compared to other nuclear structural materials, such as Zircaloy and other accident tolerance fuel (ATF) candidate materials.

10:10 AM

(ICACC-S13-022-2017) SiC-SiC Tube Mechanical Behavior of SiC-SiC Tubes at High Strain Rates During Reactivity Insertion Accidents

M. N. Cinbiz*[1]; R. R. Lowden[4]; N. Brown[3]; G. Singh[2]; K. Terrani[1]; 1. Oak Ridge National Laboratory, USA; 2. Oak Ridge National Lab, USA; 3. Oak Ridge National Lab, USA; 4. Oak Ridge National Lab, USA

Silicon-carbide fiber silicon-carbide matrix composites (SiC/SiC) are one of the candidate accident tolerant nuclear fuel cladding materials that are considered for light water reactors because of their outstanding high temperature steam oxidation resistance. As part of a holistic assessment of the potential for enhanced accident tolerance, the mechanical performance of SiC/SiC composites must be assessed during the low temperature phase of a super prompt reactivity insertion accident. During a postulated reactivity insertion event, a large deposition of energy in the UO_2 fuel causes it to expand very rapidly (within 20 to 100 μs). If pellet-cladding mechanical interaction occurs this causes a strain-controlled cladding expansion. To investigate the mechanical behavior under similar conditions to reactivity insertion event, a mechanical frame has been designed that a rapid axial pin movement is converted into a driver tube deformation in the radial direction, and the sample expansion is caused by that driver tube expansion, such as reactivity insertion event. In this study, the mechanical frame design will be introduced and the mechanical response of SiC-SiC composites that are subjected to strain pulses will be investigated. The results will focus on the mechanical response under expected reactivity insertion conditions and limiting conditions.

10:30 AM

(ICACC-S13-023-2017) Strength of SiC_f-SiC_m Composite Tube under Uniaxial and Multiaxial Loading

K. Shapovalov[1]; G. Jacobsen[2]; N. Truesdale[1]; C. Deck[2]; X. Huang*[1]; 1. University of South Carolina, USA; 2. General Atomics, USA

The authors report a systematic study of the mechanical strength of nuclear grade silicon carbide fiber reinforced silicon carbide matrix composites tubing under several different stress states. The composite tubing is fabricated via a chemical vapor infiltration process, and is intended for accident tolerant nuclear fuel cladding. Several experimental techniques were applied to evaluate the strength of the tubular samples. These includes uniaxial tension, elastomer insert burst test, hydraulic bladder burst test, closed-end burst test, and torsion tests. These tests provided critical stress and strain at proportion limit and at ultimate failure. Strain gage and digital image correlation were applied to acquire quantitative information of deformation and damage events. Acoustic emission was monitored to help detect the inital matrix cracking. Based on these test results, a failure map was constructed for the SiC_f-SiC_m composites.

10:50 AM

(ICACC-S13-024-2017) Evaluation of Mechanical Properties of SiC based Materials through Non-Destructive Technique

G. Singh*[1]; K. Terrani[1]; Y. Katoh[1]; 1. Oak Ridge National Lab, USA

The development of SiC clad technology for accident tolerant fuel-clad systems will require extensive knowledge of the mechanical properties of SiC based materials. It is also important to understand how these mechanical properties are related to the other physical properties. Non-destructive techniques are appropriate for this purpose. The current work focuses on using non-destructive techniques to evaluate the mechanical properties of SiC based materials.

11:10 AM

(ICACC-S13-025-2017) Damage Tolerance of SiC_f-SiC_m Composite Plates under Low Velocity Impact

L. Alva[1]; D. McCleeary[1]; H. Zhao[1]; H. Khalifa[2]; J. Clifford[1]; P. Majumdar[1]; X. Huang*[1]; 1. University of South Carolina, USA; 2. General Atomics, USA

Low velocity impact damage analysis of SiC_f-SiC_m composites is conducted using the drop weight impact test method at room temperature. The specimens include composite plates manufactured by transient eutectic phase assisted press sintering and chemical vapor infiltration process. Impact tests with different drop heights are conducted to evaluate the damage under different impact loads and energy. Optical microscopy was employed to examine the surface cracking patterns and X-ray tomography was used to visualize the distribution of the cracks through the thickness of the damaged specimen. The impulse excitation technique was used to determine the evolution of the natural frequencies and internal damping after impact loading. Finally, a novel burst test method was used to evaluate the residual strength of the impacted sample under bi-axial loading. Compared to monolithic ceramic, SiC_f-SiC_m composites demonstrate much improved tolerance to low velocity impact. Thick composite plates retained significant residual strength after impact loading. Typical quasi-ductile failure modes such as fiber pull-out and delamination were observed. It was also found that the natural frequency and internal damping closely follow damage evolution in the specimen.

11:30 AM

(ICACC-S13-026-2017) Hoop Tensile Strength of CMC Tubes for LWRS Applications: ASTM Draft Standard Using Direct Pressure

M. G. Jenkins*[1]; J. Salem[2]; J. E. Gallego[1]; 1. Bothell Engineering and Science Technologies, USA; 2. NASA Glenn Research Center, USA

Conventional zirconium-alloy fuel rod tubes in light water reactors (LWR) will be replaced by US DOE with those consisting of SiC/SiC CMCs to enhance fuel performance and accident tolerance. SiC/SiC CMCs are tolerant to irradiation and chemical environments of LWRs. Failure modes in LWR fuel cladding include loss of gas tightness and reduced mechanical integrity due to the build-up of internal gas pressure and the swelling of fuel pellets. Therefore, determination of the hoop tensile strength of tubes is critical for evaluating SiC/SiC CMC fuel claddings. One full-consensus ASTM test method (C1819) using internal pressurization obtained from expansion of an axially-loaded elastomer insert was published in 2015. A second ASTM test method using internal pressurization obtained from pressurized fluid has been drafted and is undergoing ballot approval for publication. Both test methods use sound, theoretical analysis of the stresses developed in tubes subjected to internal pressure over a finite length inside a semi-infinitely long tube. Test specimen dimensions, testing geometries, test conditions and results interpretation based on this theory and subsequent empirical tests are addressed. These test methods are intended for material development, material characterization, material screening, model validation, and quality assurance.

3rd Pacific Rim Engineering Ceramics Summit

Challenges and Opportunities III

Room: Coquina Salon C

Session Chairs: Yiquan Wu, Alfred University; Tohru Sekino, Osaka University

1:30 PM

(ICACC-PACRIM-038-2017) Development of SnO_2-TiO_2 Spinodal Composites and Their Semiconductor Properties (Invited)

W. Jiang[1]; T. Goto[1]; S. Cho[1]; T. Sekino*[1]; 1. Osaka University, Japan

SnO_2-TiO_2 binary system is well-known to form unique lamella structure via spontaneous spinodal phase separation by appropriate annealing. If semiconductive properties of these two phases can be controlled simultaneously with the lamella structure formation, novel oxide composites having unique semiconductor properties might be developed. In this study, we have thus focused on the simultaneous tuning of 2-dimensional lamella structure and semiconductor properties. We have found that doping of small amount of Fe^{3+} accelerated the lamella formation that occurred only by a sintering process, and semiconductor properties could be varied depending on the process parameters. However, detailed electrical properties of constituent two phases in lamella have not yet clarified. In this study, we have prepared various SnO_2-TiO_2 binary system with lamella structures and corresponding constituent phases (i.e., M-doped SnO_2 and TiO_2 phases) and investigated their semiconductor properties. It was found that the 4mol%Fe-doped TiO_2 showed megative temperature coefficient (NTC) characteristics for the resistivity and exhibited n-type nature at elevated temperature, while its mobility was rather low. Detailed semiconductive properties and their lamella formation development in relations with composition and fabrication processing for various samples will be discussed.

2:00 PM

(ICACC-PACRIM-039-2017) The dependency of crystal growth on grain size during solid-state single-crystal conversion (Invited)

Y. Liu[1]; Y. Wu*[1]; 1. Alfred University, USA

The surface energy in faceted polycrystalline material is critical in controlling the grain growth and grain boundary. In this study, the interrelation between polycrystalline size and crystal growth was investigated in a single-polycrystal system, in which a seed crystal was bonded to a polycrystalline matrix and grew towards the ceramic matrix via solid-state conversion. Among many problems awaiting solutions to promoting the single crystal growth at solid-state conversion, one problem is the limited crystal growth even after long period of heat treatment. We herein focused on the observation of grain growth and simultaneous seed crystal growth under isothermal conditions. A stagnant stage for both grain growth and seed crystal growth was formed and it was found that the dimension of the grown crystal depended on the grain size in the matrix rather than treatment temperature and duration. The role of critical grain energy, stemming from material intrinsic properties and heat treatment parameters, is less effective in the case studied in this work. This preliminary study is expected to draw more attention on the grain growth kinetics in order to grow larger size crystals by solid-state method.

2:30 PM

(ICACC-PACRIM-040-2017) Low-Temperature Spark Plasma Sintering of Ceramics by Using SiC Molding Set (Invited)

B. Kim*[1]; A. Dash[2]; Y. Kim[3]; K. Morita[1]; H. Yoshida[1]; Y. Sakka[1]; 1. National Institute for Materials Science (NIMS), Japan; 2. Katholieke Universiteit, Belgium; 3. University of Seoul, Republic of Korea

Hydroxyapatite (HAP) and alumina ceramics were sintered at low temperatures by using the SiC molding set during spark plasma sintering (SPS). Transparent ceramics were obtained at 800 C and 1000 C for HAP and alumina, respectively. The SiC is electrically conductive, so that the molding set was directly heated during SPS without external heating. Compared to the conventional graphite molding set, the voltage level during heating was higher due to lower conductivity. When the graphite molding set was used, the temperature required for transparent HAP and alumina was about 950 C and 1150 C, respectively. The SiC molding set lowered the temperatures for transparent ceramics by 150 C. For transparent HAP, the transmission is almost similar between the samples sintered at 800 C with the SiC set and at 950 C with the graphite set. For transparent alumina, the transmission is considerably affected by the heating rate. The dependence on the heating rate is explained by considering the effect of applied voltage during SPS. The high voltage level seems to enhance the diffusion during sintering. At a heating rate of 50 C/min, the transmission of the alumina decreases due to the accelerated grain growth, and the delay behavior of dynamic grain growth was also observed. In this study, the effects of the SiC molding set were examined on the densification temperature, transmission, voltage level and grain growth.

3:20 PM

(ICACC-PACRIM-042-2017) Teaching and research in ceramics supported by CES EduPack and CES Selector software (Invited)

E. Bernardo*[1]; L. Masi[2]; 1. University of Padova, Italy; 2. Granta Design Limited, United Kingdom

The most appropriate material for a given application can be rapidly selected by exploring material property graphs, according to Ashby's procedures. Among these graphs, two categories can be seen as the most interesting. In the first case, the performance of a component, with a given geometry, relies on combinations of two properties (e.g. elastic modulus and density, or strength and density), easily represented as lines in log-log plots, traced by means of CES (Cambridge Engineering Selector) software. In the second case, a component is aimed at expressing a combination of conflicting objectives (e.g. low mass and low cost, low mass and low thickness), represented in another series of log-log plots, that could be more advantageous than that of first reference solution. Starting from these material property graphs, the present paper aims at evidencing the advantages of (especially porous) ceramics in high value applications. In addition, the graphs will be presented as a way to verify the actual applicability of newly developed ceramics.

3:50 PM

(ICACC-PACRIM-017-2017) Progress in Compact, Ceramic Heat Exchangers (Invited)

C. Lewinsohn*[1]; J. Fellows[1]; R. Braun[2]; R. Kee[2]; 1. Ceramatec, Inc., USA; 2. Colorado School of Mines, USA

Ceramatec has developed compact, ceramic heat exchangers for application at high-temperature. These heat exchangers will improve efficiency in a variety of processes related to energy production and utilisation. Applications include microturbines, turbines for stationery power and propulsion, concentrated solar power cycles, and high-temperature nuclear applications. Ceramatec's approach is to build modular units so that testing on small scale units is relevant to larger scale applications. This presentation will discuss the benefits of ceramic heat exchangers, analysis of microchannel design parameters, fabrication methods, and testing at scales up to 5kW. Materials selection will be discussed. The results that will be presented validate analytical and computational fluid dynamics modeling. Likewise, effective heat transfer will be shown. Requirements and plans for additional validation testing will also be described.

4:20 PM

(ICACC-PACRIM-043-2017) Challenges in Development of Easy-to-use Torsion Test Method for Bioceramics: Toward ISO Standard Proposal (Invited)

K. Yasuda*[1]; S. Tsutsumi[2]; 1. Tokyo Institute of Technology, Japan; 2. Kanazawa Institute of Technology, Japan

Much attention has been paid to tensile fracture in ceramics for long time; however, its shear fracture also becomes more and more important in recent ceramic applications. For example, power electronics ceramic substrates are functionally damaged by shear fracture near the electrodes, and bioceramics are sometimes fatally broken in torsion in clinical cases. So, it is necessary to attain strength reliability in multi-axial stress state of ceramic devices and components. In this presentation, the authors introduce the development of an easy-to use torsion test method for bioceramics. Samples were apatite ceramics and its composites of which porosity ranged from 0 to 75%. The dog-bone type specimens were finished by machining, and set in an opening torque meter for PET bottles, together with cylindrical grip jigs. Although torsion tests were conducted by hand, spiral fracture surfaces were obtained, corresponding to the theoretical background of torsion fracture of brittle materials. And consistent data was obtained for torsion strength of each sample. Now, the authors are planning to propose this method to ISO standard for bioceramics. This work has been supported in part by METI, Japan.

FS1: Geopolymers, Chemically Bonded Ceramics, Eco-friendly and Sustainable Materials

Sustainable Materials and Novel Applications II

Room: Coquina Salon E
Session Chair: Henry Colorado, Universidad de Antioquia

1:30 PM

(ICACC-FS1-015-2017) Geopolymer Synthesis as a Low Energy, Zero Waste Route to Ceramics (Invited)

A. J. Steveson*[1]; W. M. Kriven[1]; 1. University of Illinois at Urbana-Champaign, USA

Geopolymers are traditionally synthesized under hydrothermal conditions near room temperature. The curing reaction can take 24 hours or longer depending upon the quality of the hydrothermal seal, relative humidity of the laboratory, reactivity of precursors, and curing temperature. Traditional synthetic routes to the crystalline analogues of geopolymers produce organic and nitrogenous waste byproducts and require high reaction temperatures (650-1100°C). In this study, we propose hydrothermal crystallization of geopolymers as a low energy, zero waste alternative to traditional processing routes. We report the effects of elevated curing temperature (up to approximately 250°C) on the reaction completion time, microstructure, and phase formation in sodium and potassium geopolymers.

2:00 PM

(ICACC-FS1-016-2017) Performance and durability of Fe-rich inorganic polymer composites with basalt fibers

A. Peys*[1]; M. Peeters[1]; A. Katsiki[2]; L. Kriskova[1]; H. Rahier[2]; B. Blanpain[1]; Y. Pontikes[1]; 1. KULeuven, Belgium; 2. Vrije Universiteit Brussel, Belgium

The use of inorganic instead of organic polymers in composites receives increasing interest from research institutions and industry because of their fire resistance and lower energy input upon manufacturing. It remains however unclear whether the fibers can resist the alkalinity of the inorganic polymer in a larger timeframe. This is investigated in the present work for basalt fibers. Inorganic polymers were synthesized from an iron-silicate slag, originating from non-ferrous metallurgy, and potassium silicate solutions (SiO_2/K_2O = 1.6-2.0, H_2O/K_2O = 16-24), which were mixed in a solution/slag weight ratio of 0.3. To this mixture, 3 wt.% of basalt fibers (12.7 mm long, 13 μm diameter) was added. Curing occurred at room temperature. The 3-point flexural strength as a function of time increased for all batches. The highest flexural strength was recorded when an activating solution with SiO_2/K_2O = 2.0 and H_2O/K_2O = 16 was used; values were 29 ± 1 MPa and 37 ± 5 MPa, after 7 and 90 days, respectively. Compared with the samples where no fibers were introduced, the strength gain is 290% for 7 days and 285% for 90 days. The results show that the basalt fibers retain their structural performance in time, suggesting that basalt fiber reinforced inorganic polymers can be durable. Additional experiments on samples cured for more than 90 days are currently undergoing to provide more insights.

2:20 PM

(ICACC-FS1-017-2017) Reactivity of Amazonian Metakaolin for Geopolymer Synthesis (Invited)

R. A. Sa Ribeiro*[1]; G. P. Kutyla[2]; M. G. Sa Ribeiro[1]; W. M. Kriven[2]; 1. INPA-National Institute for Amazonian Research, Brazil; 2. University of Illinois at Urbana-Champaign, USA

Metakaolin has been shown to be an important precursor material for geopolymer synthesis. Importing commercial metakaolin for construction applications may carry an unsatisfactorily high cost. To determine the viability of using a local resource for geopolymer synthesis, geopolymers (GPs) were synthesized using metakaolin made from clay mined in the Amazonian region of Brazil. Samples were made with mixed potassium-sodium and pure sodium metakaolin-based GP. Samples were also made using commercial (high-purity, high-reactivity) metakaolin (MKC) as a comparison to the Amazonian metakaolin (MKA). Characterization of the MKA showed it to be about 76% pure, with the remainder of the material being crystalline silica. Compression and flexural strength tests of the resulting GP materials were carried out according to ASTM standards. Scanning electron microscopy was used to investigate the microstructure of the materials. X-ray diffraction was used to confirm the formation of GP. The mechanical properties of the MKA material were nearly equivalent to those based on MKC. Neither the MKC nor the MKA reacted completely, although samples made with MKC showed less unreacted material. Increasing the mixing intensity and duration, the amount of residual unreacted material was substantially reduced, and mechanical properties were improved. This work has shown the viability of using local resources for producing geopolymers.

2:40 PM

(ICACC-FS1-018-2017) Preparation and properties of a potentially useful new pseudo-geopolymer material

G. P. Kutyla[1]; C. Marsh[2]; W. M. Kriven*[1]; 1. University of Illinois at Urbana-Champaign, USA; 2. ERDC-CERL, USA

It was discovered that mixtures of kaolin-based clay, talc, and sodium waterglass could be dried at 50°C to give a material with a 3-point flexure strength around 20 MPa. While compositionally similar to a geopolymer, geopolymerization does not appear to take place in this material. The lack of aluminosilicate cross-linking is apparent when upon submersion in water, the silicate species re-dissolve, causing the material to revert to a clay/powder state. Additionally, the material fails in a semi-ductile fashion, while geopolymer is brittle. From these observations, it seems that this new material is actually just clay particles bound together by sodium silicate. Although a weakness to water seems to be a critical flaw for application of this material, firing it to temperatures as low as 150°C provides it with water resistance. Additional water-passivation methods were attempted involving CO_2 and bicarbonate salts with some levels of success. Due to its reasonable strength, inexpensive precursors, and low temperature heat treatment, this new type of material could be useful for a variety of structural applications.

3:20 PM

(ICACC-FS1-019-2017) Wollastonite Based Phosphate Cement Blended with Ceramic Powders from Primary Battery Waste (Invited)

H. A. Colorado*[1]; 1. Universidad de Antioquia, Colombia

Primary batteries such as the alkaline and zinc-carbon types, after their life-cycle, are typically composed from zinc and manganese oxides, steel, carbon, plastic and paper. After a grinding process, zinc and manganese oxides can be separated and used to fabricate phosphate cements. Phosphate cements are alternative cement produced upon the reaction of an acid and a base in a process entirely conducted at room temperature in our method. In this paper, different loading contents of battery waste are mixed with wollastonite based phosphate cement in order to analyze the waste effect in microstructure, mechanical behavior, and setting time. For this, scanning electron microscopy, X-ray diffraction, X-ray fluorescence, and thermo-gravimetric analysis characterization were conducted in this research. Compressive strength and density tests were also evaluated.

