The American Ceramic Society

44th International Conference & Exposition on Advanced Ceramics and Composites

ABSTRACT BOOK

January 26–31, 2020
Daytona Beach, Florida

The American Ceramic Society
www.ceramics.org

Introduction

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How to Use the Abstract Book

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

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Monday, January 27, 2020

Plenary Session

Room: Coquina Salon D
Session Chairs: Surojit Gupta, University of North Dakota; Valerie Wiesner, NASA Langley Research Center

8:50 AM

(ICACC-PLEN-001-2020) Ion-Beam Modification and Nanostructure Evolution in Ceramics

W. J. Weber*[1]

1. University of Tennessee, Materials Science & Engineering, USA

Ion-beam modification is a result of both inelastic energy loss to electrons and elastic energy loss to atomic nuclei. The effects of these different energy loss pathways on defect production, nanostructure evolution and phase transitions in ceramics are complex. Using experimental and computational approaches, the separate and coupled effects of energy loss to electrons and atomic nuclei on ion-beam modification of ceramics have been investigated over a range of ions and energies. These studies demonstrate: 1) formation of nanoscale phase transitions by high electronic energy dissipation and by synergistic effects between pre-existing defects and electronic energy loss; 2) coupled effects of energy loss to electrons and atomic nuclei on nanograin growth; and 3) ionization-induced defect annealing. The diverse range of effects provides new paradigms in ion-beam modification of ceramics on the role of electronic energy loss in defect engineering and functional nanostructure formation. This research has advanced understanding on the role of defects in electronic energy dissipation and electron-phonon coupling, and the knowledge gained provides insights for creating novel interfaces and nanostructures with controlled morphologies, phases and local strain, which can be employed to engineer functionalized thin film structures with tunable electronic, ionic, magnetic and optical properties.

9:30 AM

(ICACC-PLEN-002-2020) Development of precursor ceramics using organic silicon polymer

T. Ishikawa*[1]

1. Tokyo University of Science, Yamaguchi, Applied Chemistry, Japan

This presentation will discuss our unique precursor ceramics developed by author and coworkers in Ube Industries, Ltd. We have developed many types of functional ceramics using polycarbosilane as a raw material. Since 1983, several grades of SiC-based fibers have been produced from polycarbosilane in Ube Industries, Ltd. Of these grades, we developed the highest heat-resistant SiC-polycrystalline fiber (Tyranno SA), which can withstand up to 2000ºC, using organic silicon polymer containing small amount of aluminum as a starting material (T.Ishikawa, et al, Nature, 391 (1998) 773). In the same year, we also developed a new type of tough, thermally conductive SiC composite (SA-Tyrannohex) with high strength up to 1600ºC in air. This ceramic consists of a highly ordered, close-packed structure of very fine hexagonal columnar SiC-fibers with a thin interfacial carbon layer between them (T.Ishikawa, et al., Science, 282 (1998) 1295). Furthermore, we successfully developed a strong photocatalytic fiber (TiO_2/SiO_2 fiber) with a gradient surface layer composed of TiO_2-nanocrystals making the best use of controlled phase separation (bleed out) of additives (titanium (IV)tert-butoxide) contained in polycarbosilane (T.Ishikawa, et al., Nature, 416 (2002) 64). In this presentation, the development story and subsequent progress will be discussed.

10:40 AM

(ICACC-PLEN-003-2020) Ceramic biomaterials: From traditional technologies to novel applications

K. Balazsi*[1]; C. Balazsi[1]

1. HAS Centre for Energy Research, Hungary

The 400 000 artificial hip joint operations made every year in the word and there are 25 000 000 people with a total hip replacement. The wear and risk of the implant loosening increases so that after 10 years 10-20% of the implants have to be renewed. Biomaterials used for implant should possess some important properties in order to long-term usage in the body without rejection. The biocompatibility, mechanical, chemical and surface properties play a key role in the creation of sufficient and long term functional replacements. New fundamental research outcomes with industrial perspectives are given for understanding the applications of ceramics in load-bearing and low-load-bearing bioimplants with directions for future developments. Nowadays, Si_3N_4 is a new bioceramic with extremely good mechanical properties. Hydroxyapatite (HA) is a widely used bioceramic in implantology considering its high bioactivity. A bioactive coating (HA) on the bioinert ceramic implant's surface (Si_3N_4) could help avoid the rejection from the body in the critical early few days after the operation. The preparation of bioceramics will be showed from traditional technologies to novel applications. The main trends and fundamental scientific problems will be discussed.

11:20 AM

(ICACC-PLEN-004-2020) Developing a Pathway to Microstructure-Aware Predictive Capability for the Shock/Dynamic Response of Materials

G. T. Gray*[1]

1. Los Alamos National Lab, USA

It is sixty years since Cyril Stanley Smith's seminal paper describing the effects of shock loading on the structure/property behavior of materials. While experimental observations have fostered the correlation of post-shock microstructural parameters, such as dislocations, point defects, deformation twins, and shock-induced phase products, quantitative predictive capability of the defect generation and damage evolution in materials has yet to be realized. Broadly based defect generation/storage phenomenology presenting a unified view of the material structure/property aspects of shock-wave deformation has proven difficult. Changes in design and manufacturing paradigms applied to events dominated by dynamic-loading processes have placed increased emphasis on developing physically-based predictive materials models of shock effects on materials as well as innovations in in-situ shock diagnostics. In this talk, a survey of the evolution in the state-of-our-understanding of defect generation and damage evolution is discussed and thoughts on the evolving capabilities to move shock/dynamic behavior of materials research from observation to design and control is presented. Examples of how utilizing "real-time", post-mortem, and "in-situ" experimental approaches together are needed to facilitate quantification 4D processes during dynamic/shock-wave loading will be discussed.

Special Focused Session on Diversity, Entrepreneurship, and Commercialization

Jubilee Global Diversity Awards; Entrepreneurship and Commercialization

Room: Coquina Salon E
Session Chairs: Surojit Gupta, University of North Dakota; Valerie Wiesner, NASA Langley Research Center

1:40 PM

(ICACC-DIV-001-2020) Current Technological Advances in Multi-Ceramic Additive Manufacturing

H. Yun*[1]

1. Korea Institute of Materials Science, Republic of Korea

Additive manufacturing (AM) is a fabrication process that used digital information from a computer-aided design file to stack 2D layers of various materials to produce a 3D object, without requiring any part-specific tooling. AM technologies have attracted much attention in various fields such as medicine, automotive, aerospace, consumer, and other industrial applications. However, there are still limitations in both selection of materials as well as controlling part performance. In the former case, polymers and metals are typically used as materials for high precision AM whereas the technology for ceramics is comparatively inadequate yet. In the latter case, functionality of 3D structure was controlled only by designing 3D architectures, however we can expect much advanced functionality by using multi-materials for AM process. To this purpose, our group has developed a novel digital light processing for multi-ceramic additive manufacturing. The fabrication system and processing has been successfully optimized for various types of materials. We could co-print multi-component in one structure using less amount of ceramic slurry with high resource efficiency. We could control cross-contamination between multi components by adapting novel washing process. We believe that this new technology may provide big turning point to overcome limitation of traditional ceramic forming process.

2:20 PM

(ICACC-DIV-002-2020) Ceramic and composite joints for nuclear applications

V. Casalegno*[1]

1. Politecnico di Torino, DISAT, Italy

The joining of ceramic based materials (bulk and composites) to themselves or to dissimilar materials (i.e metals) is a crucial point for nuclear energy applications, since their use is proposed in various advanced fission and fusion systems. In both cases, the main issues are the extreme thermo-mechanical loads on the joined components, the not completely known service conditions and requirements, their resistance to high temperatures, to ion/neutron irradiation and to harsh chemical environment. An overview of the last 18 years of activity at Politecnico di Torino (Italy) on different joining materials (glass-ceramics, customized brazes, etc) and joining processes (pressureless methods, conventional brazing, direct bonding, etc) used to join ceramic–based materials for nuclear applications will be presented. The morphological and mechanical characterization of the joints will be discussed. The characterization of some proposed joining materials and of the joints in nuclear environment will also be presented: with the aim of reproducing a similar damage scenario to that in nuclear plants, some joining materials have been irradiated with different ions (He, Si) and with fission neutrons at different temperatures.