Composites and Conversion to Ceramics

Room: Coquina Salon E

Session Chair: Ange Therese Akono, University of Illinois at Urbana-Champaign

3:40 PM

(ICACC-FS1-020-2017) Geopolymers Reinforced with Auxetic Fabrics

K. Sankar*[1]; W. M. Kriven[1]; 1. University of Illinois at Urbana-Champaign, USA

An explosion sending a blast wave can cause catastrophic damage to a structure and to people in it. The design of new, smart and lightweight materials and structures for energy-absorbing purposes is complex, requiring the structure to be able to withstand and mitigate blast loadings whilst still being lightweight. Auxetic materials are a new class of materials that can be defined as solids that possess negative Poisson's ratio. Geopolymers are inorganic polymers that are refractory and can be processed at ambient temperatures. They have very low density, high durability, excellent mechanical properties and can be processed at ambient temperatures. However, pure geopolymers are brittle and have low fracture toughness. In this work, auxetic fabrics are used to reinforce geopolymers to make a new, smart and lightweight composite material for energy-absorbing purposes. The mechanical properties of this composite are investigated by performing tensile, flexure and impact testing according to ASTM standards. SEM and EDS were performed to observe the fiber-matrix interface.

4:00 PM

(ICACC-FS1-021-2017) Ultra Refractory, Chopped Basalt Fiber-reinforced Geopolymer Composites

P. F. Keane[1]; J. S. Foltz*[2]; C. Marsh[1]; W. M. Kriven[2]; 1. Engineering Research and Development Center, USA; 2. University of Illinois at Urbana-Champaign, USA

Basalt is a common volcanic rock found all around the world. The abundance of basalt has attracted attention from construction firms and material researchers as an alternative reinforcement source. Potassium geopolymer using the stoichiometric composition of $1\ M_2O * 1Al_2O_3 * 4SiO_2 * 11H_2O$ was produced using fumed silica, deionized water, potassium hydroxide, and metakaolin. One inch chopped basalt fiber tows were received from TechnoBasalt in Kiev, Ukraine, and were added to potassium geopolymer in amounts of 0, 2, 5, and 10 weight percent. Sample geometries were ½" x ½" x 6" in dimensions. Five samples from each basalt weight class were heated to 700, 1000, 1200 and 1400°C. The ramp up and down rates were 7 C/min and a one hour soak time was held for each set temperature. Three point flexure strength data was then taken using a universal testing system. Scanning electron micrographs were used to investigate the microstructures including particle sizes and size distribution as well as failure modes. Density and porosity were determined by the Archimedes and pycnometry methods before and after heating.

4:20 PM

(ICACC-FS1-022-2017) Production and thermo-mechanical characterization of wool-geopolymer composites (Invited)

A. Natali Murri*[1]; E. Landi[1]; 1. CNR ISTEC, Italy

Thanks to its specific properties, sheep wool is particularly suitable for the design of insulating systems, especially if coupled with other materials that may impart additional mechanical properties. Also, if considered as a potential replacement of other synthetic fibrous materials, wool has the additional benefit of being completely hazard-free for human health. In this study thermal insulating boards made of a wool/geopolymer composite with load bearing and fire resistant properties were produced and characterized. Two formulations with different amounts of fibers, corresponding to 23 vol% and 31 vol% in the final composites, were tested. The composites exhibited an average density of 1.0 g/cm^3, with a thermal conductivity of 0.2 W/(mK) and compressive and flexural strength around 9 and 5 MPa respectively. The bending strength and fracture behavior of the material was improved by the presence of the fibers, which promoted the onset of a toughening mechanism. Results showed that a geopolymer matrix loaded with 23 vol% of wool fibers is suitable as a fireproof barrier, since reaction to fire is in class A2 (UNI-EN 13501-1), and as structural insulating partitions for buildings, as it ensures a consistent load bearing ability, coupled with thermal insulating properties comparable with those of other man-made fiber products, with a considerable gain in terms of cost and environmental impact.

4:40 PM

(ICACC-FS1-023-2017) TEM Studies of Silicon-Based Ceramic Nano-Particles Synthesized from Potassium Geopolymers

C. Bagci*[1]; W. M. Kriven[2]; 1. Hitit University, Turkey; 2. University of Illinois at Urbana-Champaign, USA

Nano-scale silicon-based ceramic powders were synthesized by heating carbon added potassium geopolymers over the temperature of 1400°C. By using typical geopolymer route at room temperature, 9 or 18 moles of carbon nano-powder were incorporated into pure potassium geopolymers at the stage of KGP resins or powdered KGPs after curing to make KGP carbon precursors, respectively. Prepared KGP carbon precursors were then fired in an atmosphere controlled tube furnace at temperatures of 1400°-1600°C for 2 h under dynamic high purity nitrogen (99.99%). different type SiALON compound and Si_3N_4 particles with various α/β-Si_3N_4 ratio were achieved in the case of low and high carbon conditions, respectively. TEM studies corresponding selected area patterns (SAD) were used to analyze these present ceramic powders in microstructure. The nanocrystals of these silicon based ceramics (100-200 nm) with different morphologies and the crystallite size were also determined. α and β-Si_3N_4 phases and SiALON compounds were identified from the crystallographic projection.

5:00 PM

(ICACC-FS1-024-2017) High temperature microstructural integrity and enhanced mechanical properties in bone ash/glaze frit reinforced geopolymer composites (Invited)

A. W. Bhuiya*[1]; K. Sankar[1]; D. R. Rodriguez[1]; W. M. Kriven[1]; 1. University of Illinois at Urbana-Champaign, USA

Natural bone ash (calcined) and glaze frit have been investigated for the reinforcement of potassium-based geopolymer. Bone ash and/or glaze frit as a particulate reinforcement were homogenously mixed to an amorphous three-dimensional aluminosilicate binding material known as geopolymer. A material composition of geopolymer composites with low density bone ash and glaze frit ranging from 5-15 wt. % were investigated to design a relatively light weight structural material with enhanced mechanical properties and structural integrity. Di-calcium phosphate (DCP), hydroxyapatite (HA) and glaze frit reacted with potassium based geopolymer and developed an interface with reduced porosity and structural integrity at elevated temperature. The geopolymer composites were thermally treated between1025°C and1235°C in steps of 35°C to investigate the microstructure, thermal resistance and mechanical properties. The mechanical properties such as compressive and flexure strength were tested according to ASTM standards and compared with those of pure potassium-based geopolymer using MetaMax metakaolin and Mymensingh clay metakaolin (MW). The microstructure of the geopolymer composites were also investigated by powder X-ray diffraction (XRD) and scanning electron microscopy (SEM).

S1: Mechanical Behavior and Performance of Ceramics & Composites

Processing - Microstructure - Mechanical Properties Correlation II

Room: Coquina Salon D
Session Chairs: Mehrad Mehr, University of Florida; Shengfang Shi, Osaka University

1:30 PM

(ICACC-S1-061-2017) Development of multiscale structure and function controlled Al_2O_3/Ti nanocomposites (Invited)

S. Shi*[1]; T. Goto[1]; S. Cho[1]; T. Sekino[1]; 1. Osaka University, Japan

Compositing of ceramics such as Al_2O_3 with nano-sized metal particles has been shown improvement in mechanical properties and addition of new functions. These novel nanocomposites can be used as advanced structural parts and biomaterials. Ti metal has unique modulus and performs good physicochemical consistency with Al_2O_3, so in the current study, we are focusing on the development of Al_2O_3/Ti nanocomposites and properties investigation. In order to suppress interfacial reactions and oxidation of Ti to get homogeneous Ti nanoparticles, TiH_2, instead of metal Ti, was used as starting material. It was allowed to make "in-situ nanodispersion structure" of metals within ceramic host. At last, Al_2O_3/0-30vol.% Ti nanocomposites were fabricated by hot-pressing sintering (HPS) method at 1500°C with the purpose of improving mechanical properties and providing electrical properties with Al_2O_3 ceramics. XRD analysis revealed that there were no any Ti-Al intermetallic compounds, however Al-Ti-O solid solution and Ti-O oxide were further confirmed by EDS analysis. These nanocomposites showed superior densification. Microstructure analysis revealed homogeneous distribution of Ti particles. Mechanical properties and electrical resistivity of nanocomposites and their relations with composition and microstructures will be discussed.

2:00 PM

(ICACC-S1-062-2017) Microstructure and Toughening Mechanism of Al_2O_3 /ZrO_2 Eutectic Composite by Combustion Synthesis Under High Cooling Rate

Y. Zheng*[1]; W. Yu[1]; Y. Zheng[1]; J. Pan[1]; 1. Harbin Institute of Technology, China

Al_2O_3/ZrO_2 eutectic ceramics have been attracting a great deal of attention because of their excellent high temperature property due to the special fiber or lamellar structure. However, with previous method, it's difficult to fabricate centimeter-scale Al_2O_3/ZrO_2 eutectic with fine interphase spacing (<1mm). In this work, the Al_2O_3/ZrO_2 eutectic ceramic is prepared using combustion systhesis under high cooling rate and low pressure, commercial Al powers and $Zr(NO_3)_4$ are used as reactant. The composite can reach Φ30*40mm. As the fracture morphology of Al_2O_3/ZrO_2 eutectic composite shown, the pull-out of the ZrO_2 rod and crack deflection were the main fracture modes in Al_2O_3/ZrO_2 eutectic. And nano-micron ZrO_2 rods are entirely embedded in the Al_2O_3 matrix with a spacing of 280 nm, Due to nanostructured eutectic, pull-out and bridge effects of rod-like ZrO_2, the bending strength and fracture toughness of the eutectic ceramic composites reached 1060 MPa and 10.6 MPa×m$^{1/2}$, respectively.

2:20 PM

(ICACC-S1-063-2017) Effect of sintering additives and BN ratio on high temperature performance of BN particle dispersion SiC composites

S. Yanagawa*[1]; T. Hinoki[1]; K. Shimoda[2]; 1. Kyoto University, Japan; 2. National Institute for Materials Science (NIMS), Japan

SiC/SiC composites are expected for jet engine. But high temperature mechanical properties are limited by BN or C fiber/matrix interphase which is easily oxidized. Novel SiC/SiC composites were developed with BN particles in matrix instead of the interphase (SiC/(SiC+BN) composites). Previously we fabricated SiC/(SiC+BN) composites by liquid phase sintering using Al_2O_3+Y_2O_3 as sintering additive. However the formation of liquid phase above 1300°C was concern in terms of creep. Just Al_2O_3 was also selected as sintering additive to improve creep due to no liquid phase at 1500°C according to Al_2O_3-SiO_2 binary phase diagram. SiC/(SiC+BN) composites were fabricated with various sintering additives and the amount of BN. These samples were exposed at 1500°C in air. Microstructure was observed by FE-SEM and EPMA and strength was evaluated by tensile test. Oxidation behavior was more affected by the amount of BN rather than the kind of sintering additives. The strength degraded in composites with low BN ratio and didn't depend on the kind of sintering additives. The oxidation was prevented by relatively smooth scale which is mainly composed of $Y_2Si_2O_7$ and the strength degradation was limited in the composites with high BN ratio sintered with Al_2O_3 +Y_2O_3. The strength was maintained in the composites with high BN ratio sintered with Al_2O_3, even with further oxidation.

2:40 PM

(ICACC-S1-064-2017) Mechanical and microstructural properties of TiCN / nc-TiB_2/ nc–TiN cermet processed by Spark Plasma Sintering

B. Shanmugavel*[1]; S. Emarose[1]; R. Paskaramoorthy[2]; 1. Anna Univeristy, India; 2. University of the Witwatersrand, South Africa

Titanium carbonitride (TiCN) based cermets with different percent of nc-TiN and nc-TiB_2along with other secondary carbides were fabricated by Spark Plasma sintering (SPS). The composition of TiN and TiB_2 and was varied form 5 wt.% to 15 wt.%. Scanning Electron Microscopy (SEM) and XRD analysis were done. The cermets containing TiN were found to have better fracture toughness than those containing TiB_2. In contrast, the cermets containing TiB_2 were found to have better Vikers hardness values than those containing TiN. In order to study the machining performance, tools meeting SNGN120408 standard configuration were made from the sintered bodies. The tools were then used to perform turning operations on EN24 workpiece material at cutting speed of 11.87, 29.68, 71.46, 163.88 m/min under dry conditions. The cutting force was found to be high initially but then decreased at high speed for all cermets. At high cutting speeds (71.46, 163.88 m/min), the cermet with 5% TiN and 5%TiB_2 addition showed the minimum cutting force (8.97N). At high cutting speeds the surface roughness values were very small on the machined surface. After machining, the cermet work piece containing 15% TiB_2 was found to have good surface finish (1.12-4.28μm). This cermet also displayed concurrent improvement in both Fracture toughness (6.8MPa.m$^{1/2}$) and Vickers hardness value (18.1GPa).

3:20 PM

(ICACC-S1-065-2017) Mechanical Properties of Low Temperature (850°C) Pyrolysis Bonded Silicon Carbide (Invited)

M. Mehr*[1]; M. Elbakhshwan[2]; D. Sprouster[2]; S. Gill[2]; L. Ecker[2]; G. Subhash[1]; J. C. Nino[1]; 1. University of Florida, USA; 2. Brookhaven National Laboratory, USA

Pyrolysis bonding is a process that can consolidate SiC powders into desired shapes using a SiC preceramic polymer. The advantages of this method are low firing temperatures (~850°C), no firing pressure, minimal green body formation pressure, as well as being a near net shape process. The process has potentials for low cost complex shape structural components. Here, we present the microstructure and compositional evolution as the material is exposed to higher temperatures and the effect on the mechanical properties of SiC processed by pyrolysis bonding. We discuss avenues for further property improvement. Characterization of hardness and flexural strength with respect to firing temperature will also be discussed. Furthermore, the effect of the native oxide layers of the starting powder as well as the remnant free carbon from the preceramic polymer on the properties of the material will be described. The removal of the native oxide layer prior to synthesis using hydrofluoric acid shows improved properties as well as ~6% increase in relative density. The average Vickers hardness of the control samples were 4.59 GPa which, considering the porosity (~20%), is lower than the values predicted by the empirical porosity effect model in literature. However, with the removal of the native oxide layer this value increases to 9.37 GPa and is in agreement with the model.

3:50 PM

(ICACC-S1-066-2017) Anisotropy in functional and mechanical properties of SiC/GNPs and SiC/GO composites

O. Hanzel*[1]; J. Sedlacek[1]; R. Sedlak[2]; P. Sajgalik[1]; 1. Institute of Inorganic Chemistry, Slovak Academy of Sciences, Slovakia; 2. Institute of Materials Research, Slovakia

Graphene as a single layer of sp^2 bonded carbon atoms has excellent mechanical, tribological, thermal and electrical properties and due to that fact is widely used as a filler in various polymer, metal and ceramic matrix composites. This work focuses on silicon carbide (SiC) based composites with addition of 1, 3 and 5 wt.% of graphene nanoplatelets (GNPs) or as synthesized graphene oxide (GO). Granulated powders of silicon carbide with different amount of either graphene nanoplatelets or graphene oxide were prepared by ultrasonication and stabilisation of dispersion with subsequent spraying into the liquid nitrogen and drying in freeze dryer. This freeze granulation process makes possible to retain the homogeneous distribution of graphene achieved in the suspension also in the granulated powders. The composite granulated powders were sintered by rapid hot pressing (RHP) at 1800°C (heating rate 100°C/min) and load of 50 MPa for 5 minutes in vacuum. Reference powders without GNPs or GO were sintered also in HP at the similar conditions. Formation of in-situ graphene domains were observed in reference sample sintered in RHP. Electrical and thermal conductivity as well as mechanical and tribological properties of all samples were studied both parallel and perpendicular directions to the RHP pressing axis.

4:10 PM

(ICACC-S1-067-2017) Effect of Solids Loading and Particle Size on the Fracture Toughness of Wet Particulate Materials

M. L. Sesso*[1]; G. V. Franks[1]; 1. The University of Melbourne, Australia

The fracture toughness of ceramic materials in an important property commonly used to evaluate and classify their use for potential applications. Although many advanced ceramics undergo pre-processing in the wet state, including preparation of suspensions and subsequent casting or other forming operations, the fracture properties of these ceramics in the transitioning state from wet to dry has not been extensively studied. Understanding the fracture mechanisms that control the propagation of cracks through saturated, partially saturated and dry green body ceramics may offer additional insight into drying cracks. This work demonstrates a method of measuring the mode I fracture toughness of green body ceramics at various stages of saturation. In addition, the effect of solids loading and particle size distribution on the fracture toughness are discussed. Decreasing the saturation of the particulate body was accompanied by a decrease in fracture toughness, which was related to the interparticle forces and reduced plastic flow.

4:30 PM

(ICACC-S1-068-2017) High hardness and toughness cubic BN-WRe nanocomposite

T. Semenic*[1]; O. Sudre[1]; 1. Teledyne Scientific Company, USA

Cubic boron nitride (cBN) monoliths were fabricated using powders produced by high energy mechanical milling of hexagonal boron nitride (hBN). The powder exhibited a highly disordered atomic structure rich in sp^3 hybridized bonding. Conversion to cBN was achieved at pressure and temperature that were lower than those traditionally used in the direct conversion from hBN. The study investigated the effect of hBN starting particle size, milling time, powder-to-milling ball ratio. The amount of sp^3 bonding for milled hBN powders was determined using ^{11}B solid-state NMR. Consolidated compacts were also analyzed by XRD, Raman, and HRTEM. The results showed that hBN milled for 100 h contained 68% sp^3 bonding, consolidated at 6.5 GPa and 1450°C to full density and produced a nano-size cBN microstructure with hardness and fracture toughness comparable to that of a single crystal. Addition of a hard metallic phase of Tungsten-Rhenium alloy (WRe) milled together with hBN created a nanocomposite structure as a means to improve fracture toughness. Vickers hardness of 42 GPa and fracture toughness of 5 MPam^0.5 was achieved with a precursor that only contained 6 vol.% of WRe dopant. The hardness and fracture toughness of the composite can be tuned by controlling the amount of WRe alloy.

4:50 PM

(ICACC-S1-069-2017) Mechanical properties of WC-FeAl-Al_2O_3 composites and wear properties of a WC-FeAl-Al_2O_3 cutting tool against ductile cast iron

R. Furushima*[1]; 1. National Institute of Advanced Industrial Science and Technology (AIST), Japan

WC-FeAl composite is a candidate of alternative materials for WC-Co hard materials used as cutting tools or dies. Due to the fabrication process, WC-FeAl composite inevitably contains Al_2O_3 in its microstructure. The contained Al_2O_3 may decrease the strength and fracture toughness because the wettability of the FeAl against Al_2O_3 is poor at high temperature, leading to form weak interface between FeAl and Al_2O_3. On the other hand, Al_2O_3 is chemically stable, which is advantageous for cutting tool material because of low reactivity with various metals and alloys. However, the effect of Al_2O_3 on mechanical properties and wear properties of WC-FeAl composite (to be exact WC-FeAl-Al_2O_3) composite is not investigated in detail. In this study, dependence of mechanical properties of WC-FeAl-Al_2O_3 composites on the amount of Al_2O_3 was investigated and wear property of a WC-FeAl-Al_2O_3 cutting tool against ductile cast iron were examined by machining tests.