3:20 PM

(ICACC-DIV-003-2020) Multi-scale thermal protective systems for extreme environments: Design, processing, properties and modeling

C. Tallon*[1]

1. Virginia Tech, Materials Science and Engineering, USA

In this work, a multi-faceted approach for design and manufacturing of multi-scale porous UHTCs is presented. Highly aligned porous channels are produced in titanium diboride using electrospun fibers as sacrificial fillers, with a multi-scale porosity between 50 and 80%, with controlled alignment to direct the flow of a second phase or divert the heat away by tailoring thermal conductivity. The microstructure and thermal and mechanical properties are characterized along and across the pores axis, and compare with analytical models. The results show that the manipulation of the highly aligned porous microstructure allow to control the thermal and mechanical properties as a function of the direction of the pores. Computational approaches are validated by experimental characterization of actual samples, as a pathway to develop predictive capabilities. This integrated combination of predictive modelling of porous UHTCs with tailored processing routes to create porous structures is key to match the desired performance.

4:00 PM

(ICACC-DIV-004-2020) Diversity and Inclusion in a large, international materials company (Invited)

K. Breder*[1]

1. Saint-Gobain, Saint-Gobain Research North America, USA

Saint-Gobain operates in 64 countries with more than 180 000 employees world-wide. Diversity and inclusion is integral to the company's strategy and is a key factor in innovation and growth for the company. At Saint-Gobain, we believe diversity is a competitive advantage. Diversity of gender, background, ethnicity, experience and thought all enable us to develop solutions that are smarter, clearer and more effective. In this presentation I will discuss why diversity and inclusion is important for the business, for the employees, and for continued innovation. It is recognized that diversity is not only about numbers and quotas, but about how employees that feel included, supported, and safe, are more innovative and productive. For example, according to the Harvard Business review, "Fostering an LGBT-inclusive workplace helps a company attract and retain top talent, woo and win critical consumer segments, and innovate for underserved markets." I will show examples of how company sponsored Employee Resource Groups (ERGs) such as the Women's Network and Live Open, the LGBTQ Employee Network, are contributing to increased awareness and diversity.

4:30 PM

(ICACC-DIV-005-2020) High Temperature Pressure (HTP) Boron Nitride Nanotube (BNNT): Discovery and Commercialization (Invited)

C. Park*[1]; S. Chu[2]; C. Fay[1]

1. NASA Langley Research Center, Advanced Materials and Processing Branch, USA
2. NASA Langley Research Center, National Institute of Aerospace, USA

Multifunctional materials enable revolutionary design schemes for future aerospace vehicles and structures for NASA missions. Recent studies on nanocomposite materials have revealed a great potential for both structural integrity and multifunctional capabilities, such as sensing, actuating, health monitoring, radiation shielding, energy harvesting, thermal management, and thermal protection. After the advent of carbon nanotube (CNT) in 1991, scientists predicted that boron and nitrogen, carbon's immediate neighbors on the periodic table, might also form perfect nanotubes. Even though first proposed in 1994 and synthesized in 1995, the boron nitride nanotube (BNNT) has proven to be very difficult to make until now.

The discovery and progress of a new catalyst-free method for synthesizing highly crystalline, very long, and small diameter BNNTs under a high temperature and pressure (HTP) environment have enabled new applications for multifunctional materials. The white color BNNTs offer extraordinary properties including neutron radiation shielding, piezoelectricity, thermal oxidative stability (> 800°C in air), and mechanical strength with flexibility and toughness. The inception, discovery, and technology transfer efforts on the novel BNNTs and their composites are discussed in this presentation along with their potential applications in extreme environments.

9th Global Young Investigator Forum

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

Room: Coquina Salon G

Session Chairs: Matthew Appleby, NASA Glenn Research Center; Andrew Rosenberger, Purdue University

1:30 PM

(ICACC-GYIF-001-2020) Advantageous crystalline–amorphous phase boundary for water oxidation (Invited)

S. Mhin*[1]; H. Han[3]; Y. Chung[2]; T. Song[2]

1. Korea Institute of Industrial Technology, Heat Treatment R&D Group, Republic of Korea
2. Hanyang University, Republic of Korea
3. Hongik University, Republic of Korea

Development of efficient electrocatalysts for water oxidation, which is considered as the most sluggish reaction, is a challenge to overcome. Although novel metal based catalysts such as RuO_2 are regarded as the best oxygen evolution reaction (OER) catalyst, their high-cost and comparatively lower long-term stability still limit the practical applications of water splitting. From this perspective, exploring non-novel-based catalysts with high OER performance is highly desired. Recently, we report that phase boundary between crystalline and amorphous of transition metal compounds plays an important role in boosting the OER catalytic activity. Also, increasing the density of crystalline-amorphous phase boundary further enhances the OER performance. Herein, a novel strategy to fabricate high-performance water oxidation catalysts via facile engineering of the crystalline–amorphous (c-a) interface is demonstrated. Also, the OER mechanism of the catalysts from experimental results combined with theoretical approach is discussed, which can give further insight into c-a interface engineering to improve OER performance.

2:00 PM

(ICACC-GYIF-002-2020) Microstructure-based modeling of the thermomechanical properties of ceramic matrix composites (Invited)

E. Maillet*[1]; M. Moscinski[1]; A. Bagri[1]; P. Meyer[1]; D. Dunn[1]

1. GE Research, USA

A fundamental requirement for designing mechanical components with ceramic matrix composites (CMCs) is understanding their thermomechanical properties. This is usually achieved by extensive testing and characterization. The present approach aims at developing models to quantify the effects constituent and microstructure variations have on the thermomechanical behavior of CMCs. The properties of interest include thermal-elastic, ultimate tensile strength and interlaminar strength. This presentation will discuss the generation of representative modeled microstructures, modeling frameworks, effects of key constituent properties and microstructural features, as well as validation with experimental data on known microstructures.

2:30 PM

(ICACC-GYIF-003-2020) Influence of Hydrogen and Dual Atmosphere on the Defect Structure of Oxide Scales Formed on Stainless Steels (Invited)

M. Reisert*[1]; A. Aphale[1]; Y. Tsur[2]; P. Singh[1]

1. University of Connecticut, Materials Science and Engineering, USA
2. Technion - Israel Institute of Technology, Chemical Engineering, Israel

Stainless steels have been used for the fabrication of interconnects and balance-of-plant components in solid oxide fuel cells as well as for high temperature fuel delivery system components. These materials are inexpensive and typically alloyed with a significant amount of chromium to ensure the development of a passivating chromium-based oxide scale under system operating conditions. When exposed simultaneously to a reducing hydrogen-containing atmosphere on one side and an oxidizing atmosphere on the opposing side, the passivating nature of the chromium-based scale breaks down resulting in accelerated oxidation of the base alloy. In this work, the defect structure, scale chemistry, and morphology of oxides grown on stainless steels will be reviewed to provide a better understanding of the processes responsible for accelerated metal loss via oxidation. The role of hydrogen, pertaining to oxide defect structure changes and its incorporation in the oxide scale, will be discussed.

3:20 PM

(ICACC-GYIF-004-2020) Solid Particle Erosion in Gas-Turbine Grade Ceramic Matrix Composites and Protective Coatings (Invited)

M. J. Presby*[1]

1. NASA Glenn Research Center, USA

A significant challenge exists for gas-turbine engines when operating in dust-laden environments. Solid erosive particles entrained in the gas flow can impact engine components resulting in significant material removal leading to deleterious effects in overall engine performance. Accordingly, it is important to characterize and understand the erosion mechanisms in gas-turbine grade materials as resistance to solid particle erosion is necessary for the long life and durability needed for turbine engine applications. This presentation will provide an overview of the erosion facility located at NASA Glenn Research Center, and discuss the erosion response of several gas-turbine grade ceramic matrix composites (CMCs), as well as protective coatings; thermal barrier coatings (TBCs) and environmental barrier coatings (EBCs).