5:10 PM

(ICACC-S1-070-2017) Microstructural Effects on Glass-Ceramic Mechanical Response and Slow Crack Growth Behavior

K. T. Strong*[1]; S. X. Dai[2]; D. N. Bencoe[3]; T. Diebold[1]; K. G. Ewsuk[3]; 1. Sandia National Laboratories, USA; 2. Sandia National Laboratories, USA; 3. Sandia National Laboratories, USA

The average linear coefficient of thermal expansion (CTE) of lithium aluminosilicate glass-ceramic (GC) can be tailored by systematically modifying the thermal profile used to crystallize the starting amorphous material. This tunability is useful for applications such as designing and fabricating a matched expansion glass-ceramic (GC) to metal seal. In addition to producing GCs with different thermophysical properties, modifications to the thermal profile will produce GCs with drastically different microstructures. The objective of this study was to correlate the effects of microstructure on GC mechanical properties, and on slow crack growth (SCG) behavior in particular. Image processing analysis is used to quantify GC microstructures, as well as relationships between GC microstructure and mechanical response including fracture toughness, hardness, and strength. The SCG behavior was characterized by measuring the fracture strength at varying stressing rates. Finally, the use of the SCG parameters obtained from dynamic measurements to calculate the lifetime of GC materials under static stress conditions will be discussed. Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

5:30 PM

(ICACC-S1-071-2017) Effect of surface compressive stresses on the subcritical crack growth in glass-ceramic composites

R. Bermejo*[1]; C. Krautgasser[3]; P. Supancic[1]; F. Aldrian[2]; R. Danzer[1]; 1. Montanuniversitaet Leoben, Austria; 2. EPCOS OHG, Austria; 3. Materials Center Leoben GmbH, Austria

The strength of Low Temperature Co-fired Ceramics (LTCC), used as substrates in the production of electronic circuits, can be significantly affected by the environmental and thermal conditions under which the material is loaded in service. In this work, the strength degradation associated with Subcritical Crack Growth (SCCG) mechanisms is investigated in several multilayer LTCC architectures designed with surface compressive residual stresses. The magnitude of the residual stresses was tailored combining two LTCCs (with different ceramic fillers), varying the volume ratio of the two bulk materials utilized. Biaxial strength measurements were performed at room temperature in humid environment at different stress rates, using the ball-on-three-balls testing method. Experimental findings showed a clear increase in the characteristic strength corresponding to the compressive residual stress in the surface layer, as measured using photoluminescence spectroscopy. The strength distribution in dependence of the surface stresses in the outer layer can be represented by a three-parameter Weibull distribution, thus providing a "threshold strength" (i.e. minimum strength) for the material. Analogue, it is demonstrated that the use of compressive residual stresses in LTCCs causes a threshold intensity factor for the SCCG behaviour, below which no environmental assisted cracking can occur.

S3: 14th International Symposium on Solid Oxide Fuel Cells (SOFC): Materials, Science and Technology

Degradation / Modeling

Room: Coquina Salon G
Session Chairs: Prabhakar Singh, University of Connecticut;
Jeffry Stevenson, Pacific Northwest National Lab

1:30 PM

(ICACC-S3-052-2017) Modeling of Electrochemically Induced Degradation of Solid Oxide Fuel Cells and Electrolyzers (Invited)

A. V. Virkar*[1]; 1. University of Utah, USA

It is well known that electrochemical devices degrade under the passage of current. The degradation is more common during the electrolytic mode than during the galvanic mode. Electrolysis, an unbalanced cell in fuel cell mode, charging of a battery, an unbalanced cell in a battery during discharge, are all examples of the electrolytic mode. While coarsening of electrodes and other changes such as reaction between mating phases at contacts do occur, the fundamental reason for degradation is rooted in the thermodynamics of the system itself. Much of the research on electrolyte materials has focussed on developing materials with the highest possible ionic conductivity and the highest possible ionic transference number, experiments and theory demonstrate that electrolytes with some level of electronic conductivity increase the device stability without significantly lowering efficiency. The basis of this stability lies in coupling between fluxes and the role of Onsager equations. This presentation is on the modeling of degradation mechanism and kinetics. Role of electrolyte properties and specifically the role of electrode/electrolyte interfaces will be addressed. Various electrolyte materials, such as zirconia, ceria, and LSGM will be examined from the standpoint of performance and degradation.

2:00 PM

(ICACC-S3-053-2017) Microstructural and Chemical Evolution of Various LSM-YSZ Cathode Compositions during 12,000 h Exposure to 800-1000°C

J. S. Hardy*[1]; C. A. Coyle[1]; R. M. Webb[2]; N. L. Canfield[1]; J. W. Stevenson[1]; 1. Pacific Northwest National Laboratory, USA; 2. University of Portland, USA

LSM-YSZ cathodes made from 8YSZ [$Zr_{0.84}Y_{0.16}O_{2-\delta}$] mixed with LSM-20-98 [$(La_{0.8}Sr_{0.2})_{0.98}MnO_{3-\delta}$], LSM-20-95 [$(La_{0.8}Sr_{0.2})_{0.95}MnO_{3-\delta}$], LSM-20-90 [$(La_{0.8}Sr_{0.2})_{0.90}MnO_{3-\delta}$], or LSM-10 [$La_{0.9}Sr_{0.1}MnO_{3-\delta}$] were screen printed on YSZ electrolytes and sintered in the lab. They were then placed in furnaces together with several samples of a commercially available LSM-YSZ cathode at 800, 900, or 1000°C. One of each variation of cathode was removed from the furnace at each temperature after 1000, 2000, 4000, 8000, and 12000 hours and examined together with as-sintered cathodes in a field emission SEM using EBSD and EDS. The resulting maps were analyzed to characterize the morphological (including grain size, grain shape, and area fraction) and chemical (including composition and secondary phases) changes in each phase occurring due to thermal exposure without applied electrical current or potential. This work will aid in distinguishing changes observed in long term LSM-YSZ cell tests that may be attributable solely to thermal exposure. The effects of temperature and composition on cathode evolution in the absence of current and potential will be discussed.

2:20 PM

(ICACC-S3-054-2017) Performance Degradation of LSM-based Cathode in Solid Oxide Fuel Cells Poisoned by Chromium Vapor Species

R. Wang*[1]; M. Würth[2]; B. Mo[1]; U. Pal[3]; S. Gopalan[3]; S. Basu[3]; 1. Boston University, USA; 2. Technische Universität München, Germany; 3. Boston University, USA

Chromium (Cr) vapor species from chromia-forming alloy interconnects are known to cause performance degradation of cathodes in solid oxide fuel cells (SOFCs). Anode supported cells, which are comprised of a Sr-doped $LaMnO_3$ (LSM) + yttria-stabilized zirconia (YSZ) cathode active layer and a LSM cathode current collector layer, were electrochemically tested with direct and indirect contact of chromia-forming alloy interconnect. Oxygen and water vapor contents on the cathode side were varied to investigate the effects of Cr vaporization under different conditions. Current-voltage (I-V) measurement and impedance spectroscopy were used to characterize the performance of the cells. The polarization losses at the cathode under different experimental conditions were obtained by curve-fitting the measured voltage vs current density (V-i) plots to a polarization model. Microstructures of the cathode cross sections were examined to determine the location, composition and quantity of the Cr-containing deposits. Free energy minimization calculations and thermogravimetric experiments were performed to determine the nature and rate of Cr-containing vapor species that form over chromia-forming alloy interconnect. From these experimental results, the mechanism of Cr poisoning of the LSM-based cathode is proposed and possible mitigation strategies are suggested.

2:40 PM

(ICACC-S3-055-2017) Study of LSM and LSCF Stability in "Real-world" Cathode Environment by Thermodynamic and Experimental Approaches

B. Hu*[1]; S. Krishnan[2]; C. Liang[1]; S. Heo[1]; A. Aphale[1]; R. Ramprasad[2]; P. Singh[1]; 1. University of Connecticut, USA; 2. University of Connecticut, USA

The chemical and structural stability of Lanthanum Strontium Manganite (LSM) and Lanthanum Strontium Cobalt Ferrite (LSCF) have been studied using a combination of thermodynamic and experimental approaches. Pure LSM and LSCF cathodes have been electrochemically tested in Cr-containing humidified air using LSM/yttria doped zirconia (YSZ)/Pt and LSCF/gadolinium doped ceria(GDC)/Pt half-cells. A three-electrode mode was used for the evaluation of cathode performance. For the 100-hour tests, the LSCF/GDC/Pt half-cell exhibited a slight decrease of electrode performance while the LSM/YSZ/Pt half-cell exhibited a rapid decrease of electrode performance in an I-t curve. Select-Area scanning electron microscopy shows that Cr deposited mainly at LSCF surface whereas Cr species deposited predominantly at LSM/YSZ interface. Raman spectra show the $SrCrO_4$ formation on the posttest LSCF cathode but not on the posttest LSM cathode. The bulk reaction energetics calculated by the first principle and a linear programming indicate that the stoichiometric LSM remains unreacted for the whole range of experimental p_{CrO3} and temperatures (T) while the formation of $SrCrO_4$ was observed to be energetically favorable on $La_{0.9}Sr_{0.1}CoO_3$ cathode for the experimental p_{CrO3}-T range. The calculations show excellent agreement with experimental results.

3:20 PM

(ICACC-S3-056-2017) Microstructure degradation of Ni/CGO anodes for solid oxide fuel cells after long operation time using 3D reconstructions by FIB tomography

A. Zekri*[1]; M. Knipper[1]; J. Parisi[1]; T. Plaggenborg[1]; 1. University of Oldenburg, Germany

The performance of solid oxide fuel cells (SOFCs) as well as their lifetime are keenly dependent on electrode microstructure. In order to understand the microstructural evolution in SOFC cermet anodes, the effects of long exposure time (up to 20 000 h) under realistic operating conditions (T= 850°C, J= 190-250) on the kinetics of microstructural degradation are investigated for porous Ni/CGO anodes. Data acquired from 3D-tomography technique (FIB/SEM) was used in the quantification of various anode structures aged during different operating times (2 500 h, 15 000 h and 20 000 h), which offered an exact quantification of Ni-grain growth, phase-connectivity, tortuosity factor and triple phase boundary length (TPB_L). Furthermore, the representative volume element (RVE) was also studied and its dependence on aging time was confirmed. The results showed significant 3D microstructural changes in the anode with increasing operating time, including Ni-coarsening, changes in the connectivity of the ceramic part (CGO) and a significant decrease of triple phase boundary densities from 3.08 to 1.78 after 20 000 h. The quantitative results are then combined with additional qualitative observations which lead to a more complete description of the degradation phenomena in anodes.

3:40 PM

(ICACC-S3-057-2017) Life-testing of $Sm_{0.5}Sr_{0.5}CoO_3$-infiltrated $Ce_{0.9}Gd_{0.1}O_2$ oxygen electrodes: Effect of ZrO_2 atomic-layer deposition

J. Railsback*[1]; K. Zhao[1]; M. Y. Lu[1]; A. Call[1]; S. Barnett[1]; 1. Northwestern University, USA

Infiltrated oxygen electrodes for solid oxide cells have been able to deliver small area specific resistances at low temperature (T) for a range of mixed electronic-ionic conductors. However, the high surface area to volume ratios of the infiltrated particles provide a driving force for coarsening that occurs measurably over a

T range from 500-800°C. In a previous study of $Sm_{0.5}Sr_{0.5}CoO_3$ (SSC) infiltrated into $Ce_{0.9}Gd_{0.1}O_2$ (GDC), electrode performance degradation in accelerated life tests was mathematically described by combining electrochemical and coarsening models; the resulting model predicted that the range of useful SSC particle sizes and cell operating Ts were limited by coarsening. Here we aim to slow the degradation process, allowing reduced SSC particle sizes and useful operating T, by coating the electrodes with atomic-layer deposition (ALD) Zirconia layers. The ALD-ZrO_2-coated symmetric cells are subjected to accelerated life testing at 700-800°C over 1500 hours. The degradation rate is decreased for 10-nm ZrO_2 compared with the 5-nm ZrO_2 and uncoated cells. The degradation is fit well by the model, but with reduced apparent coarsening rate for the 10-nm-ZrO_2 coated electrodes. Electron microscopy of the pre- and post-test microstructures is presented and used to explore whether the ALD-ZrO_2 layers reduce coarsening or act via other mechanisms.

4:00 PM

(ICACC-S3-058-2017) Analysis of Electrochemical Processes in SOFC (Invited)

A. Weber*[1]; S. Dierickx[1]; J. Szasz[1]; E. Ivers-Tiffée[1]; 1. Karlsruhe Institute of Technology (KIT), Germany

Present high performance SOFCs exhibit complex multiphase electrodes. Their performance and durability is governed by material as well as microstructural properties. To deconvolute the different electrochemical processes in such cells and their impact on the cell performance, advanced measurement and data analysis methods are required. In this contribution our approach to develop SOFC models based on impedance spectroscopy, the DRT and a subsequent CNLS-fit will be discussed in view of transmission line models. They imply the coupling of electronic and ionic charge transport in the electrode materials, electrochemical reactions at phase boundaries and the impact of the electrode microstructure and thus enable a physicochemical meaningful modeling of multiphase electrodes. The parameterization of such complex equivalent circuit models solely based on electrochemical measurements is often ambiguous. An overlapping of anode and cathode processes in the frequency domain can even result in a misinterpretation and wrong assignment of loss mechanisms. We will show how additional information as electronic and ionic conductivity of the applied materials, the line specific charge transfer resistances of the three phase boundaries (evaluated by model electrodes) and microstructural parameters (obtained from FIB-tomography) reduce the degrees of freedom and support an accurate parameterization of a physicochemical meaningful electrode model.

4:30 PM

(ICACC-S3-059-2017) Electrochemical Impedance Spectroscopy of the LSM/YSZ Interface using Phase Field Theory

J. M. Vielma*[1]; T. Cheng[1]; Y. Wen[1]; 1. National Energy and Technology Laboratory, USA

Solid oxide fuel cells (SOFCs) are currently intensely investigated as a direct chemical to electrical energy device with high conversion efficiency and a clean power generator from fossil energy resources. However, the high operating temperature of SOFCs subjects the cell to conditions that shorten its lifetime, in particular, the corruption of the ideal interface from cation interdiffusion and chemical contaminates. To study the degradation, Electrochemical Impedance Spectroscopy (EIS) is a powerful technique that provides insight into the multiple components or processes across the cell, many of the coupled. To better understand the coupled processes, electrochemistry-based phase field models are developed for an impedance study of the electrode/electrolyte interface in SOFCs. The models incorporate electronic and ionic transport of diffusing species and chemical reactions at the interface. Electrocapillary theory is used for differential capacitance calculations of the electric double layer. A one-dimensional impedance model illustrates the impedance data for an electrode/electrolyte (LSM/YSZ) interface with and without a triple phase boundary. A second two-dimensional model expands on the one-dimensional model to illustrate the effect of the presence of secondary phases $La_2Zr_2O_7$ and $SrZrO_3$ within the LSM/YSZ interface.

S5: Next Generation Bioceramics and Biocomposites

Bioceramics III

Room: Coquina Salon F

Session Chairs: Tolou Shokuhfar, University of Illinois at Chicago; Pelagia Gouma, SUNY Stony Brook; Mohamed Rahaman, Missouri University of Science & Technology

1:30 PM

(ICACC-S5-016-2017) Dissolution Kinetics of Bioactive Glass Particles and Scaffolds in a Continuous Flow of Tris Buffer and SBF

L. Aalto-Setälä*[1]; O. Karlström[1]; M. Engblom[1]; L. Hupa[1]; 1. Åbo Akademi University, Finland

The goal of this study was to analyze in detail the ion release and dissolution kinetics of bioactive glasses in buffered solutions fed continuously through particles and scaffolds of bioactive glass S53P4. The granule size used was 300-500 μm, and the scaffolds were sintered at 720°C for one hour, leaving the surface of the scaffold struts crystallized. Fresh Tris and SBF buffer solutions were fed at the flow rate of 0.2ml/min through glass particles/scaffolds inside a reactor for 1-7 days. The pH of the solution after the reactor was measured and the concentrations of the ions dissolving from the glass were analyzed using ICP-OES from solution samples collected throughout the run time. The surfaces of the samples were studied using SEM-EDXA after the experiment in order to reveal the surface reaction type of the glasses. For both solutions, the pH changes were very similar, and from 3 days and on, no significant pH changes were measured for either. Still, for runs in SBF, the formation of hydroxyapatite is seen after 48 hours of flow, whereas for Tris there is none. This affects the dissolution: in SBF the dissolution rates slow down considerably after the HA formation; for Tris runs no similar barrier layer retarding glass dissolution. The scaffolds showed similar differences between Tris and SBF but the partial surface crystallization slowed down the dissolution.

1:50 PM

(ICACC-S5-017-2017) Production and applications of dicalcium phosphate dihydrate (DCPD) for precursor of fluorapatite (FAp) (Invited)

M. Tafu*[1]; 1. National Institute of Technology, Toyama College, Japan

Dicalcium phosphate dihydrate have used as precursor of hydroxyapatite (HAp) and fluoraptite (FAp) by using its transform reaction in an aqueous solution. We have applied the transform reaction of DCPD for immobilizing fluoirde in the environments such as water, soil, and industrial wastes. By using the DCPD reaction, a small amout of fluoride ion is immbilize into FAp crystal. For eco-solutions of the DCPD reaction, important points are not only its reactivity but also particle properties. We have appared that reactivity of the DCPD with fluoride ion was able to improve by hybridization with another calcium salts. And particle properties was controlable by selection of reaction condition on soft solution process. In this presentation, it was summarized our achivements on production and application of the DCPD for precursor of FAp.

2:10 PM

(ICACC-S5-018-2017) Bone-mimetic 3D Oxide Nanostructures for Next Generation Implant Surfaces (Invited)

T. Shokuhfar*[1]; 1. University of Illinois at Chicago, USA

Future prospects for nanobiomaterials in medical applications appear to be excellent. In orthopedic applications, there is a significant need and demand for the development of a bone implant that is bioactive and exhibits mechanical and surface properties comparable with those of natural, healthy bone. Particularly, implants with nanometer-sized surfaces have been receiving much attention recently due to their ability to mimic the dimensions of constituent components of natural bone. Bone-mimetic 3D oxide nanotubes have been developed and studied as novel surface modification that promote osteointegration and can be a successful alternative to conventional implants surfaces. In addition, these structures can serve as a suitable route for the development of drug-eluting medical implants for drug therapy regiments to prevent infection, or decrease inflammation. Drug release derived directly from such implant surfaces can reduce unnecessary side effects and increase efficiency.

2:40 PM

(ICACC-S5-019-2017) Tough and Strong Porous Bioactive Glass-Polylactic Acid Scaffolds for Structural Bone Repair (Invited)

M. N. Rahaman*[1]; 1. Missouri University of Science & Technology, USA

Bioactive glass has attractive properties for healing bone defects such as the ability to convert to hydroxyapatite, bond strongly to bone and soft tissues, and stimulate osteogenesis. Strong porous bioactive glass scaffolds can also be created by rapid prototyping techniques with compressive strengths comparable to human cortical bone. However, the low fracture toughness of bioactive glass scaffolds is a concern in structural bone repair. The development of strong yet tough composites composed of bioactive silicate glass (13-83) with a porous grid-like architecture and a biodegradable polymer (polylactic acid) coating is described. The effect of the coating properties and bioactive glass architecture on the strength and toughness of the composite scaffold will be discussed.

3:20 PM

(ICACC-S5-020-2017) High-throughput electrospinning of the next generation of biomaterials (Invited)

P. Gouma*[1]; 1. SUNY Stony Brook, USA

The work presented here addresses a novel nanomaterials processing method that we developed in my lab at Stony Brook, that is high-throughput electrospinning. The advantages of this nanomanufacturing process is that it can use processing recipes developed for single jet systems for a variety of composite materials, while providing a high product output. In this way, it offers a scalable and affordable process for the manufacturing of polymer, ceramic, and composite mats with controlled 3D architectures. There are used as scaffolds for tissue engineering, nanofibrous mats for drug delivery and biosensing, nanowire sensors for breath and skin-based non-invasive diagnosis based on gaseous biomarker detection, and photocatalysts for non-invasive cancer treatment, among others. Examples of the next generation biomaterials processed by electrospinning and used for each of these applications will be provided in this talk, spanning bone engineering to asthma monitoring.