3:50 PM

(ICACC-GYIF-005-2020) Nano SiC - nano TiC composites fabricated via two-step sintering

R. Malik*[1]; Y. Kim[1]

1. University of Seoul, Dept. of Materials Science & Engineering, Republic of Korea

Nanostructured ceramics are known to exhibit unique properties like high mechanical strength, improved wear resistance, superplasticity, etc. However, their full potential could not be tapped due to processing difficulties like preventing grain growth in the final stage of sintering and achieving full densification without grain growth. The two-step sintering is a promising technique to synthesize nano-nano composites that relies on the slower kinetics of grain boundary migration compared to grain boundary diffusion at lower temperatures. We have employed two-step sintering technique to fabricate nano SiC- nano TiC composites that showed interesting mechanical, thermal and electrical properties. These materials could be potential materials for thermoelectric applications. Fully densified nanostructured composites were further hot forged. The hot-forged nano SiC – nano TiC composites exhibited anisotropic microstructure and improved properties which will be presented at the conference.

4:10 PM

(ICACC-GYIF-006-2020) Alternative Transport Phases for Cold Sintering (Invited)

S. Lowum*[1]; R. Floyd[1]; J. Maria[1]

1. Pennsylvania State University, Materials Science and Engineering, USA

Reduced sintering temperatures is a topic of growing interest given current needs for materials integration, microstructure control, and dense monoliths from temperature-sensitive materials. A technique termed cold sintering enables one to densify materials at temperatures 300°C or below via the addition of a secondary mass transport phase and moderate pressures. Initially, primarily aqueous solutions of acids, salts, or bases were used as mass transport phases in this process, however recent experimentation raised questions regarding the necessity of water. This presentation will first report a set of experiments that explore and quantify the importance and need for liquid and/or bound water in cold sintering. We ultimately show that only bound water is needed in several material systems. Motivated by these results, we proceeded to conduct experiments with a new set of non-aqueous transport phases inspired by bulk crystal growth methods. We will show how these new phases expand the spectrum of chemistries that can be densified at these extreme low temperatures. This presentation will investigate the mechanisms of densification involved in cold sintering with both aqueous and non-aqueous transport phases as well as the dependence on process variables such as pressure, temperature, and transport phase quantity. Additionally, progress in cold sintering functional materials with targeted engineering properties will be demonstrated.

4:40 PM

(ICACC-GYIF-007-2020) Characterization of Sphere Impact Response of Ice-templated Hierarchical Porous Ceramics

D. Terrones*[1]; S. Akurati[2]; D. Ghosh[2]

1. Old Dominion University, Mechanical Engineering, USA
2. Old Dominion University, Mechanical and Aerospace Engineering, USA

Ice-templated porous ceramics are a class of novel materials which exhibit hierarchical microstructure and directional porosity. Due to unique microstructure and strength advantage over the conventionally fabricated ceramic foams, ice-templated ceramics are potential as lightweight structures for mechanical load-bearing applications. Our recent studies revealed significant enhancement of compressive mechanical properties in these materials under dynamic loading conditions. In this work, we investigated sphere impact response of ice-templated porous ceramics in relation to porosity, microstructure and impact velocity (50-200 m/s) to understand the correlations of mechanical performance and mechanical properties. Composition of ceramic suspensions was adjusted to modify porosity, whereas unidirectional freezing condition was varied to tailor templated microstructure. A custom-made impact tester was employed to study the sphere impact response of the materials and a high-speed camera was used to capture the projectile-ceramic interactions. Post-impact, the ice-templated materials were analyzed for mass loss, depth of penetration, and fracture. To correlate impact behavior with mechanical property, compressive strength was characterized under dynamic loading conditions employing a split-Hopkinson pressure bar (SHPB) device.

5:00 PM

(ICACC-GYIF-008-2020) ZrC-W composites prepared by reactive melt infiltration of Zr_2Cu alloy into binder-jet printed WC/ZrC preforms

R. Mudanyi*[1]; C. L. Cramer[2]; A. Elliott[2]; D. Kumar[1]

1. North Carolina A&T State University, Mechanical Engineering, USA
2. Oak Ridge National Lab, Manufacturing Demonstration Facility, USA

High-temperature carbide/metal composites exhibit excellent ultra-high temperature and hypersonic properties such as desirable flexural strength at elevated temperatures, high hardness, resistance to wear, creep and corrosion, and low density. Applications for these materials include aerospace, automotive, energy production, and defense. ZrC-W is studied for use in these environments because W and ZrC are mechanically, chemically, and thermally stable; i.e., they have similar thermal expansion coefficients, low solid solubility in each other at elevated temperatures, and high melting points, respectively. In this study, a displacement reaction of WC and Zr to ZrC-W was performed via melt infiltration of Zr_2Cu into a binder-jetted WC preform. ZrCu was used for its low melting point; however, the copper precipitates out of the composite sample, leaving ZrC-W. To increase the ZrC content in the final composite, ZrC and WC powders were mixed before printing. Microstructure, phase analysis, and mechanical properties of the cermet are analyzed.

S1: Mechanical Behavior and Performance of Ceramics & Composites

Environmental Effects and Thermo-mechanical Performance I

Room: Coquina Salon D
Session Chair: Jonathan Salem, NASA Glenn Research Center

1:30 PM

(ICACC-S1-001-2020) Progress and Plans for CMC Research at NASA Glenn in 2020 (Invited)

J. E. Grady*[1]

1. NASA Glenn Research Center, Ceramic & Polymer Composites Branch, USA

As part of NASA's Aeronautics research, Glenn Research Center has developed SiC/SiC Ceramic Matrix Composites for 2700°F turbine engine applications in the next generation of ultra-efficient aircraft. In this presentation, the development of fiber and matrix constituents and fabrication processes that enabled this advancement will be reviewed, and characterization of the resulting improvements in CMC mechanical properties and durability will be summarized. Progress toward the development and validation of models predicting the effects of the engine environment on durability of Ceramic Matrix Composites and Environmental Barrier Coatings will be summarized. Results from current collaborative research with industry and other government agencies will be reviewed. Research plans for 2020 and opportunities for future collaborations with NASA will also be summarized.

2:00 PM

(ICACC-S1-002-2020) Creep in interlaminar shear of a Hi-Nicalon™/SiC-B_4C composite at 1300°C in air and in steam

M. Ruggles-Wrenn*[1]; T. Wallis[1]

1. Air Force Institute of Technology, Aeronautics & Astronautics, USA

Creep behavior in interlaminar shear of an advanced SiC/SiC composite with a self-healing matrix was investigated at 1300°C in laboratory air and in steam. The composite was processed via chemical vapor infiltration (CVI). The composite has a self-healing oxidation inhibited matrix comprised of alternating layers of silicon carbide and boron carbide and is reinforced with laminated woven Hi-Nicalon™ fibers. Fiber preforms were coated with pyrolytic carbon fiber followed by a boron carbon overlay. The interlaminar shear properties were evaluated at 1300°C. The creep behavior was examined for interlaminar shear stresses ranging from 13 to 20 MPa in air and in steam. Primary and secondary creep regimes were observed in all tests conducted in air and in steam. Creep run-out (defined as 100 h at creep stress) was achieved at 13 MPa in air and in steam. Presence of steam had little influence on creep strain rates and creep lifetimes. However, larger creep strains were accumulated in steam than in air. The retained properties of all specimens that achieved creep run-out were characterized. Composite microstructure, as well as damage and failure mechanisms were investigated.

2:20 PM

(ICACC-S1-003-2020) Fatigue Characterization of Melt-Infiltrated Ceramic Matrix Composites in a Combustion Facility

R. Panakarajupally*[1]; M. Kannan[1]; G. N. Morscher[1]

1. University of Akron, Mechanical Engineering, USA

Ceramic matrix composites (CMCs) are evolved as the candidate materials for the future propulsion systems because of their low weight, high temperature capability and higher specific strength. Despite the recent efforts and progress in CMCs one challenge is that these materials undergo oxidation followed by surface recession in high temperature oxidizing environments. For successful implementation of CMCs in jet engines one has to characterize these materials under similar jet engine conditions. Therefore, a new combustion facility has been developed where simultaneous mechanical and combustion loading can be applied. Melt infiltrated (MI) woven SiC/SiC ceramic matrix composites are investigated in a combustion environment under tension-tension fatigue loading to evaluate the effect of combustion environment on the fatigue life. Non-destructive evaluation (NDE) techniques such as electrical resistance (ER) is used as a health monitoring technique. Specimen front and back surface temperatures are monitored by using two infrared cameras (FLIR). Similar tests are performed in isothermal static furnace for comparison. Fracture surfaces of the failed specimens were analyzed using scanning electron microscope (SEM) to identify the role of hostile environment present in the burner rig on fatigue life and damage mechanisms.