3:50 PM

(ICACC-S5-021-2017) Additive manufacturing of calcium phosphate based ceramics for bone tissue

F. E. Weber*[1]; 1. University Zurich, Switzerland

The treatment of large bone defects still poses a major challenge in orthopaedic and cranio-maxillofacial surgery. The aim of this project is to determine the osteoconductive potential of titanium- and ceramic-based implants generated by additive manufacturing and the application. Defined implants of titanium and tri-calcium phosphate were produced by laser-sintering or by lithography-based ceramic manufacturing, characterized and tested in calvarial defects in rabbits. Analysis by μCT and histomorphometry revealed that all generatively produced structures either titanium based or calcium-phosphate-based were well osseointegrated into the surrounding bone. The histomorphometric analysis, based solely on the middle section, revealed that bone formation was significantly increased in all implant treated groups compared to untreated defects. In the critical size defect, the scaffolds alone were sufficient to yield defect bridging after 16 weeks. Addition of BMP and epigenetically small chemicals was able to increase the area of osseous regeneration when delivered with a faster degrading polylactide-glycolide polymer used to generate an in situ forming implant. Designed porous, lightweight structures from titanium and ceramics have potential for bone regeneration and augmentation purposes, especially when complex and patient-specific geometries are essential.

4:10 PM

(ICACC-S5-022-2017) Flash sintering of TCP bioceramics

M. Frasnelli*[1]; V. M. Sglavo[1]; 1. University of Trento, Italy

In this work, sintering behavior of tricalcium phosphate (TCP) ceramics under the effect of an external electrical field (in flash sintering configuration) was analyzed to obtain dense bio-resorbable components. The aim was to understand the physical condition leading to the flash phenomenon and to study the effect of consequent reduction in sintering time and temperature on the undesired $\beta \rightarrow \alpha$-TCP phase transition occurring at high temperature. TCP powders were synthesized by conventional solid state reaction and then shaped into cylindrical green body by uniaxial cold-pressing. Their sintering behavior was studied by dilatometry under different E-field at constant rate heating. The presence of α-TCP and the microstructure were investigated by XRD and SEM techniques. It is shown that a flash phenomenon takes place at furnace temperature below 1000°C. In addition, although $\beta \rightarrow \alpha$ transition occurs at ~1150°C for pure TCP, the detection of both polymorphs within the sintered bodies indicates that higher temperature is reached in the material, which is very likely associated to Joule effect induced by the current flow along the sample.

4:30 PM

(ICACC-S5-023-2017) Temporal study of the shell formation of European abalone Haliotis tuberculata

W. Ajili*[1]; T. Azaïs[1]; S. Auzoux-Bordenave[2]; N. Nassif[1]; 1. Laboratoire Chimie de la Matière Condensée de Paris, France; 2. Muséum National d'Histoire Naturelle UMR Borea, France

Nacre, the inner part of mollusk shells is made of a mineral phase (calcium carbonate) associated to an organic counterpart (mostly chitin). The mineral phase is mainly composed of aragonite, a polymorph of $CaCO_3$. This composite material displays an original 3D organization where aragonite micrometric crystals are stacked together and surrounded by chitin and mineralizing proteins. In this work we are using the European abalone Haliotis tuberculata as a model to study shell and nacre formation from the early stages of the larval development to the adulthood. By using ^1H and ^{13}C NMR, we are able to edit individually the different components of nacre namely (i) aragonite, forming the core of the tablets, (ii) the disordered surface and (iii) the surrounding organic phase. Comparison with synthetic calcium carbonate reference sample reveals the presence of HCO_3^- ions at the surface of the aragonite tablets. This work has been carried out with financial aid of the MATISSE Labex.

S7: 11th International Symposium on Nanostructured Materials: Functional Nanomaterials and Thin Films for Sustainable Energy Harvesting, Environmental and Health Applications

Biomedical Application I

Room: Coquina Salon A
Session Chair: Motoyuki Iijima, Yokohama National University

1:30 PM

(ICACC-S7-049-2017) Hard carbon coatings for medical applications (Invited)

R. Narayan*[1]; 1. North Carolina State University, USA

The medical applications of hard carbon coatings, including diamond-like carbon, nanocrystalline diamond, and ultrananocrystalline diamond, have recently been investigated by several groups. Ultrananocrystalline diamond coatings contain grain sizes below 10 nm. Nanocrystalline diamond coatings contain grain sizes between 10 nm and 500 nm. In this talk, the physical, chemical and in vitro biological properties of hard carbon coatings will be considered. Use of in vitro biological studies to assess hard carbon coatings for orthopaedic and blood-interfacing applications will be described. Functional studies of medical devices containing hard carbon coatings will also be considered.

2:00 PM

(ICACC-S7-050-2017) Functionalised Cellular Metals for Biotechnological Applications (Invited)

R. Hauser*[1]; 1. Fraunhofer IFAM, Germany

Cellular metals such as metallic hollow spheres structures, single hollow spheres and open-cell metal foams are lightweight, with an enormous application potential. The surface functionalization occurs by a wet chemical process to form ceramic or thermoset coatings on cellular metals as an alternative technique to the established PVD and CVD methods. The coatings were synthesised by Liquid Phase Deposition (LPD), of the cellular structures with sol/gel - suspensions. Subsequent thermal treatment of the as coated materials provided a glass like or ceramic coating under good reproducible conditions. The as obtained highly porous coatings are tuneable in regard to pore size, form and volume. The target in the biotechnology research is the developing of applications for the new porous metallic working materials with biofunctional surfaces such as single enzymes or coupled enzyme systems, receptors, different microorganisms and biospecifical dyes. First results can be indicated for the biotechnological application in the field of biocatalysis, bioanalytic and biosensor technology. Therefore, the materials were tested in continuous microfluidic systems. Statements on the enzyme activity and stability can be determined by interaction with the functionalized metallic materials. Enzymes can be immobilized for industrial biocatalysis and applications and tested also in hydrophobic mediums, for instance in organic solvents.

2:30 PM

(ICACC-S7-051-2017) Cost-effective nanowire device optimized for chemical and biological sensor (Invited)

Y. Im*[1]; 1. Chonbuk National University, Republic of Korea

Semiconductor nanowire field effect transistors (NWFETs) have attracted strong research interest as a promising platform for the construction of chemical and biological sensors. Despite a wide range of potential applications, there still remain critical issues to be resolved for the industrial applications, including Debye screening effects, and cost-effective route for mass production. To address these issues, we developed a cost-effective fabrication method of NWFET based on sequential processes of metal oxide deposition, nano-imprint lithography, low-damage plasma etching, and non-thermal plasma deposition. This approach is compatible with the conventional semiconductor process. Furthermore, we demonstrate that nanowires with different cross-sectional shape can be created by controlling the plasma etch conditions, leading to improvement of total sensing performance. It is demonstrated that the proposed NWFET device with effective surface functionalization can be applied successfully to chemical and biological sensors. Finally, the nanowire device for chemical and biological sensor is optimized through comparison studies of 3D device simulation considering electrochemical phenomena. We believe that this work can support better insights to overcome main bottlenecks concerning the commercialization of these technologies.

2:50 PM

(ICACC-S7-052-2017) Synthesis and functionalization of Janus-type particles for biomedical applications

I. Gessner*[1]; D. Hoffelner[1]; S. Siribbal[1]; T. Fischer[1]; S. Mathur[1]; 1. University of Cologne, Germany

Janus type particles which combine the specific properties of different materials are currently gaining great interest in research due to their side selective surface functionalization. These asymmetric particles can hence be used in a wide range of potential applications including biomedicine. However, to date synthesis protocols are limited in the usage of chemicals and an easy, controllable and flexible step by step approach for the design of Janus particles is still missing. In this approach, a reproducible and cheap method for designing gold or silver covered silica particles with asymmetric chemical functionalities is reported. In more detail, functionalized silica particles were prepared, dried on a substrate and finally covered with the metal by sputtering. Several specific functionalities were attached to the silica side including amino, thiol and azide groups for potential carbodiimide, disulfide or click reactions, respectively. The presence of these groups on the particle surface was demonstrated by Raman and FTIR spectroscopy as well as XPS, DLS and zeta-potential measurements. After Au/Ag sputtering, SEM and TEM analysis proved the successful synthesis of uniform Janus type spheres. The as-described particles are promising tools for biomedical applications including, photothermal therapy, drug delivery and bioimaging.

New Materials and Processing V

Room: Coquina Salon A
Session Chair: Partha P. Mukherjee

3:10 PM

(ICACC-S7-053-2017) Multifunctional metal oxide nanocrystals by designed inorganic surface modification (Invited)

M. Epifani*[1]; 1. CNR-IMM, Italy

Surface management is critical in such fields as heterogeneous catalysis, gas-sensors, photocatalysis. Several material features play a critical role in determining the final properties, such as the catalyst size and shapes, surface oxygen vacancies (often correlated with in situ formation of active intermediates) and oxidation state of surface atoms. Classical heterogeneous catalysis can provide suggestive concepts of surface modifications. In fact, it may expected that the nanocrystalline version of combinations of catalytic oxides (COX) supported onto another metal oxide (support oxide, SOX) can feature enhanced synergistic effects. If the crystallite size of the SOX is decreased more and more, the relative electronic contribution generated by the reactions at the COX is increasingly important, due to the enhanced surface/volume atoms ratio in nanocrystalline materials. This work will review the main results obtained by the application of such concept: the design of materials architectures, the subsequent wet chemical synthesis, the complex determination of the materials structure, the related applications. Particular focus will be given to the TiO_2-V_2O_5 system, featuring a complex set of different properties, ranging from supercapacitors, gas-sensing, CO_2 photoreduction etc. Then extension to other systems like SnO_2-V_2O_5, TiO_2-WO_3 and TiO_2-MoO_3 will also be shortly reviewed.

3:40 PM

(ICACC-S7-054-2017) Enhanced hydrogen release of light metal-borohydrides mixed with nanostructured carbon materials

F. Pendolino[1]; A. Nale[1]; A. Maddalena[1]; P. Colombo*[1]; 1. University of Padova, Italy

Carbon allotropes and derivates show novel behavior in a framework of promising technologies. Among them, nanohorns attracted attention as an alternative to graphene because of their reactivity, the prospective to trap molecules and their biocompatibility. A modification of the nanohorn surface provides reactive sites in order to engineer advanced composite materials for energy storage application with hydrogen content materials. A suitable strategy consists in oxidizing and reducing the nanohorns; oxidation was carried out with a novel synthetic method, comprising of 4-steps chemical synthesis at low temperature. Reduced nanohorns oxide (rNHO) were prepared by clean thermal treatment in hydrogen atmosphere. Light metal borohydrides are good candidate materials for hydrogen storage but they are limited by their excessive stability. The effect of rNHO on lithium boronhydride (LiBH4) have been investigated with thermal programmed desorption volumetric analysis, to test the gas release in presence of different modifications of carbon nanomaterials. A decrease of gas release temperature and a different profile compared to pure LiBH4 are observed. rNHO show the most pronounced impact on the gas release performance of LiBH4 even without metal additives with catalytic properties.

4:10 PM

(ICACC-S7-055-2017) Citrate Precursor Synthesis, Structural Characterization and Dielectric Properties of $Ba_{1-x}Ca_xZrO_3$ (0.05 £ x £ 0.20) Nanoparticles

M. Ubaidullah*[1]; T. Ahmad[2]; 1. Glocal Univeristy, India; 2. Jamia Millia Islamia (A Central University), 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 – $271 m^2 g^{-1}$ 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.

4:30 PM

(ICACC-S7-056-2017) Role of Metal Nanoparticles in Photo/Catalytic Activation of Carbon Dioxide

K. Bienkowski*[1]; R. Solarska[1]; 1. University of Warsaw, 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 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 Au, Ag and Cu surfaces enhanced by the extra activation of the process provided by incorporated plasmonic metallic nanostructures is supported by the occurrence of the photo-emission process associated with the decay of photo-excited surface plasmons. Of particular interest will be a strong influence of the Ag/FTO electrode roughness upon the photo-emissive cathodic currents of electro-reduction of CO_2 in aqueous solution. 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 work.

4:50 PM

(ICACC-S7-058-2017) Solution chemical processing of nano phase ZrN and ZrC powders

S. N. Katea*[1]; G. Westin[1]; 1. Uppsala University, Sweden

ZrN has many extreme properties such as high melting point, hardness, durability, thermal and electric conductivity. ZrN nanophase powders are required for use in hard cermets and lately ZrN is chosen as the matrix for fuels such as; U, Pu and Am in the Pb and Pb-Bi cooled Gen IV nuclear reactors. The present micron sized and impure ZrN produced through carbothermal nitridation require very high sintering temperatures even with SPS which is not suitable for production. The poor powder quality probably stems from the uneven mixing of the ZrO2 and carbon black. Here the phase development in a new solution based process to nano-phase ZrN based on sucrose-Zr-alkoxide precursors is described in detail with TG-DTA, XRD, IR and Raman spectroscopy SEM-EDS and HR-TEM-EDS/EELS. It was found that an extremely intimate mixing of ZrOx(OH)y and graphenic carbon was obtained at 200C, and that the Zr-particles were converted to ZrO2 at 600C with retained structure at 600C. The carbothermal nitridation reactions started at 1200C and were finished at 1500C, resulting in a phase pure ZrN according to XRD, but TEM showed that the ca 20-30 nm sized particles had a 4-5nm thick amorphous or fine-crystalline shell. The details of this shell are discussed, as well as its formation.

S8: 11th International Symposium on Advanced Processing and Manufacturing Technologies for Structural and Multifunctional Materials and Systems (APMT11)

Design-Oriented Manufacturing I

Room: Coquina Salon B

Session Chairs: Satoshi Tanaka, Nagaoka University of Technology; Csaba Balazsi, HAS Centre for Energy Research

1:30 PM

(ICACC-S8-033-2017) Microstructure Tailoring of C/C-SiC Composites (Invited)

W. Krenkel*[1]; 1. University of Bayreuth, Germany

Ceramic matrix composites (CMC) manufactured via Reactive Melt Infiltration (RMI) processes, in particular by Liquid Silicon Infiltration (LSI), are a well-established structural ceramic materials, used in gas turbines, spacecraft or brake systems. Typically, the requirements on these applications differ widely with the consequence that the microstructure should be tailored to different properties, like high thermal conductivity, high stiffness or low porosity. The RMI-process offers an unique opportunity for these matrix modifications as the fiber/matrix interface can be modified easily by thermal fiber treatment which varies the functional groups on the fiber surface. As a result, these fiber pretreatments, prior to the infiltration of the preform, lead to SiC-matrices with a high variation of compositions. The correlations between the mechanical strength, the microstrusture and the material's composition is discussed.

2:00 PM

(ICACC-S8-034-2017) Hybrid technology for complex shape ceramics and cermets parts (Invited)

F. J. Cambier*[1]; F. Petit[1]; G. Martic[1]; E. Juste[1]; 1. Belgian Ceramic Research Center, Belgium

Techniques used to process models of parts starting from 3-dimensional computer aided design data are commonly defined as rapid prototyping. Such a definition includes not only the very fashionable additive manufacturing but also the classical subtractive milling method. One problem with additive methods is the low mechanical strength obtained up today. For technical ceramics and cermets, the subtractive approach is the computer numerical control machining of presintered blanks, as it is largely used in industry, the low strength of the blanks may result in weak machining accuracy, damage at the edges of fine features and even partial rupture. In this talk, results obtained by combining the conventional machining method with spindle and a proprietary laser machining at the green state are reviewed. Classical milling of green component allows rapid roughing of parts. Since it is a non-contact machining, the laser technique allows to process fine details on the part, without applying any mechanical stresses and therefore avoiding risk of breakage The precision (mismatch between expected and real measurement) and the reproducibility (sintered densities through the sample, hardness, etc.) will be discussed in terms of geometrical and dimensional tolerances and machining speed for various shapes. Some examples of dense components made of alumina, zirconia, tungsten carbide produced by the hybrid technique will be shown.

2:30 PM

(ICACC-S8-035-2017) Ceramics with a dual pore morphology mimicking natural bone structure (Invited)

A. L. Leriche*[1]; D. Hautcoeur[2]; S. Chamary[1]; J. Hornez[1]; F. J. Cambier[2]; 1. University of Valenciennes, France; 2. BCRC, Belgium

For the last decades, research on bone substitutes processing has been focused on the development of new ceramic shaping methods able to mimic the complex structure of natural bone. Indeed, the human bone is constituted by two kinds of interconnected porous architecture: the trabecular or spongious bone with a high porosity and large pore sizes (between 500 to 600 μm) and compressive strength of a few tenth MPa and the cortical or compact bone with a much lower global porosity, smaller pore sizes between 190 and 230 μm and higher compressive strength between 80 to 200 MPa. Two processes to produce porous ceramic parts were applied to calcium phosphate scaffolds: the first is the ceramic slurry impregnation of organic skeleton which allows the development of isotropic spherical porous structure with 250 to 600 μm pore diameter, the second is a casting method based on ceramic slurry ice-templating producing tubular interconnected pores with ellipsoidal shape and smaller diameters (100 to 300 μm). It is known that the tubular porosity structure presents better compressive strength than the isotropic structure. By inserting isotropic porous materials in the center of an ice templating mold, it was possible to process a dual core-shell structure with large porosity inside the part to mimic the bone spongy structure while smaller size and elongated shape porosity around the part which mimics the compact bone.

2:50 PM

(ICACC-S8-036-2017) Multi-functional Ti3SiC2 modified SiC matrix composites

X. Yin*[1]; L. Cheng[1]; L. Zhang[1]; 1. Northwestern Polytechical University, China

Owing to the unique nanolaminate crystal structure, the MAX phases with A = Al and Si offer superior mechanical properties and multi-functional properties, which make them a potential reinforcement for brittle ceramic matrix materials. Capillary driven infiltration of a reactive melt allows near-net shape manufacturing of MAX phase reinforced composites with high flexibility in component geometry at low production costs. Formation of MAX phase in fiber reinforced ceramic matrix composites could improve not only the mechanical properties but also the wear resistance, oxidation resistance, ablation resistance and electromagnetic shielding effectiveness, extending the application fields of advanced ceramic composite materials. This review work will focus on the recent progress of fibers reinforced SiC matrix composites modified by Ti3SiC2.

3:30 PM

(ICACC-S8-037-2017) Novel Functional Materials Bioinspired from Nature Microstructures (Invited)

D. Zhang*[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. 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, electromagnetic shielding, 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.

3:50 PM

(ICACC-S8-038-2017) Si_3N_4/graphene nanocomposites for tribological application in aqueous environments prepared by attritor milling and hot pressing (Invited)

C. Balazsi*[1]; Z. Fogarassy[1]; A. Kailer[2]; C. Schroder[2]; M. Parchoviansky[3]; D. Galusek[3]; J. Dusza[4]; K. Balazsi[1]; 1. HAS Centre for Energy Research, Hungary; 2. Fraunhofer IWM, Germany; 3. Alexander Dubček University of Trenčín, Slovakia; 4. IMR SAS, Slovakia

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.

4:10 PM

(ICACC-S8-039-2017) Enhanced Photoluminescent Property of Crystalline-Oriented β-SiAlON:Eu^{2+} Phosphor Layer Prepared by Magnetic Field-Assisted Electrophoretic Deposition

T. Uchikoshi*[1]; C. Zhang[1]; L. Liu[1]; T. S. Suzuki[1]; Y. Sakka[1]; N. Hirosaki[1]; 1. National Institute for Materials Science, Japan

β-sialon:Eu^{2+}, one of the important members in the oxy-nitride phosphor family, has been known as a promising green phosphor with very narrow emission band (centered at ~535 nm) and high color purity. Besides, this green phosphor has high thermal and chemical stabilities, due to its stiff framework crystal structure of the host lattice. In this study, electrophoretic deposition (EPD) technique assisted with a 12 T strong magnetic field was utilized to fabricate crystalline-oriented β-sialon:Eu^{2+} phosphor deposits on ITO glass substrates. The direction of the applied magnetic field was perpendicular to that of the electric field for EPD. The influence of the horizontally-applied magnetic field assisted the phosphor particles to be oriented in the suspension, simultaneously, the vertically-applied electric field contributed the deposition of the positively-charged β-sialon colloidal particles onto a cathodic substrate. Therefore, the deposit had dominant characteristic that the a,b-crystal plane of the hexagonal β-sialon phosphor turned to upwards. Photoluminescence (PL) intensity of the oriented deposits was improved by comparing with that of the randomly oriented samples. This technique, EPD process performed in a strong magnetic field, would be a promising method for the packaging of flat LEDs.