2:40 PM

(ICACC-S1-004-2020) Comparison between the thermomechanical behavior of ceramic parts obtained by plasma spray and conventional sintering

V. Badea*[1]; A. Denoirjean[1]; G. Antou[1]; T. Chotard[1]

1. University of Limoges, IRCER, France

This project is supported by Andra under the "Investments for the Future Programme" ("Investissement d'Avenir") organized in cooperation with the French National Research Agency (ANR). Additive manufacturing is a technology that has emerged in different work sectors. Currently, it is used for manufacturing of whole structural parts or for repair works. In the case of ceramics, a technique which can be implemented for rapid manufacturing, could be plasma spraying. In this study, 3D ceramic samples of different alumina-silica ratios are obtained through conventional sintering and by plasma spraying. The microstructure and chemical composition of these samples were characterized by SEM and XRD, respectively. In addition, mechanical characterizations at low and high temperature were conducted to compare their behavior during a thermal cycle.

Environmental Effects and Thermo-mechanical Performance II

Room: Coquina Salon D
Session Chair: Joseph Grady, NASA Glenn Research Center

3:20 PM

(ICACC-S1-005-2020) Static Fatigue of Hi-Nicalon™-S Fiber at Elevated Temperature in Air, Steam and Silicic-Acid-Saturated Steam

S. Robertson[1]; M. Ruggles-Wrenn*[1]; R. Hay[2]; T. Shillig[1]; R. Mitchell[1]; B. Kroeger[1]; L. Gumucio[1]

1. Air Force Institute of Technology, Aeronautics & Astronautics, USA
2. Air Force Research Laboratory, USA

A facility for testing SiC fiber tows in static fatigue at elevated temperatures in air, in steam and in steam saturated with silicic acid was developed. Static fatigue of Hi-Nicalon™-S fibers was investigated at 800-1100° C at stresses ranging from 115 to 1250 MPa in air, in steam, and in steam saturated with silicic acid. The presence of steam reduced fiber lifetimes under static fatigue. Fiber lifetimes were much shorter in unsaturated steam than in $Si(OH)_4(g)$-saturated steam. The Monkman-Grant (MG) relationship was applied to the stress-rupture experimental results. The test environment had little influence on the MG parameters. The MG relationship was demonstrated to successfully predict creep lifetimes in air, steam and steam saturated with silicic acid.

3:40 PM

(ICACC-S1-006-2020) New perspectives on boria-accelerated oxidation of silicon carbide

B. McFarland[1]; E. Opila*[1]

1. University of Virginia, USA

Oxidation of silicon carbide (SiC)-based ceramic composites with a boron nitride fiber/matrix interphase has resulted in rapid degradation and consumption of SiC fibers in some cases. It is believed that boria formed from the oxidation of boron nitride accelerates SiC oxidation by many orders of magnitude, yet the mechanism is poorly understood. In this study, SiC coupons were coated with sol-gel derived borosilicate coatings of varying composition (0-38, 92-94 mol% boria). The coated coupons were exposed to oxygen or argon environments at 800 or 1200C for 100 hours. Weight change, oxide thickness, and oxide composition were characterized by thermogravimetric analysis, scanning electron microscopy, and inductively coupled plasma optical emission spectroscopy, respectively. It was found that boria-rich (92-94% boria) coatings significantly accelerated SiC oxidation rates only at 800C in oxygen environments. Surface etching of the SiC surface was observed when the oxide was removed in a two-step water and acid dissolution. This etching was not observed for any other coating/exposure conditions. It is proposed that high boria contents, oxidizing conditions, and minimal boria volatility are required to promote accelerated SiC oxidation through an oxidation/dissolution mechanism at the oxide/carbide interface. Implications for SiC-based composites will be discussed.