4:30 PM

(ICACC-S8-040-2017) Elecroconductive oxide ceramics with graphene-encapsulated fillers

I. Hussainova*[1]; M. Drozdova[1]; S. S. Kale[2]; F. A. Sabet[2]; I. Jasiuk[2]; 1. Tallinn University of Technology, Estonia; 2. University of Illinois at Urbana-Champaign, USA

Miniaturization of components with complex shapes is a great challenge in emerging applications. The main limiting factors for wide usage of ceramics 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 electroconductive materials. In this study, an ex situ strategy for fabrication of electrically conductive and cost-effective metal oxide ceramics by incorporation of hybrid graphene-wrapped alumina nanofibers is presented. Fully dense alumina and zirconia based composites with homogeneously distributed hybrid nanofillers of length ranging from 0.1 to 10 mkm and a single fibre diameter of 10 nm is produced by spark plasma sintering. 3D Monte Carlo simulations are performed using a tunneling-percolation model based on critical path approximation. The fillers are modeled as randomly oriented and polydisperse hard-core soft-shell prolate ellipsoids. The percolation threshold is predicted following Weibull distribution. A percolation threshold of 0.2 wt.% is achieved and the electrical conductivity surpasses 103 Sm^{-1} when hybrid filler content is only 2 wt.%. An increase in conductivity does not result in deterioration of the mechanical properties as hardness and fracture toughness increased up to 10% and 40%, respectively, as benchmarked against plain alumina or zirconia.

4:50 PM

(ICACC-S8-041-2017) Ceramics Intergrain Contact Distribution and Microimpedance Fractality

V. Mitic*[1]; L. Kocic[2]; V. Paunovic[3]; 1. Serbian Academy of Sciences, Serbia; 2. Faculty of Electronic Engineering, Serbia; 3. Faculty of Electronic Engineering, Serbia

The only realistic model of ceramics structure is the fractal model. One component of this model is grains' distribution throughout the ceramics body. Using the Minkowski-Hlawka theorem of spherical contacts space distribution, the model of micro-capacitors distribution in electronics ceramics bulk is formulated. The intergrain capacity storage amount depends on the Riemann Zeta function value, fractal dimension of three-dimensional graph of contact points, corrected dielectric constant (depends on specific specimen fractal morphology) and temperature, which also involves a fractal correction parameter that emerges from the Curie-Weis law. In this manner, the resulting integral capacity is determined by two main factors: The geometry of intergranular configuration and the physico-chemical properties of consolidated sintered ceramics. Both components can be influenced by adjusting consolidation procedure and parameters. The main technological problem here is to get impedances with suitable RLC components. For example, getting super-micro capacitors can be used in high density energy storage. These new frontiers also open a quite new perspective in precise controling of these capacitors topology in higher electronic integration level within the fractal microelectronic.

5:10 PM

(ICACC-S8-042-2017) Metamaterials: The Challenge and Opportunity for the Multifunctional Ceramic (Invited)

R. Fan*[1]; 1. Shanghai Maritime University, China

Metal/ceramic micro and nanocomposites, also known as cermets, are excellent candidates to fabricate multifunctional devices due to the combination of two distinctly dissimilar components that have very different mechanical, thermal, optical, electrical and magnetic properties. For example, lossy cermets exhibiting microwave absorption, good heat resistance, good thermal conductivity and stability in a vacuum are required in a number of applications in microwave electronics. However, the contradictory properties of multifunctional cermets require their microstructure to be precisely tailored. We herein proposed some facile and highly effective strategy for tailoring the microstructure and properties of cermets. A series of micro and nanocermets containing metallic phase of different morphology were prepared. Interestingly, negative permittivity and negative permeability were observed in some cermets. The novel double negative properties ever reported only in so-called metamaterials are beneficial for the strong microwave absorption. The low-temperature process opens a new way for the realization of tunable physical properties in cermets, and has great potential for the preparation of new types of multifunctional materials.

S13: Advanced Materials for Sustainable Nuclear Fission and Fusion Energy

Accident-tolerant Fuels I

Room: Coquina Salon H
Session Chairs: Theodore Besmann, University of South Carolina; Peng Xu, Westinghouse Electric Company

1:30 PM

(ICACC-S13-027-2017) Development of Engineered SiC-SiC Accident Tolerant Fuel Cladding (Invited)

C. Deck*[1]; H. Khalifa[1]; G. Jacobsen[1]; J. Sheeder[1]; J. Zhang[1]; C. Bacalski[1]; J. Stone[1]; C. P. Shih[1]; G. Vasudevamurthy[1]; E. Lahoda[2]; C. Back[1]; 1. General Atomics, USA; 2. Westinghouse Electric Company, USA

A multi-layered cladding design has been developed which couples the advantages of monolithic silicon carbide (SiC) with a SiC fiber reinforced (SiC-SiC) composite. This engineered cladding provides high temperature strength, toughness, low permeability, and stable behavior under neutron irradiation and has potential to offer improved performance and enhanced accident tolerance. The ability to produce long, thin-walled cladding tubes meeting strict dimensional tolerances with controlled surface roughness is essential, and fabrication of parts up to ~1m length has been demonstrated. Fuel cladding must also be hermetically sealed, and recent efforts have focused on adapting an irradiation-tolerant SiC-based joining process to form an impermeable joint between and endplug and a fueled rod. Cladding and joint performance have been assed using both destructive and non-destructive methods. A large set of cladding mechanical data was obtained using both quasi-static and dynamic loading, and under ambient and elevated temperature conditions. Non-destructive x-ray computed tomography (XCT) examination was also used to evaluate microstructure and geometric tolerances. Characterization results for these SiC-based cladding tubes are presented and assessed relative to anticipated performance requirements. * This work was supported by General Atomics IR&D funding and the Department of Energy under Award Number DE-NE0000566.

1:50 PM

(ICACC-S13-028-2017) Development of SiC/SiC composites for accident tolerant fuel (Invited)

T. Hinoki*[1]; M. Lee[1]; S. Kondo[1]; S. Yanagawa[1]; K. Kawasaki[1]; F. Shinoda[1]; 1. Kyoto University, Japan

Silicon carbide (SiC) is one of very attractive engineering ceramics in particular for high temperature use and nuclear application. Fundamental mechanical properties of highly crystalline nuclear grade SiC composites are stable following neutron irradiation. Silicon carbide composites are promising materials for accident tolerant fuel. However highly crystalline nuclear grade SiC composites are different from high purity SiC like CVD SiC. They have carbon (C) interphase between fiber and matrix to retain ductile fracture behavior. Liquid phase sintered (LPS) SiC has highly crystalline microstructure. However it also has remained sintering additives as impurities. Remained Al2O3 and Y2O3 phase in grain boundaries are the weakest link. Those impurities are concerns in particular for corrosion behavior by high temperature water and high temperature steam. The grain boundary of LPS SiC was modified. High temperature water corrosion resistance of the LPS SiC was significantly improved. The SiC composites were developed with the particle dispersion in matrix instead of C fiber/matrix interphase by LPS. The LPS composite tube didn't fracture even after thermal shock from 1200 C to ambient temperature in air.

2:10 PM

(ICACC-S13-029-2017) Anisotropic swelling of SiC composites during neutron irradiation

Y. Katoh*[1]; T. Koyanagi[1]; L. L. Snead[2]; K. Yueh[3]; 1. Oak Ridge National Laboratory, USA; 2. Massachusetts Institute of Technology, USA; 3. Electric Power Research Institute, USA

Radiation-induced swelling is a primary factor that determines the stress state in SiC composite-based fuel cladding for light water reactors. To properly assess the evolving stresses, it is required to understand potential anisotropy in swelling for SiC composites. In this study CVI SiC composites with Hi-Nicalon Type S and additional few variations of SiC fibers were irradiated in the High Flux Isotope Reactor (HFIR) at ~300°C to 0.01 - 12 dpa followed by measurement of all dimensions of the coupon specimens to an accuracy of 0.1 micron and irradiation temperature determination for individual specimens. The accurate dimensional measurement employed in this work lead to multiple interesting findings including the significant differences in swelling between the in-plane and transverse orientations. Detailed results and the driving mechanisms will be discussed in presentation. This research was supported by Office of Nuclear Energy, U.S. Department of Energy under Contract No. DE-AC05-00OR22725 with UT-Battelle, LLC, and Electric Power Research Institute under Contract No. NFE-13-04618. The use of HFIR was sponsored by the Scientific User Facilities Division, Office of Basic Energy Sciences, U.S. Department of Energy.

2:30 PM

(ICACC-S13-030-2017) Effects of Non-Uniform Boundary Conditions on the Performance of SiC/SiC Cladding

G. Singh*[1]; N. Brown[1]; B. Wirth[2]; K. Terrani[1]; Y. Katoh[1]; 1. Oak Ridge National Lab, USA; 2. University of Tennessee, USA

SiC/SiC cladding is one of the leading concepts being considered for the accident tolerant fuel-cladding systems. A thermo-mechanical analysis of the cladding was performed to evaluate the effect of non-uniform circumferential boundary conditions on its performance. State of the art properties of SiC/SiC composites and boundary conditions based on thermal hydraulics calculations were employed to accurately determine the thermo-mechanical behavior of the cladding. Results indicate that significant bending-induced stresses will develop in the cladding.

3:10 PM

(ICACC-S13-031-2017) Influence of the PyC Interphase on Mechanical Properties and Failure Mechanisms of SiC-SiC Composites

J. Kabel*[1]; Y. Yang[1]; M. Balooch[1]; P. Hosemann[1]; K. Terrani[2]; T. Koyanagi[2]; 1. University of California Berkeley, USA; 2. Oak Ridge National Lab, USA

SiC-SiC composites exhibit exceptional high temperature properties including high strength, high radiation tolerance, and low thermal neutron cross sections. As a result, it is a candidate for accident tolerant fuel cladding and advanced reactor components. In order to overcome the inherent brittleness of the ceramic, engineers have developed composites that exploit pseudo-ductility through interfacial shear and increased crack propagation at the fiber-matrix interphase. This research implements small scale mechanical testing and TEM analysis to evaluate the Pyrolytic Carbon (PyC) interphase properties that are responsible for this increased toughness. Focused ion beam milling was used to cut a variety of geometries that promote fracture at the interphase during compressive loading. Resolved normal and resolved shear stress plots for a variety of interphase inclines allowed for the extraction of an internal friction coefficient that ranged from 0.1 to 0.6, and a debond shear strength that ranged from 120 MPa to 460 MPa depending on the fiber type and PyC first layer thickness. TEM analysis provided insight to the interphase structure and failure mechanisms. Fracture was consistently observed in the first graphitic PyC layer, with thicker layers exhibiting shear strength similar Highly Oriented Pyrolytic Graphite (HOPG), regardless of fiber type.

3:30 PM

(ICACC-S13-032-2017) Uranium Silicide-Nitride Fuels: Thermochemical Behavior and Compatibility (Invited)

T. M. Besmann*[1]; M. Noordhoek[1]; T. Wilson[1]; A. Nelson[2]; E. Wood[2]; J. McMurray[3]; D. Shin[3]; E. Lahoda[4]; S. Middleburgh[5]; 1. University of South Carolina, USA; 2. Los Alamos National Lab, USA; 3. Oak Ridge National Lab, USA; 4. Westinghouse Electric Company, USA; 5. Westinghouse Electric Company, Sweden

New fuel and cladding systems are being explored to identify more efficient and accident tolerant materials than the existing urania-zirconium alloy LWR systems. Among the materials of interest are uranium silicide and uranium silicide-nitride composite fuels and cladding composed of ferritic alloys or silicon carbide composites. To aid in screening proposed systems, and in understanding potential in-reactor behavior under normal and off-normal conditions, efforts have been made to determine thermochemical behavior under irradiation and likely interactions between fuel and cladding. The presentation will cover the initial results of the thermochemical analysis of the fuels together with potential interaction with prospective alternative cladding materials. Research supported by the U. S. Department of Energy, Office of Nuclear Energy Fuel Cycle R&D Program and through the Nuclear Energy University Programs.

4:00 PM

(ICACC-S13-033-2017) The Role of Pd in the Chemical Attack and Migration of Silver-Palladium in Silicon Carbide

H. Liu*[1]; P. Xiao[1]; 1. University of Manchester, United Kingdom

The silicon carbide (SiC) coating included in tristructural-isotropic (TRISO) particle acts as the main barrier against the radioactive fission products (Ag, Pd, Cs etc) release into the coolant circuit. However, high fraction of Ag transport through SiC and chemical attack of Pd on SiC were identified and remained a problematic issue for licensing these fuel particles. In this study, the chemical attack of AgPd on SiC and the role of Pd in Ag migration through SiC were studied by using the advanced electron microscopy (SEM, TEM and STEM-EDS). Diffusion couples of AgPd-SiC were annealing from 1100°C to 1400°C in Argon. Compared with silver alone, the silver-palladium mixture was more permeable in SiC and showed a Ag-rich diffusive band in front of the reaction zone where palladium silicide Pd2Si and C were produced. In the diffusion front, the Ag preferred to present in the nano-scale nodules rather than the large nodules composed of Pd2Si. Additionally, the reaction zone showed an evolving microstructure instead of the periodical pattern which was always observed in Pd-SiC reaction zone, hence showing the decrease of Pd activity and mobility with the presence of Ag. The current results indicated that the Ag migration in the SiC was mainly accompanied or assisted by the nodules containing Pd, Si and Ag, probably the glass phase, rather than the stable palladium silicide.

4:20 PM

(ICACC-S13-034-2017) Thermal Analysis of ATF Fuel Materials at Westinghouse

L. Cai*[1]; P. Xu[1]; A. Andrew[1]; F. Boylan[1]; E. Lahoda[1]; 1. Westinghouse, USA

Accident tolerant fuels (ATF) attract a lot of attention due to the improved safety margins and potential economic benefits. The proposed uranium fuels for ATF including UN, U_3Si_2, and U_3Si_2/UN composites, offer significant potentials for improvement as light water reactor (LWR) fuel over the current nuclear fuel standard, UO_2, specifically in thermal conductivity and fissile content. However, these compounds have never before been applied as fuels for LWR use. To transition them to functional LWR fissile fuel, significant investigation and understanding of these compounds is required. An experimental thermal analysis of these candidates is undertaken to quantitatively determine the behaviors of these compounds at elevated temperatures in a water vapor atmosphere. The oxidation temperature for U_3Si_2 and UN powders is determined using thermogravimetry (TG) during continuous heating. The behavior of U_3Si_2 and U_3Si_2/UN at the typical LWR coolant temperatures in a water vapor atmosphere are revealed by TG isothermal experiments. The results will be discussed and compared to UO_2 for better understanding of their behaviors. © 2016 Westinghouse Electric Company LLC. All Rights Reserved.

4:40 PM

(ICACC-S13-035-2017) Simulating Transport of Chemical Species in Nuclear Fuel: Coupling Thermodynamics and Fuel Performance Codes

B. G. Gaston*[1]; T. M. Besmann[1]; S. Simunovic[2]; J. McMurray[2]; M. H. Piro[3]; 1. University of South Carolina, USA; 2. Oak Ridge National Lab, USA; 3. Canadian Nuclear Laboratories, Canada

The integration of the Thermochimica thermodynamic equilibrium code with the nuclear fuels performance code BISON continues to be explored for use in chemical transport calculations in light water reactor fuels. Current models rely only on chemical species concentration and empirical relationships, and do not account for the effects of fission products and evolution of complex chemical species. Thermochimica utilizes a computationally efficient approach to Gibbs energy minimization employing a material-specific database to calculate the thermodynamic equilibrium in the fuel material within each element for each timestep and determine the chemical species/phases present. Also determined are properties directly related to transport, such as chemical potential and defect concentrations. Oxygen transport, driven by the gradient of chemical potential, is of particular interest due to the difficulty of modeling diffusion in complex multicomponent systems, such as urania fuel undergoing burnup. This work includes the use of Thermochimica in calculations of transport and precipitation of hydrogen in the zircalloy fuel clad as well as the redistribution of oxygen in the fuel pellet throughout the burnup cycle. This work is supported by the U.S. Department of Energy, Office of Nuclear Energy Nuclear Energy Advanced Modeling and Simulation Program and Fuel Cycle R&D Program.

5:00 PM

(ICACC-S13-036-2017) Thermodynamic Assessment of Nepheline Formation in Nuclear Waste Glass

S. A. Utlak*[1]; T. M. Besmann[1]; C. H. Henager[2]; S. Hu[2]; Y. Li[3]; 1. University of South Carolina, USA; 2. Pacific Northwest National Lab, USA; 3. Pacific Northwest National Lab, USA

Vitrified high-level radioactive waste that contains high concentrations of Na_2O and Al_2O_3, such as the waste stored at the Hanford site, can cause nepheline to precipitate in the glass upon cooling in the canisters. Nepheline removes oxides such as Al_2O_3 and SiO_2 from the host glass, which can reduce its chemical durability. Uncertainty in the extent of precipitated nepheline necessitates operating at an enhanced waste loading margin, which increases operational costs by extending the vitrification mission as well as increasing waste storage requirements. A critical evaluation of the Na_2O-Al_2O_3-SiO_2 system that forms nepheline was conducted utilizing the compound energy formalism and ionic liquid models to represent the solid solution and liquid phases, respectively. These were optimized with experimental data and used to extrapolate phase boundaries into regions of temperature and composition where measurements are unavailable. The intent is to import the determined Gibbs energies into a phase field model to more accurately predict nepheline phase formation and morphology evolution in waste glasses. We gratefully acknowledge the financial support of the U.S. Department of Energy's Waste Treatment and Immobilization Plant Federal Project Office under the direction of Dr. Albert A. Kruger.

5:20 PM

(ICACC-S13-037-2017) Irradiation and Storage Effects on Nuclear Fuel Young's Modulus Characterized by High Frequency Acoustic Microscopy

M. Marchetti*[1]; T. Wiss[1]; D. Laux[2]; G. Despaux[2]; V. Rondinella[1]; E. Fontana[1]; 1. European Commission - Joint Research Centre, Germany; 2. University of Montpellier, France

The evolution of the elastic properties of nuclear fuel in nuclear reactors has a direct consequence on their thermo-mechanical behaviour. Moreover, nuclear fuels are subjected to additional damage caused by alpha self-irradiation after their discharge and during their storage period. In this context, separation of the effects due to the irradiation in the reactor and the self-irradiation during storage enables a better understanding of the phenomena affecting the fuel performance and its safety under normal operational conditions. Here, we present the global effect of the irradiation on Young's modulus of light water reactor fuels in the 0-100 GWd/tM burnup range, using an acoustic microscope (60-90 MHz) installed in the hot-cell facility of the Joint Research Centre in Karlsruhe. These measurements have been combined with data available in the open literature to obtain an empirical model describing the variation of the Young's modulus as a function of the burnup. In addition, the variation of the Young's modulus, as a function of the cumulated alpha dose during storage has been determined for irradiated uranium dioxide fuels and uranium dioxide analogues samples doped with different concentration of ^{238}Pu. The latter provides a detailed insight into the causes of deviations in published data on the Young's modulus of mixed oxide fuels.

Friday, January 27, 2017

FS1: Geopolymers, Chemically Bonded Ceramics, Eco-friendly and Sustainable Materials

Construction Materials

Room: Coquina Salon E
Session Chair: Flavio Silva, Pontificia Universidade Católica do Rio de Janeiro (PUC-Rio)

8:30 AM

(ICACC-FS1-025-2017) Pathways to Commercial Adoption of Chemically-Activated Cement (Invited)

J. S. Van Deventer*[1]; 1. Zeobond Group, Australia

Geopolymers synthesised from calcium aluminosilicate waste as a cement with low CO_2 emissions have been investigated widely. Nevertheless, commercial application of this disruptive construction material has been limited owing to numerous obstacles. Most cement and concrete standards prescribe a minimum Portland content for different applications. This prescription is highly inhibitive to innovation and presents a formidable challenge to the commercial adoption of new cementitious binders, including geopolymers. When Portland cement is no longer the key binder and various non-Portland binder chemistries could be used instead, a prescriptive standards framework becomes inappropriate. Prescriptive standards give structural engineers, concrete specialists, contractors, asset owners and insurance companies a sense of security. It will be shown that the link between durability testing and the adoption of performance-based cement and concrete standards are pivotal to the valorization of waste in the construction industry. Despite the obstacle of establishing a new supply chain for source materials, it will be shown here that commercial adoption of geopolymer concrete has been achieved in many applications. Recently, there has been an increase in interest in commercial application of geopolymers in new markets, with the driver being valorization of fly ash and slag, or a push to further reduce CO_2 emissions.

9:00 AM

(ICACC-FS1-026-2017) On the durability behavior of natural fiber reinforced geopolymers (Invited)

F. d. Silva*[1]; 1. Pontificia Universidade Católica do Rio de Janeiro (PUC-Rio), Brazil

Geopolymers may be described as solid and stable synthesized aluminosilicate materials which properties, presented in previous studies, are generally believed to provide a superior durability behavior for components used in the construction industry. The work in hand presents the preliminary results of an experimental investigation on the chemical and thermal durability behavior of jute fiber reinforced geopolymers. Silica fume, metakaolin and blast furnace based geopolymers were produced with several types of aggregates in order to increase the material behavior under moderated and elevated temperatures and under wetting and drying cycles. Compressive, tensile and flexural tests were performed, before and after the aging regimes and exposition to elevated temperatures. Microstructural analyses were performed to evaluate the degradation of the jute yarn and of its matrix using an environmental scanning electron microscope. Thermogravimetric analysis and x-ray diffraction was used to investigate the possible degradation in the geopolymer matrices. Finally, in order to investigate the fiber-matrix bond under the several studied regimes, pullout tests were performed.

9:30 AM

(ICACC-FS1-027-2017) Processing Microstructure and Properties of Slag-Fly ash Geopolymers (Invited)

K. Sankar*[1]; P. Stynoski[2]; G. Al-Chaar[2]; I. Al-Qadi[3]; W. M. Kriven[1]; 1. University of Illinois at Urbana-Champaign, USA; 2. U.S. Army Construction Engineering Research Laboratory (ERDC-CERL), USA; 3. University of Illinois at Urbana-Champaign, USA

Slag-Class F fly ash based geopolymers are room-temperature hardening, structural materials that have shown promise as alternative binders to ordinary Portland cement (OPC). These geopolymers exhibit desirable mechanical properties and a much lower carbon footprint than OPC. In this work, we investigate the compressive strength, flexural strength, workability, setting time, permeability, shrinkage, and durability of various slag-fly ash based geopolymer mixtures. The effects of varying the mixing method and process temperature on strength and setting time were systematically studied. Scanning electron microscopy and energy dispersive x-ray spectroscopy (SEM-EDS) revealed how the microstructure and composition of the geopolymer relate to the observed properties. X-ray diffraction (XRD) revealed that no new crystalline phases were formed in the cured geopolymer, as expected. Finally, the feasibility of slag-fly ash geopolymer systems as a sustainable construction material is discussed.

10:20 AM

(ICACC-FS1-028-2017) Eco-friendly geopolymer composite for winter season pavement pothole patching

M. Sarkkinen*[1]; K. Kujala[2]; S. Gehör[2]; 1. KAMK, Finland; 2. Solid Liner Ltd., Finland

The purpose of the study was to examine usability of geopolymer materials for pavement pothole patching. Special focus in the study was use of materials under cold (5…-10°C) climate concerning repairs executed in the end of the winter season when the problem is most acute. Industrial by-products, such as mining tailings and slags from steel production were used as primary raw materials. In addition, recycled crumb rubber was used as a component. Influence of chosen factors on the performance of the material was studied by using Taguchi method comprising 13 factors and three levels. The results indicate that binder composition, aggregates, and admixtures

have significant influence to the strength development in cold conditions. Good compressive strength and bonding to asphalt could be achieved also under -10°C without heating or use of anti-freeze agents. Freeze-thaw resistance and resistance against chlorides were excellent after 50 freeze-thaw and after 25 chloride-freeze-thaw cycles. The results indicated that use of superplasticizer was beneficial despite of short pot-life (15 min). On the other hand, use of air-entraining –agent was not required to provide sufficient frost resistance. So far, the results indicate that use of industrial waste materials provide for geopolymer cements sufficient binder characteristics such as rapid hardening and durability also in cold and wet conditions.

10:40 AM

(ICACC-FS1-029-2017) Cementitious Composites for Structural Applications based on Metakaolinite Phosphate Cements

A. Katsiki*[1]; A. Peys[2]; T. Tysmans[3]; J. Wastiels[3]; H. Rahier[1]; 1. Vrije Universiteit Brussel, Belgium; 2. KULeuven, Belgium; 3. Vrije Universiteit Brussel, Belgium

Metakaolinite/phosphate cement (MKPC) is studied as a new environmentally friendly alternative to Portland cement with remarkable properties. In this work, an experimental study on the chemistry, microstructure and mechanical properties of the matrix material is presented. In parallel, an investigation of composites produced with the developed cementitious inorganic matrix and different types of reinforcement is undertaken. Due to the acidic environment of the fresh 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. Two-dimensional sheets, randomly chopped, tri-axial as well as 3D symmetric glass fiber fabrics are applied in order to check the impregnation, workability, mechanical strength and durability of the final composite. The mechanical properties are evaluated on beams loaded in tension. 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 building system.

11:00 AM

(ICACC-FS1-030-2017) Utilization of Geopolymer in Solidification/Stabilization of Sorbent-Impacted Fly Ash and Power Plant Wastewater Treatment Residuals (Invited)

C. Montes*[1]; S. Gomez[1]; E. Allouche[2]; K. Ladwig[3]; J. Mejia[4]; 1. Louisiana Tech University, USA; 2. Stantec, Canada; 3. EPRI, USA; 4. Universidad del Valle, Colombia

The air emission standards of acid gases, such as HCl and SO_2, have been tightened by federal and state regulatory agencies in recent years due to environmental concerns. Most traditional scrubbing systems include wet scrubbers and spray dryers, however, dry sodium injection (DSI) is a cost-effective alternative technology which utilizes trona as sorbent. Sodium-based reagents, however, yield solid reaction products, such as sodium sulfate and sodium chloride, which are mixed and collected with the fly ash in the primary particulate control device. In addition to sulfate leaching, the leachability of certain elements in the fly ash when proper stabilization has not been performed is a major concern. The use of geopolymers for waste stabilization has been documented since the early 1990s, and has shown good promise for the encapsulation of several heavy and toxic metals. The objective of the present research is to evaluate the effectiveness of geopolymers in the leaching reduction of soluble elements, in particular, Cr, As, B, Se and Mo from sorbent-impacted fly ash. Leaching tests results of geopolymer formulations designed from 6 coal fly ash samples, 3 of class C ash and 3 of class F ash, impacted with trona, lime or hydrated lime are presented, as well as results from the mechanical and rheological evaluation of such binders.

11:30 AM

(ICACC-FS1-031-2017) Novel 'inorganic gel casting' process for the manufacturing of glass-ceramic foams

A. Rincon Romero[1]; E. Bernardo*[1]; 1. University of Padova, Italy

A new technique for the production of glass foams was developed, based on gel casting. The new process is less expensive and more environmentally friendly than the current procedures, based on glass powders mixed with foaming agents, which decompose and release gases at temperature well exceeding the glass softening point. The alkali activation of soda-lime waste glass allowed for the obtainment of well-dispersed concentrated suspensions, undergoing gelification by treatment at low temperature (80°C), owing to the formation of calcium-rich silicate hydrates. An extensive direct foaming was achieved by vigorous mechanical stirring of partially gelified suspensions, comprising also a surfactant. A sintering treatment, at 700-800°C, applied to stabilize the cellular structures, particularly for limiting the leaching of alkaline ions, led to secondary foaming effects, as a consequence of decomposition of hydrated compounds, and to partial crystallization. An extension of the approach to mixtures of soda-lime glass and metallurgical slags, leading to further foaming effects (owing to reduction of ferric oxide) will be presented as well.

S1: Mechanical Behavior and Performance of Ceramics & Composites

Small-scale Testing and Applications

Room: Coquina Salon D
Session Chairs: Andy Nieto, University of California Davis; Benjamin Dillinger, Virginia Tech

8:30 AM

(ICACC-S1-072-2017) Indentation Creep Behavior of WC-Co processed by HVOF and SPS (Invited)

A. Nieto*[1]; J. M. Schoenung[2]; 1. University of California, Davis, USA; 2. University of California, Irvine, USA

Tungsten carbide cobalt (WC-Co) cermets are fabricated by spark plasma sintering (SPS) and high velocity oxyacetylene flame spray (HVOF). Nanoindentation is used to access room temperature creep behavior of WC-Co. Creep indentation tests are conducted using a Berkovich tip under load control, using a 10 min hold time. Low load (1000 μN) indentations are used to assess localized (single phase) creep behavior, and high load (30000 μN) indentations are used to assess the creep behavior of the combined cermet system. Creep behavior is discussed in terms of the varying chemical and microstructural features in the SPS consolidated and HVOF sprayed WC-Co cermets. Scanning electron microscopy, X-ray diffraction, and energy dispersive spectroscopy are used to characterize the microstructure and composition of WC-Co.

9:00 AM

(ICACC-S1-073-2017) Observations of Surface and Subsurface Cracks in Fused Quartz Using Nanoindentation and Focused Ion Beam Milling

B. A. Mound*[1]; G. M. Pharr[1]; 1. University of Tennessee, USA

With a growing number of brittle materials being used as thin films for microelectronics, wear-resistant coatings for energy storage, and coatings on orthopedic implants, there is a need to understand the fracture of brittle materials on the micro- and nano-scale. Nanoindentation is a technique in which the mechanical properties of ceramics and glasses can be studied at micron and smaller scales. We present the cracking modes present in fused quartz after fracture was induced using the nanoindentation technique. A range of loads (1 mN to 1000 mN) and three sided pyramidal indenters with angles ranging from 35.3° to 75° were utilized to study the effects of various

conditions on the cracking behavior of this typical brittle material. Cracks were observed using a dual beam focused ion beam (FIB) mill in a high resolution scanning electron microscope (SEM). The effects of load and tip geometry on the cracking modes that result from the indentations are discussed. Such cracking modes include lateral, radial, and edge cracks. The results, in conjunction with finite element simulations, provide important insight into how these cracks initiate within a small volume of material.

9:20 AM

(ICACC-S1-074-2017) Stress Mapping in Glass-to-Metal Seals using Indentation Crack Length Measurement

T. Buchheit*[1]; R. Jamison[2]; M. Teague[1]; K. T. Strong[1]; C. Newton[1]; T. Diebold[1]; K. G. Ewsuk[1]; 1. Sandia National Laboratories, USA; 2. Sandia National Laboratories, USA

Predicting the residual stress in a glass-to-metal compression seal requires a materials model that can accurately predict the effects of processing on the sealing glass. Validation of the predictions requires measurements on a representative test geometry to accurately capture the interaction between the seal materials from room temperature to 600°C, slightly above the glass transition temperature. To this end, a concentric seal test geometry consisting of a stainless steel shell enveloping a commercial sealing glass disk has been designed, fabricated, and characterized as a model validation test geometry. To obtain data to test/validate finite element (FE) stress model predictions, spatially-resolved residual stress was calculated from the measured lengths of the cracks emanating from radially positioned Vickers indents in the glass. The indentation crack length method will be described, and the spatially-resolved residual stress determined experimentally will be compared to FE stress predictions made using a nonlinear viscoelastic materials model adapted to inorganic sealing glasses. Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000

9:40 AM

(ICACC-S1-075-2017) In-situ measurement of mechanical properties of compressive layer in ion-exchanged glass using micro cantilever beam specimens

J. Tatami*[1]; S. Fujita[1]; M. Iijima[1]; T. Yahagi[2]; 1. Yokohama National University, Japan; 2. Kanagawa Academy of Science and Technology, Japan

Although glass is a typical brittle material, improvement of the strength resulted from compressive stress by replacement of Na^+ with K^+. Such ion-exchange should cause structure change, which means that the ion-exchange possibly results in change in the mechanical properties in the compressive layer. In this study, mechanical properties, such as bending strength, fracture toughness and elastic constant of compressive stress layer in ion-exchanged glass were directly measured using microcantilever specimens prepared by focused ion beam technique. The bending strength of compressive layer in ion-exchanged glass is about 40% higher than that of as-polished glass. There is no significant variation with the fracture toughness before and after ion-exchange. Young's modulus of ion-exchanged glass was higher than that as-polished glass. Consequently, it was shown that the strengthening of the ion-exchanged glass resulted from the improvement of Young's modulus.

10:20 AM

(ICACC-S1-076-2017) Crush Strength Analysis of Hollow Glass Microspheres

B. Dillinger*[1]; D. Clark[1]; C. Suchicital[1]; G. Wicks[2]; F. Humes[2]; 1. Virginia Tech, USA; 2. Applied Research Center, USA

Porous wall hollow glass microspheres (PWHGMs) are a unique type of microsphere characterized by the presence of interconnecting porosity spread throughout their wall. The crush strength of these microspheres is an important factor in the design of their applications. Since it is difficult to test the crush strength of microspheres due to their size and shape, the goal of this research was to develop a method to crush individual microspheres and analyze the data using Weibull analysis. The microsphere size distribution was determined using laser light scattering and the wall thickness and morphology were analyzed using scanning electron microscopy (SEM). Using nanoindentation with a diamond flat end tip, it was possible to crush individual microspheres between the tip and a fused silica substrate. Data were statistically analyzed using Weibull analysis to determine the characteristic strength and Weibull modulus of Hollow Glass Microspheres (HGMs) and PWHGMs. The results showed that the characteristic strength of the microspheres was inversely related to the microsphere diameter while there was little variation in the Weibull modulus. It was also shown that the characteristic strength of the PWHGMs was about one fourth of the strength of the HGMs.

10:40 AM

(ICACC-S1-077-2017) Graphene in Action: Understanding Toughening in Graphene/Ceramic Nanocomposites

M. C. Ramírez*[1]; Y. Liu[1]; Q. Wang[1]; M. I. Osendi[2]; P. Miranzo[2]; M. Belmonte[2]; B. W. Sheldon[1]; N. P. Padture[1]; 1. Brown University, USA; 2. Institute of Ceramics and Glass, CSIC, Spain

Increasing fracture toughness of advanced ceramics is one of the main challenges for augmenting the range of applications of these materials. Here the use of carbon nanostructures as reinforcements not only has marked influence on their mechanical behavior but also allows the development of tailored multifunctional properties. Recently, some studies have shown that graphene-nanoplatelets, obtained directly from graphite exfoliation or by its oxidation, produce significant toughening when they are incorporated in ceramic matrices. This effect has been explained in terms of crack-wake bridging during crack propagation. However, the details of the toughening mechanisms are obscure. Here we have performed in situ fracture tests on spark-plasma-sintered graphene/ceramic nanocomposites inside a scanning electron microscope (SEM) to gain a deep understanding of the toughening mechanisms. The wedge-splitting and flat-punch-compression sample geometries have been used to achieve stable crack growth, which has allowed us to observe continuously the crack propagation and deformation of graphene-nanoplatelets during bridging. Independent R-curve measurements and crack profiles measurements have also been conducted, which are correlated to the microstructures and the toughening mechanisms, enhancing our understanding of the mechanical behavior of these novel nanocomposites.

11:00 AM

(ICACC-S1-078-2017) Thermophysical and High-Temperature Mechanical Properties of CeO_{2-x}

U. Carvajal Nuñez*[1]; J. T. White[1]; N. A. Mara[2]; A. Nelson[1]; 1. Los Alamos National Lab, USA; 2. Center for Integrated Nanotechnologies (CINT), USA

Due to its identical fluorite-type structure, microstructural and thermal properties, CeO_2, has been proposed as a surrogate material to better understanding of the behavior of nuclear fuels during in pile. Importance of its use depend on its properties determined by stoichiometry associated with lattice defects. Structural defects, like vacancies, affect the material properties of both fresh samples and those during irradiation in reactors, with potential influences on fuel performance and eventually on the safety of the reactor operation. For instance, vacancies lead notably to distortion of lattice structure and macroscopic swelling, eventually altering material properties such as strength and ductility. The structure of nuclear fuels at high burnup can prove difficult given the significant changes in both chemistry and microstructure caused by fission products. Nanoindentation may prove to be a valuable addition to post irradiation examination. Thermophysical CeO_{2-x} data compared to the bibliography values for UO_2 to evaluate its suitability as a surrogate in nuclear fuel systems where thermal transport is a primary criterion for performance. In this study, we report nobel results of hardness at relevant temperatures of ceria at small scales and thermophysical properties by the use of Nanoindentation and Laser Flash Analysis (LFA) methods.

11:20 AM

(ICACC-S1-079-2017) Experimental Investigation on Impact Strength of low temperature treated Glass/Carbon epoxy Hybrid Laminated Composites

N. Kumar*[1]; S. Nagaraj[2]; 1. Christ University, India; 2. R V College of Engineering, India

The present study deals with the determination of peak load, deformation and total energy absorbed by the low temperature treated glass/carbon epoxy hybrid laminated composites during drop impact test. Laminates of three different stacking orders were prepared according to ASTM D5628-10 and were treated at a temperature of -80°C for a period of 28 hours. Experiments was conducted on the laminates using drop impact testing machine. The cross head with the indenter of diameter of 20mm is dropped down to the specimen from a height of 600 mm. From the experiment, the force, deformation and total energy absorbed by the specimen were evaluated. The results shows that the laminate with carbon fibres in the middle region was found absorb maximum energy and has undergone maximum deformation when compared with other two laminates. The laminate with carbon fibres on the outer layer was found to withstand maximum load when compared with other two laminates.

S3: 14th International Symposium on Solid Oxide Fuel Cells (SOFC): Materials, Science and Technology

Oxygen Ion Conductors

Room: Coquina Salon G
Session Chair: Radenka Maric, University of Connecticut

8:30 AM

(ICACC-S3-060-2017) Investigation on the material of electrolyte for improving electrical efficiency of SOFCs and/or PCFCs (Invited)

Y. Matsuzaki*[1]; Y. Tachikawa[2]; T. Somekawa[1]; K. Sato[1]; H. Matsumoto[3]; S. Taniguchi[2]; K. Sasaki[2]; 1. Tokyo Gas Co.,Ltd., Japan; 2. Kyushu University, Japan; 3. Kyushu University, Japan

An electric efficiency analysis of SOFCs with multi-stage electrochemical oxidation has been conducted to investigate the possibility of further improvement of the electric efficiency. We have developed an algebraic analysis method for the multi-stage oxidation. The multi-stage electrochemical oxidation using SOFCs is considered to be promising technology for further improving the electrical efficiency of SOFCs. However, the improvement is limited by fuel dilution downstream of the fuel flow. In a previous study, we have developed a conceptual design that has a potential to realize a critically-high power generation efficiency of up to 85% (LHV, gross DC), in which a multi-stage SOFC is combined with a proton-conducting solid electrolyte assuming protonic transport number of 1. However, typical proton-conductive solid oxide has mixed conductions of proton, oxide-ion, and hole. The influence of the conductivities of oxide-ion as well as electron and hole in the proton-conducting solid electrolyte on the electrical efficiency has been investigated. The existence of measurable conduction of hole was found to cause leakage current resulting in obvious deterioration of the electrical efficiency. Acknowledgements: This research is supported by JST through its COI program.

9:00 AM

(ICACC-S3-061-2017) Sintering and electrical properties of yttrium-doped barium zirconate proton conducting ceramics by addition of $YBa_2Cu_3O_7$

L. A. Villas-Boas*[1]; C. A. Goulart[2]; M. R. Morelli[2]; D. Souza[2]; 1. São Paulo State University, Brazil; 2. Federal University of Sao Carlos, Brazil

Proton conductivity perovskite-type materials have been investigated as alternative electrolyte materials for low temperature working Solid Oxide Fuel Cells (LT-SOFC). Perovskite materials such as yttrium-doped barium zirconates are the most attractive, however, the refractory nature of zirconium often requires high processing temperatures (>1500°C) to reach almost full densification. In this work we report the impact of 0.5 mol% $YBa_2Cu_3O_7$ (YBC) additions to $BaZr_{0.8}Y_{0.2}O_{3-\delta}$ (BZY), since YBC possess low melting point (<1200°C) and its composition has compatibility with the BZY system elements, being an interesting sintering aid to form a liquid phase. The BZY powders were prepared by solid state reaction while YBC were prepared through a modified citrate process. The ceramic samples were shaped by powder pressing, sintered at 1300-1500°C in air for 5 hours and characterized by X-Ray Diffraction, Scanning Electron Microscopy and Impedance Spectroscopy which were performed in moist atmosphere ($pH_2O = 0.49$ atm) after water incorporation by exposure to water saturated at 600°C during 12 h. YBC-additions remarkably enhanced the sintering behaviour of BZY allowing sintering temperatures in order of 1300°C to reach 93% of densifications. The total conductivity at 600°C under humidified atmosphere of pressed samples sintered at 1300°C were $1.3x10^{-3}$ S/cm.

9:20 AM

(ICACC-S3-062-2017) Development of novel low-temperature processes for perovskite oxide materials for SOFCs

Y. Yamaguchi*[1]; T. Yamaguchi[1]; H. Shimada[1]; H. Sumi[1]; K. Nomura[1]; 1. National Institute of Advanced Industrial Science and Technology (AIST), Japan

Perovskite oxide materials are widely used in solid oxide fuel cells. Some perovskite oxides were successfully prepared at lower temperature less than 100°C from starting material mixtures of hydroxides and transition-metal oxide gels, compared with conventional methods. In addition, these oxides had quite high crystallinities and small particle sizes. This required only mixing and quite low-temperature heat-treatment. By-product is only water. In this study, we prepared the various materials for cathodes and electrolytes by modifying the above method. For example, it was confirmed that barium zirconate type electrolyte materials prepared at 100°C could be used as a proton-conducting electrolytes.

Proton Conductors

Room: Coquina Salon G
Session Chairs: Enrico Traversa, King Abdullah University of Science and Technology; Mihails Kusnezoff, Fraunhofer IKTS

10:00 AM

(ICACC-S3-063-2017) Understanding proton-conducting cathode for intermediate temperature proton-conducting SOFC

S. Sun*[1]; Z. Cheng[1]; 1. Florida International University, USA

Proton conducting solid oxide fuel cells (PC-SOFC) have attracted great attention due to potential for substantially improved performance at intermediate temperatures (400-600°C) over conventional oxide ion based SOFCs. However, the cathode for PC-SOFC is often considered as the rate limiting factor. In our work, electrochemical behavior and stability of $Ba_{0.5}Sr_{0.5}Co_{0.8}Fe_{0.2}O_{3-\delta}$ (BSCF) as cathode for PC-SOFC was studied using both BSCF/ $BaZr_{0.1}Ce_{0.7}Y_{0.1}Yb_{0.1}O_{3-\delta}$ (BZCYYb)/ BSCF symmetrical cell and Ni-BZCYYb/ BZCYYb/ BSCF anode-supported cells. The impacts of water (H_2O) and carbon dioxide (CO_2) on the cathode electrochemical behavior were investigated by subjecting the cathode to simulated air with different concentrations of H_2O and CO_2. Apart from expected CO_2 poisoning, significant change in impedance spectra with introduction of 3-20 vol.% moisture was observed especially at lower temperature (~450°C) with large decrease in high frequency loop and increase in middle and low frequency loops. This suggests that for PC-SOFC, water adsorption on BSCF cathode and/or BZCYYb surface can inhibit oxygen adsorption/dissociation for symmetrical cell operation. In addition, comparison of BSCF cathode with alternative cathodes such as LSCF and A_2MO_4-type cathodes with lower affinity to water was carried out to help understand the underlying oxygen reduction reaction mechanism for PC-SOFC.

10:20 AM

(ICACC-S3-064-2017) Manufacturing of $BaCe_{0.65}Zr_{0.2}Y_{0.15}O_{3-\delta}$-based membranes for hydrogen separation

D. Montaleone[1]; E. Mercadelli[1]; A. Gondolini[1]; A. Sanson*[1]; 1. ISTEC-CNR, Italy

Ceramic hydrogen separation membrane is a promising technology for obtaining pure hydrogen in a wide range of processes including power generation with pre-combustion CO_2 capture, water-gas shift, methane reforming, etc. Among the different materials, the Y-doped $BaCe_xZr_{1-x}O_{3-\delta}$ system is one of the best candidate for this application thanks to its good compromise between conductivity at medium temperatures (600 - 800°C) and stability in CO_2 and H_2O rich ambient. In this study, asymmetrical porous-dense membranes consisting of thick porous $BaCe_{0.65}Zr_{0.2}Y_{0.15}O_{3-\delta}$ (BCZY) support and a dense thin BCZY-based layer were successfully produced by tape casting. A chemical compatibility study between the proton conductive system and different electron conductive ceramic phases was considered. The different critical issues linked to the ceramic process to obtain planar and defect free BCZY-based membranes with the suitable porous-dense microstructure were studied and faced as well.

10:40 AM

(ICACC-S3-065-2017) Low Temperature Solid Oxide Fuel Cell (LT-SOFC) for direct conversion of methane to methanol and other liquids (Invited)

R. Maric*[1]; A. Poozhikunnath[1]; A. Torabi[2]; M. Aindow[3]; 1. University of Connecticut, USA; 2. Fuel Cell Energy, USA; 3. University of Connecticut, USA

The direct conversion of methane to liquid fuels and chemicals is highly sought, especially at low-scale to more effectively utilize remote natural gas resources. Direct conversion has proven to be very challenging, because methane is highly unreactive, and tends to over-oxidize once activated. In this study a process for direct catalytic Gas-To-Liquid (GTL) conversion of methane by electrochemical means is discussed. The process uses the Low Temperature Solid Oxide Fuel Cell (LTSOFC) with an alkali carbonate/ceria based composite electrolyte and a GTL catalyst impregnated anode that converts methane rich fuel to methanol. The work described here focuses on the development of the LTSOFC, with an operating temperature < 500°C, with emphasis on the development of a fully dense ceria based electrolyte on a porous anode substrate using a versatile and cost-effective flame spray pyrolysis technique known as Reactive Spray Deposition Technology (RSDT). This synthesis technique has been used to create electrodes with gradient porosity and a fully dense electrolyte layer on a porous catalyst infiltrated anode support while keeping the substrate temperature at less than 700°C. The characterization of the deposited ITSOFC layers have been done using a combination of XRD, SEM and TEM, while a Ga ion FIB has been used to analyze the cross-section.

11:10 AM

(ICACC-S3-066-2017) Application of Bilayer MIECs in Low Temperature SOFCs

A. Jaiswal[2]; A. Pesaran*[1]; K. Duncan[2]; s. Omar[2]; E. Wachsman[2]; 1. University of Maryland College Park, USA; 2. University of Maryland, USA

Mixed ionic electronic conductors (MIECs) are critical components for a number of applications including solid oxide fuel cells (SOFCs), batteries and permeation membranes. In particular for SOFCs, MIECs are useful as cathode and anode materials ($La_{1-x}Sr_xCo_{1-y}Fe_yO_3$ [1], $Ba_{1-x}Sr_xCo_{1-y}Fe_yO_3$ [2], $La_{1-x}Sr_xCr_{1-y}V_yO_3$ [3]). Application of MIECs as electrolytes in SOFCs is limited due to their electronic conductivity, which results in internal short circuit and reduced cell open circuit voltage. An interesting approach to alleviate this problem is a combination of two MIECs in a bilayer form like doped-CeO_2/stabilized-Bi_2O_3 [4] and doped-CeO_2/stabilized-ZrO_2 [5]. Stabilized-ZrO_2 is a pure ionic conductor under fuel cell operating conditions, while doped-CeO_2 becomes an n-type electronic conductor under reducing conditions [6]. On the other hand, stabilized-Bi_2O_3 has been reported to be a p-type electronic conductor under oxidizing conditions [7]. In this paper, we look at bilayer MIEC electrolytes, based on doped-CeO_2 and stabilized-Bi_2O_3, in terms of the defect chemistry, transport properties and resultant electrochemical performance. The analysis will be supported by electrochemical cell test results under fuel cell operating conditions and application of a continuum-level electrochemical model [8]. The findings will be discussed with respect to the design of better electrolytes and electrodes for low temperature SOFCs.

11:30 AM

(ICACC-S3-067-2017) Tuning the electrical and dielectric properties of $Na_{0.5}Bi_{0.5}TiO_3$ perovskite by chemical doping

F. Yang*[1]; L. Li[1]; P. Wu[1]; C. Tumilson[2]; E. Velázquez[1]; H. Zhang[1]; I. M. Reaney[1]; D. C. Sinclair[1]; 1. University of Sheffield, United Kingdom; 2. The Queen's University Belfast, United Kingdom

Our previous studies show the conventional lead-free piezoelectric material $Na_{0.5}Bi_{0.5}TiO_3$ (NBT) can be tuned into a novel oxide-ion conductor by controlling the Na/Bi ratio of the starting composition. Here we demonstrate the electrical properties of NBT and its conduction mechanism can be further tailored by chemical doping. NBT can exhibit thress types of electrical behaviour depending on the doping mechanism, doping level and dopant nature. Acceptor doping, e.g., Na^+, Ca^{2+}, Sr^{2+} and Ba^{2+} on Bi-site of Mg^{2+}, Sc^{3+} and Ga^{3+} on Ti-site can enchance the oxygen ion conductivity; Partial replacement of Ti^{4+} by transition metal ions, e.g., Ni^{2+}, Fe^{3+}, Co^{3+} and Mn^{5+}, can turn NBT into a mixed ionic-electronic conductor; Donor doping, e.g., Nb^{5+} on Ti-site, changes the conduction mechanism from predominately ionic to mixed ionic-electronic and finally predominately electronic with increasing doping level. Tuning of dielectric properties is also feasible, e.g., a combination of doping Ba^{2+} onto A-site to increase permittivity and doping Nb^{5+} onto Ti-site to kill the loss. This work shows the flexibility of controlling the electrical and dielectric properties of NBT by chemical doping, which makes NBT promising materials in various applications as oxide-ion conductor, mixed ionic/electronic conductor and dielectric/piezoelectric materials.

S5: Next Generation Bioceramics and Biocomposites

Bioceramics IV

Room: Coquina Salon F
Session Chairs: Faleh Tamimi, McGill University;
Mohamed Rahaman, Missouri University of Science & Technology

8:30 AM

(ICACC-S5-024-2017) Robocasting of Silicon Nitride Implants with Complex Shape and Architecture for Orthopedic Applications

M. N. Rahaman*[1]; W. Xiao[1]; S. Zhao[1]; D. O'Brien[2]; S. Bal[3]; 1. Missouri University of Science & Technology, USA; 2. Amedica Corporation, USA; 3. University of Missouri, Columbia, USA

Silicon nitride implants are currently used clinically in spinal repair and are under consideration for use as bearings in total joint arthroplasty. In the present study, silicon nitride parts with complex three-dimensional architectures were fabricated by robocasting. The development of an aqueous paste with the requisite rheology for continuous printing of silicon nitride parts is described. A combination of sintering and hot isostatic pressing resulted in the production of parts with complex architectures and a fully dense silicon nitride phase composed of fibrous grains. Four-point bending of the as-fabricated parts showed an average flexural strength of 552 MPa. Together, these results indicate that robocasting could provide a viable route to create silicon nitride implants with complex architectures and anatomically relevant shapes for applications in orthopedic surgery.

8:50 AM

(ICACC-S5-025-2017) Two-Dimensional Magnesium Phosphate Nanosheets Form Highly Thixotropic Gels That Up-Regulate Bone Formation (Invited)

F. Tamimi*[1]; M. Laurenti[1]; A. Al Subaie[1]; M. Abdallah[1]; A. Cortes[2]; J. Ackerman[2]; H. Vali[1]; K. Basu[1]; Y. Zhang[1]; M. Murshed[1]; S. Strandman[3]; J. Zhu[3]; N. Makhoul[1]; J. Barralet[1]; 1. McGill University, Canada; 2. Harvard Medical School, USA; 3. Université de Montreal, Canada

Hydrogels composed of two-dimensional (2D) nanomaterials have become an important alternative to replace traditional inorganic scaffolds for tissue engineering. Here, we describe a novel nanocrystalline material with 2D morphology that was synthesized by tuning the crystallization of the sodium-magnesium-phosphate system. We discovered that the sodium ion can regulate the precipitation of magnesium phosphate by interacting with the crystal's surface causing a preferential crystal growth that results in 2D morphology. The 2D nanomaterial gave rise to a physical hydrogel that presented extreme thixotropy, injectability, biocompatibility, bioresorption, and long-term stability. The nanocrystalline material was characterized in vitro and in vivo and we discovered that it presented unique biological properties. Magnesium phosphate nanosheets accelerated bone healing and osseointegration by enhancing collagen formation, osteoblasts differentiation, and osteoclasts proliferation through up-regulation of COL1A1, RunX2, ALP, OCN, and OPN. In summary, the 2D magnesium phosphate nanosheets could bring a paradigm shift in the field of minimally invasive orthopedic and craniofacial interventions because it is the only material available that can be injected through high gauge needles into bone defects in order to accelerate bone healing and osseointegration.

9:20 AM

(ICACC-S5-026-2017) Bone Regeneration in Rat Calvarial Defects Implanted with Closed and Open Hollow Hydroxyapatite Microspheres

M. N. Rahaman*[1]; Y. Shen[1]; 1. Missouri University of Science & Technology, USA

Hollow hydroxyapatite microspheres with a high surface area nanoporous shell can provide an attractive device for controlled delivery of proteins such as growth factors in bone regeneration. In the present study, hollow hydroxyapatite microspheres were sectioned to reveal their inner concave surface. The capacity of these "open" hydroxyapatite microspheres to regenerate bone in a rat calvarial defects model was compared with the conventional "closed" hydroxyapatite microspheres. Open and closed microspheres without or with an osteogenic growth factor (bone morphogenetic protein-2) were evaluated and the results will be discussed.

9:40 AM

(ICACC-S5-027-2017) Synthesis and Characterization of $CaSiO_3$-$CaCO_3$-SiO_2 Composites Produced via Hydrothermal Reaction

B. Beyoglu*[1]; R. Riman[1]; 1. Rutgers University, USA

$CaSiO_3$-$CaCO_3$-SiO_2 bioactive ceramic composites have been produced by hydrothermal liquid phase densification (HLPD) of high temperature sintered (HTS) $CaSiO_3$ ceramics. The effects of HLPD and HTS on microstructure and its relation to mechanical strength were investigated. During HTS, with increasing sintering temperature to 1200°C, an increase in grain and pore size, the disappearance of the needle-shape of crystals and phase transformation to pseudowollastonite was observed. Rapid grain growth resulted in a decrease in the strength. Therefore, 1150°C is ideal HTS temperature to provide bonding and neck growth between the particles, so to achieve strengthening to some extent. Our results show HLPD allows for gradual densification and strengthening of the material by introducing $CaCO_3$, a secondary phase, into the pores of the partially densified $CaSiO_3$ compact and provides a solution to the low mechanical strength issue arising from poor densification of $CaSiO_3$ by HTS. Achieved relative density is 68.5% for HTS-$CaSiO_3$ and 84.3% for HLPD/HTS-$CaSiO_3$. Compression strength increased to 279±31MPa from 130±6MPa by applying HLPD to HTS-$CaSiO_3$. The results suggest that $CaSiO_3$ bioactive ceramic composite treated with HLPD and HTS is a promising material for bone implant applications due to its mechanical properties being similar to those of human cortical bone.

S8: 11th International Symposium on Advanced Processing and Manufacturing Technologies for Structural and Multifunctional Materials and Systems (APMT11)

Design-Oriented Manufacturing II

Room: Coquina Salon B
Session Chairs: Francis Cambier, Belgian Ceramic Resaerch Centre;
Surojit Gupta, University of North Dakota

8:30 AM

(ICACC-S8-043-2017) Innovations with advanced ceramics (Invited)

R. Lenk*[1]; 1. CeramTec GmbH, Germany

Advanced ceramic materials make applications possible today that were virtually inconceivable just yesterday. Due to their unique material properties, technical ceramics are considered to be one of the most efficient materials of our time. Advanced ceramics are also referred to as technical ceramics, engineering ceramics or industrial ceramics. These terms cover many different and in part highly specialized ceramic materials with unique mechanical, electrical, thermal and biochemical properties and property combinations. These specific characteristics can be further developed, optimized and matched for use in technical applications. It is also possible to combine different properties. Materials like these can be used to design advanced components that perform the required tasks with optimum precision and can be light years ahead of competing materials, such as metal or plastic, in the respective field of application. Components made from ceramic materials are increasingly the only solution available for technical challenges that cannot be overcome by conventional materials. The areas of application for components made from technical ceramics are as varied as the advantages of advanced ceramic materials themselves. Markets include vehicle and automotive engineering, electronics, energy and environment, equipment and mechanical engineering, and medical engineering.

9:00 AM

(ICACC-S8-044-2017) Sintering of high-energy ball milling $CaLa_2S_4$-ZnS powder mixtures (Invited)

Y. Li[1]; Y. Wu*[1]; 1. Alfred University, USA

Due to their favorable infrared (IR) transmittance, calcium lanthanum sulfide ($CaLa_2S_4$) and zinc sulfide (ZnS) are considered as attractive ceramics for the IR optical applications. In this study, commercially available $CaLa_2S_4$ powders have been mixed and ground with colloid-processed ZnS nanopowders by using a high-energy ball milling. $CaLa_2S_4$-ZnS composite IR optical ceramics have been fabricated via the hot pressing and field assisted sintering of the composite powders. The phase compositions and microstructures of the $CaLa_2S_4$-ZnS ceramics have been characterized to investigate the influences of composites compositions and processing conditions on the cubic-hexagonal phase transition of ZnS. The sintering behaviors have also been studied to understand the densification mechanisms of the composite powders with the different consolidation routes. The research has also studied the optical and mechanical properties of the composited ceramics.

9:30 AM

(ICACC-S8-045-2017) Solid-state single crystal conversion in isotropic and anisotropic ceramics

Y. Liu*[1]; Y. Wu[1]; 1. Alfred University, USA

Solid-state single crystal conversion has been widely observed in some oxide materials. The microstructure control is very important in single crystal conversion, and there are some factors needed to be considered in order to achieve a better control. The grain growth behaviors and grain morphologies from various materials may impose different energy potentials for the single-crystal to grow, since the single-crystal has to concur the energy barrier of the polycrystalline matrix. In this study, both of an isotropic CaF_2 and an anisotropic ceramic $Sr_5(PO_4)_3F$ were examined for their potentials in solid-state single crystal conversion. The grain growth in CaF_2 was almost equiaxed whereas the $Sr_5(PO_4)_3F$ grains underwent elongation and rotation during the heat treatment. However, the dimensions of the grown crystals are similar in two ceramic systems in terms of the crystal growth. It was found that the crystal growth was more related to the average grain size rather than the grain morphology.

9:50 AM

(ICACC-S8-046-2017) Innovative processing and manufacturing of SiC/SiC CMCs for energy/environmental applications (Invited)

K. Shimoda*[1]; T. Hinoki[2]; 1. National Institute for Materials Science (NIMS), Japan; 2. Kyoto University, Japan

Over the past two decades, the vast majority of CMCs' research in harsh environments for aerospace and nuclear applications has focused on the SiC/SiC system, which consists of the building up of a SiC matrix around the SiC fibers, due to low gravity, excellent high temperature strength and oxidation resistance. There are four common ways to create SiC matrix: through CVI, MI, PIP and HP via LPS. In general, SiC/SiC CMCs exhibit a pseudo-ductile fracture behavior by de-bonding and sliding at the weak fibers/matrix interface like PyC or BN. The interface material and its thickness are keys to determine mechanical properties and fracture behavior. However, PyC or BN at the interface is the weakest link considering the environmental effect because of its tendency to oxidize in air or steam. Recently, we have successfully developed a pseudo-ductile SiC/SiC CMC without any interface using BN particle distribution in SiC matrix (SiC/BN+SiC). The SiC/BN+SiC displayed the very high mechanical values, such as the UTS (~300 MPa), the PLS (~250 MPa). No apparent degradation in strength after air exposure up to 1500°C was observed compared to that of the as-sintered material. This is because oxidation was very limited near surface.

10:30 AM

(ICACC-S8-047-2017) Hybrid aerosol deposition as a new tool for delight design

K. Shinoda*[1]; T. Saeki[1]; M. Mori[2]; J. Akedo[1]; 1. National Institute of Advanced Industrial Science and Technology (AIST), Japan; 2. Ryukoku University, Japan

Ceramic coatings onto three-dimensional objects that are consisting of metals, ceramics, and polymers will provide us a new tool for additive manufacturing and new design for lightweight components and heat resistant components. Plasma spray technology has several advantages such as its applicability to three-dimensional objects with a combination to an industrial robot but is yet to solve several issues such as thermal damage to substrates, adhesion strength between coatings and substrates, and density of coatings. Aerosol deposition technology is a relatively new method that can fabricate dense ceramic coatings that adhere strongly to substrates without thermal damage. We explored a boundary between plasma spray and aerosol deposition and found that a certain regime could be used as new coating solution, which we named a hybrid aerosol deposition (HAD) process. In this study, we will present our recent challenge on the development of the HAD process and also present its applicability to delight design. This work was supported in part by NEDO SIP, Innovative Design /Manufacturing Technologies, High-Value Added Ceramic Products Manufacturing Technologies.

10:50 AM

(ICACC-S8-048-2017) Environmental-friendly fine-particle spray coatings by rf induction plasma

T. Saeki*[1]; K. Shinoda[1]; M. Mori[2]; J. Akedo[1]; 1. National Institute of Advanced Industrial Science and Technology (AIST), Japan; 2. Ryukoku University, Japan

The ceramic materials are used in many industrial applications. The realization of environmental-friendly process is one of the important issues in manufacturing and coating technologies of ceramics. In this aspect, the formation of dense, uniform, and homogeneous coatings by the use of fine powder as raw material attracts attention in the recent spray coating technologies. Thermal spray is a popular process that utilizes the melting and solidification process of the particles, meanwhile aerosol deposition (AD) method is also gaining the attention because this method can form a very dense ceramic coatings without thermal heating. However, the AD method is inferior to thermal spray method in terms of deposition efficiency of raw material and deposition rate. Therefore, we have developed a new coating process named a hybrid aerosol deposition (HAD) method. The HAD process utilizes rf induction plasma with a lower power input (~a few kW level) compared to conventional plasma spray and improves the weak points of the AD method mentioned above with keeping the advantage of the AD method. As a case study, the formation of Al_2O_3 coatings having high density and uniformity by using HAD process will be shown. This work was supported in part by NEDO SIP, Innovative Design /Manufacturing Technologies, High-Value Added Ceramic Products Manufacturing Technologies.

11:10 AM

(ICACC-S8-049-2017) TEM Analysis of Diffusion-Bonded Silicon Carbide Ceramics Joined Using Metallic Interlayers

T. Ozaki*[1]; Y. Hasegawa[1]; H. Tsuda[2]; S. Mori[2]; M. C. Halbig[3]; R. Asthana[4]; M. Singh[5]; 1. Technology Research Institute of Osaka Prefecture, Japan; 2. Osaka Prefecture University, Japan; 3. NASA Glenn Research Center, USA; 4. University of Wisconsin-Stout, USA; 5. Ohio Aerospace Institute, USA

Silicon Carbide (SiC) is a promising material for thermostructural applications due to its excellent high-temperature mechanical properties, oxidation resistance, and thermal stability. However, joining and integration technologies are indispensable for this material in order to fabricate large size and complex shape components with desired functionalities. Although diffusion bonding techniques using metallic interlayers have been commonly utilized to bond various SiC ceramics, detailed microstructural observation by Transmission Electron Microscopy (TEM) of the bonded area has not been carried out due to difficulty in preparing TEM samples. In this study, we tried to prepare TEM samples from joints of diffusion bonded SiC ceramics by Focused Ion Beam (FIB) system and carefully investigated the interfacial microstructure by TEM analysis. The samples used in this study were SiC fiber bonded ceramics (SA-Tyrannohex™: SA-THX) diffusion bonded with metallic interlayers such as Ti, Ti/Mo, Mo-B and Ti/Cu. In this presentation, we report the microstructure of diffusion bonded SA-THX mainly with Ti/Cu interlayers obtained by TEM observations, and the influence of metallic interlayers on the joint microstructure and microhardness will be discussed.

11:30 AM

(ICACC-S8-050-2017) Electric-field assisted mass transport and its applications in ceramic joining and shaping

Y. Gao*[1]; Y. Wang[3]; L. An[2]; 1. Southwest Jiaotong University, China; 2. University of Central Florida, USA; 3. Northwestern Polytechical University, China

"Flash-sintering", where with the assistance of electric field ceramics can be densified in just a few seconds at furnace temperatures much lower than normal sintering temperatures, has received extensive attention. Typically, flash sintering consists of following stages: an incubation stage where electric current increases with time slowly; an avalanche stage where the current increases quickly with time, followed by a rapid current surge; and a steady stage where both current and voltage become constant. It was found that the most of densification process occurred concurrently with the current surge in the avalanche stage. While flash-sintering has been demonstrated in a variety of oxide material systems, the mechanism behind this process is still an open question. In this study, the effect of applied voltage and electric current on the electric conduction, densification, and grain growth of zirconia ceramics has been systemically investigated and discussed, with the focus on their effect on the defect generation and transport. A possible mechanism of flash-sintering initiation is proposed. Furthermore, owing to the rapid mass transport induced by electric filed, flash-sintering has been employed to ceramics welding and plastic deformation. It is believed that these applications with electric field will greatly advance the ceramic manufacturing techniques.

S13: Advanced Materials for Sustainable Nuclear Fission and Fusion Energy

Accident-tolerant Fuels II and Radiation Effects

Room: Coquina Salon H
Session Chairs: Yutai Katoh, Oak Ridge National Laboratory; Izabela Szlufarska, University of Wisconsin

8:30 AM

(ICACC-S13-038-2017) Westinghouse Accident Tolerant Fuel Program Update on SiC Composite Cladding Development (Invited)

P. Xu*[1]; E. Lahoda[2]; Y. Long[1]; 1. Westinghouse Electric Company, USA; 2. Westinghouse Electric Company, USA

The Westinghouse accident tolerant fuel (ATF) program is aimed at providing a significant increase in fuel robustness and accident tolerance for beyond design basis accidents at an economically attractive price to nuclear power providers. The Westinghouse ATF program, which began in 2004, advanced significantly during the current first phase (1A and 1B) begun in 2012 of this Department of Energy (DOE) program. Phase 1B will culminate in the manufacture of fuel rods that will be irradiated at steady state conditions in the Advanced Test Reactor and the Halden test reactor (Phase 2) for up to 6 years. Fuel transient tests in TREAT and Halden will follow. The goal is to load lead test assemblies that consist of SiC cladded fuel rods in a commercial reactor in 2022. This talk will provide the current status of the Westinghouse ATF program and outline the tasks that will be performed in Phase 2 with a focus on SiC ceramic composite matrix cladding. The corrosion resistance was evaluated using out-of-pile autoclave tests. In-pile irradiation data from the MIT reactor will be discussed. Fuel rod performance analysis was performed for normal operating conditions and off-normal conditions. Mechanical properties were determined using expanding plug tests with in-situ x-ray tomography and digital image correlation. Acoustic emission was used to detect cracking formation.

8:50 AM

(ICACC-S13-039-2017) Irradiation behaviors of SiC/SiC candidated for PWR accident tolerant fuel cladding (Invited)

X. Li*[1]; 1. Northwestern Polytechical University, China

Due to its excellent high temperature strength and high temperature chemical inert to environmental, SiC/SiC is considered as a promising material for PWR accident tolerant fuel cladding. However, this kind of material is not only seriously lack of the reliable in-pile service data of PWR, but also lack of the data when a LOCA event happens. In order to provide a technical support for cladding fabrication and in-pile assessment, this work focuses more on studying of the microstructure evolution and its consequent results in mechanical property, dimensional stability, pressure tightness and thermal conductivity degradation behaviors of SiC/SiC composite after neutron irradiation tailored in both normal and LOCA conditions. Tested materials were developed in our laboratory, and were put in a Chinese Research PWR reactor. The main objectives of this work are to deduce the degradation mechanisms of the material after in-pile PWR irradiation.

9:10 AM

(ICACC-S13-040-2017) Permeability of SiC composite LWR fuel cladding under in-situ loading conditions

G. Vasudevamurthy[1]; E. Song[1]; G. Jacobsen[1]; C. Deck*[1]; 1. General Atomics, USA

Excellent thermo-mechanical and irradiation properties make chemical vapor infiltrated (CVI) continuous SiC fiber reinforced SiC matrix (SiC_f/SiC_m) composites highly desirable candidates to replace Zircaloy in light water reactors (LWRs) to improve accident tolerance. However, these ceramic composites have to be engineered to achieve the same level of impermeability 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 ultimate deployment in LWRs. In this context, a test method for measuring the permeability of LWR sized SiC_f/SiC_m tubes under internal mechanical loading conditions was designed and conducted. Impermeability was measured using a highly sensitive helium leak detector connected to a customized test rig. Several methods were investigated for applying in-situ loads and results from this investigation will be presented. Impermeability data will be presented in the context of the stress/strain response of the cladding and the resulting implications to normal operating and accident conditions discussed. Acknowledgements : This work was supported by General Atomics IR&D funding and the Department of Energy under Award Number DE-NE0000566.

9:30 AM

(ICACC-S13-041-2017) Post-Irradiation Validation of High Heat Flux SiC/SiC Cladding Irradiation Design

C. Petrie*[1]; T. Koyanagi[1]; C. Deck[2]; Y. Katoh[1]; K. Terrani[1]; 1. Oak Ridge National Lab, USA; 2. General Atomics, USA

SiC/SiC composite materials are being considered for nuclear fuel cladding applications due to their high temperature strength, radiation hardness, and oxidation resistance. For fuel cladding applications, these composites are required to withstand the evolving stress state over the fuel lifetime when exposed to intense neutron radiation under a prototypic high heat flux (and thus a large temperature gradient) as the rod internal pressure increases due to released fission gas buildup. An irradiation capsule was designed to test SiC/SiC composites under representative light water reactor heat flux (~0.6 MW/m^2) and outer surface temperature (300-350°C) during irradiation in the High Flux Isotope Reactor. An engineered aluminum foil was developed to absorb the swelling of the cladding while keeping the outer surface temperature constant to within 12°C. This presentation will discuss the capsule design and thermal validation efforts, as well as post-irradiation examination of two capsules that contained a total of six cladding specimens. Specimens showed no signs of surface cracking or other surface degradation. Dilatometric analysis of the passive temperature monitors placed inside the capsules predicts temperatures during irradiation that agree well with design calculations, with two calculated temperatures agreeing with experiment to within 10°C.

9:50 AM

(ICACC-S13-042-2017) Mechanical properties of SiC composites neutron irradiated under LWR relevant temperature and dose conditions

T. Koyanagi*[1]; Y. Katoh[1]; 1. Oak Ridge National Laboratory, USA

Silicon carbide (SiC) fiber reinforced SiC matrix (SiC/SiC) composites have historically been investigated for high-temperature structural applications in nuclear area. Recent reserach and development of accident tolerant SiC/SiC fuel cladding of light water reactors (LWRs) revealed the lack of neutron irradiation effect studies in the LWR-relevant conditions. The limited data for an early nuclear grade SiC/SiC composite irradiated at 325°C to 39dpa indicated significant degradation in the mechacnial properties, presenting a concern for radiation stability of these materials. This study counducted neutron-irradiaitons of newer generation nuclear grade SiC/SiC composites in the LWR-relevant temperature and dose conditions, at 230-340°C to 2.0 and 11.8 dpa. The materials used were chemical vapor infiltrated SiC/SiC composites reinforced with pyrolytic carbon coated Hi-Nicalon Type S, Tyranno SA3, and Ultra SCS SiC fibers. Irradiaiton resistance of these composites will be discussed based on the dynamic Young's modulus, flexural behavior, and microstructure. This research was supported by the Advanced Fuels Campaign of the Fuel Cycle R&D program in the Office of Nuclear Energy, U.S. DOE under contract DE-AC05-00OR22725 with ORNL managed by UT-Battelle, LLC.

10:30 AM

(ICACC-S13-043-2017) Defect kinetics and long-term evolution of grain boundaries in irradiated SiC (Invited)

I. Szlufarska*[1]; X. Wang[1]; H. Jiang[1]; 1. University of Wisconsin, USA

While grain boundaries (GBs) can act as sinks of defects in irradiated materials, much less is known about the effects of radiation on the evolution of atomic structure and the sink strength of GBs. In SiC there is an unbalanced flux of C interstitials to GBs, but it is unclear how this excess of C is accommodated by GBs. Here, we combined multi-scale modeling, scanning transmission electron microscopy, and electron energy loss spectroscopy to investigate defect kinetics and chemical changes taking place in GBs during irradiation. We found that GBs in CVD-SiC are intrinsically carbon-poor and the relative C composition can be as low as 45%. This carbon depletion disappeared in samples irradiated to 1 dpa at 300 C, which is likely due to the unbalanced flux of C interstitials to defect sinks and low diffusivity of defects at GBs. Interestingly, the C depletion appeared again in samples irradiated at 600 C and the depletion was found to more significant than in the non-irradiated sample. These results indicate that the role of GBs may change from defect clustering reservoirs at low irradiation temperatures to defect diffusion channels at high irradiation temperatures. Changes in GBs chemistry due to irradiation are expected to impact such properties of SiC as its fracture strength, corrosion resistance, its ability to provide a barrier to diffusion of fission products in nuclear reactor applications.

10:50 AM

(ICACC-S13-044-2017) Advanced X-ray characterization of neutron irradiated SiC (Invited)

D. Sprouster*[1]; L. Ecker[1]; E. Dooryhee[1]; T. Koyanagi[2]; Y. Katoh[2];
1. Brookhaven National Laboratory, USA; 2. Oak Ridge National Lab, USA

In the present work, we discuss the application of advanced, high-resolution X-ray characterization methods to quantify radiation damage in polycrystalline ceramics. We focus on results relating to the X-ray diffraction and pair distribution function analysis of neutron irradiated 3C-SiC. We readily observe several irradiation-induced structural changes including an increase in the anisotropic microstrain, a decrease in the coherent grain size and an expansion in the crystalline lattice. We also identify several features in the microstructure that are fluence and irradiation temperature dependent. Some of the changes in the microstructure can be directly linked to specific defects observed with other characterization methods (such as microscopy). By quantifying changes in the microstructure and atomic structure, we aim to develop a more detailed understanding of the radiation response of SiC. We also discuss the usefulness of the X-ray techniques in characterizing bulk samples, simultaneously measuring short, intermediate and long range order. Such studies are potentially important to build mechanistic models of material performance and to understand the susceptibility of various microstructures to radiation damage for advanced energy applications. The work is partially supported by the U.S. DOE, Office of FES under contract DE-AC05-00OR22725 with ORNL managed by UT-Battelle, LLC

11:10 AM

(ICACC-S13-045-2017) Probing structural disorder in neutron irradiated silicon carbide by Raman spectroscopy

T. Koyanagi*[1]; M. Lance[1]; Y. Katoh[1]; 1. Oak Ridge National Laboratory, USA

Structural disorder by neutron bombardment is known to cause swelling, irradiation creep, and degradation of thermal diffusivity of silicon carbide (SiC). It will be useful to develop nondestructive techniques to probe and quantifiy the structural disorder in SiC for monitoring material performance in nuclear applications. The objective of this study is development of such an evaluation technique using Raman spectroscopy. High-purity polycrystalline beta-SiC was irradiated in the High Flux Isotope Reactor at temperatures of 380–1180°C and damage ranging from 0.011–1.87 dpa. We will show how peak positions of transverse optical phonon lines in the Raman spectra correlate with the radiation-induced lattice strain obtained from X-ray diffraction. In addition, the changes in the Raman-measured longitudinal optical phonon line shape and position, by irradiation, are shown to be caused by a combination of the lattice strain and the phonon confinement. Lastly, we will discuss the application of Raman spectroscopy in determining of the localized irradiation temperature, i.e. temperature mapping, based on the peak positions. This work is supported by the U.S. DOE, Office of Fusion Energy Sciences and Office of Nuclear Energy under contract DE-AC05-00OR22725 with ORNL managed by UT-Battelle, LLC. Research was conducted using HFIR, which is sponsored by the Office of Basic Energy Sciences, U.S. DOE.

11:30 AM

(ICACC-S13-046-2017) Ostwald Ripening Retardation Due to Radiation Induced Point Defect

E. Hernandez*[2]; V. Tikare[3]; L. Wang[1]; 1. University of Michigan, USA;
2. US Army Research Laboratory, USA; 3. Sandia National Laboratories, USA

Understanding how radiation damage affects precipitate evolution could play a vital role in determining optimal nanostructured material configurations for radiation tolerance applications,. e.g. ODS. We developed a model that is able to simulate the influence of radiation damage defects on microstructural evolution. Furthermore, we implement a systematic approach for generating NRT defects, which allows for straightforward inclusion of physical input parameters like ion flux. Our model couples a hybrid phase field–Potts Monte Carlo code (developed to study grain growth) to a rate theory framework that models the evolution of the radiation defects. We are able to show that radiation damage has a significant influence on the evolution of precipitates. Varying the defect production rate, we can identify how precipitate evolution deviates from Ostwald ripening kinetics. Lastly, we are able to capture the different stages of defect accumulation and link it to the precipitate evolution.

11:50 AM

(ICACC-S13-047-2017) Positron Annihilation Spectroscopy Investigation of Vacancy Clusters in Neutron-irradiated 3C-SiC

X. Hu*[1]; T. Koyanagi[1]; Y. Katoh[1]; 1. Oak Ridge National Lab, USA

Silicon carbide (SiC) has been considered as a fuel and structural material in nuclear systems since the 1960s, due to its high-temperature stability, chemical inertness, and exceptional irradiation resistance. Microstructural development and mechanical property changes in SiC subject to neutron and ion irradiation have been studied extensively and understood well. However, the information on small vacancy cluster (i.e., those invisible in TEM) evolution in SiC exposed to neutron irradiation is still lacking, and such small clusters are suspected to play an important role in swelling, radiation creep, and thermal conductivity degradation. In this study, positron annihilation lifetime spectroscopy was used to characterize the vacancy clusters in 3C-SiC subject to various neutron irradiation conditions, while positron coincidence Doppler broadening enabled the determination of the chemical identity of the atoms surrounding the positron trapping sites. The correlation between the detected vacancy clusters and the measured swelling will also be discussed. Acknowledgement The work presented in this paper was supported by Laboratory Directed R&D funds at ORNL and by the US Department of Energy Office of Fusion Energy Science under grants DE-AC05-00OR22725 with UT-Battelle LLC.

Author Index

*** Denotes Presenter**