4:00 PM

(ICACC-S1-007-2020) Coupling Chemistry and Mechanics to Model Oxidation Embrittlement of SiC/BN/SiC Ceramic Matrix Composites

V. Collier*[1]; M. Begley[1]; W. Xu[2]; F. W. Zok[2]; R. McMeeking[2]

1. University of California, Santa Barbara, Materials, USA
2. University of California, Santa Barbara, USA

The oxidation embrittlement of SiC/BN/SiC ceramics matrix composites at intermediate temperatures significantly impacts component durability, but existing models do not satisfactory describe the impacts of environment and specimen geometry on the observed embrittlement. Therefore, a model has been developed to investigate how chemistry impacts component mechanics. Three coupled phenomena are considered, namely: (i) diffusion of oxidative species down a pre-existing matrix crack; (ii) volatilization of the BN coating upon exposure to oxidants; and (iii) oxidation of the SiC fibers and matrix. Scaling arguments identify key species and processes e.g. the scale growth is dominated by water vapor transport and the dominate volatilization product for the temperature range of interest is $H_3B_3O_6$. Furthermore, the coupling between transport-oxidation and mechanics is explored by calculating fiber bridging stresses. The model accounts for the effects of BN recession by adjusting the shear sliding length of classical fiber bridging models, thereby informing how chemistry changes fiber stress distributions in CMCs. The results highlight key sensitivities to environmental conditions and advance efforts to model time-to-rupture as a function of environment and temperature.

4:20 PM

(ICACC-S1-008-2020) Effect of oxygen content on oxidation resistance of SiC fibers

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2. IHI ASIA PACIFIC(Thailand)Co., Ltd., Thailand

Ceramic Matrix Composites (CMCs) are a new alternative material to Ni-based super alloys for aero engines, and are being applied in civil aircrafts. In recent years, to expand the range of applications of CMCs, development efforts have been made to increase the mechanical properties of CMCs, and many reports indicate that the oxidation resistance of reinforced SiC fibers affects the mechanical properties of CMCs. On the other hand, only few studies have been reported on the effect of oxygen content on the oxidation resistance of SiC fibers. In this study, in order to clarify the effect of oxygen content on the oxidation resistance of SiC fibers, SiC fibers with various oxygen contents were exposed to different heat treatment conditions. During the heat treatment, the microstructure of the fibers was observed and the oxidation rates were determined by observing the oxide thickness.

4:40 PM

(ICACC-S1-009-2020) Oxidation of SiC fibers and bulk SiC at intermediate temperatures

V. Christensen*[1]; F. W. Zok[1]

1. University of California, Santa Barbara, Materials, USA

The nature and rates of internal oxidation processes in ceramic matrix composites remain poorly understood. To address some of the outstanding issues, we performed oxidation experiments on both bare and BN-coated SiC fibers (Tyranno ZMI and Hi-Nicalon-S) in dry air and water vapor at temperatures ranging from 800-1100°C. In some cases, BN-coated fibers are sandwiched between bulk SiC plates during oxidation. Scale composition and thickness on the fibers and on bulk SiC are characterized using scanning electron microscopy (SEM) and x-ray photoelectron spectroscopy (XPS). Such tests provide a means for elucidating the role of the BN in oxidation processes involving pure SiC as well as amorphous, non-stoichiometric fibers (Tyranno ZMI) and crystalline, nearly-stoichiometric fibers (Hi-Nicalon-S).

5:00 PM

(ICACC-S1-010-2020) High Temperature Mechanical Properties of Monolithic Tungsten Carbide

B. Currie*[1]; L. J. Vandeperre[2]; S. A. Humphry-Baker[2]

1. Imperial College, CASC, United Kingdom
2. Imperial College London, Materials, United Kingdom

High temperature deformation kinetics of monolithic tungsten carbide (WC) have been studied, observing and quantifying power-law creep rates for the first time. WC has long been used in cobalt-based cemented carbides. Whilst its properties in cemented carbide form are well studied, monolithic WC has remained less so because of challenges in consolidating WC without a liquid phase. Previous works have highlighted inconsistencies in the reported high temperature hardness and creep data was found to be limited to 1200 – 1500°C, with all data appearing to be representative of diffusional creep. This paper reports firstly: mutual indentation testing, expanding hardness data of polycrystalline WC from < 900°C up to 2000°C; and secondly, compressive creep testing within the temperature range of 1300 - 1900°C, i.e. higher than previously studied. We observe a transition from diffusional flow creep to power-law creep between 1500 - 1600°C. The latter mechanism is yet to be reported. The transition we observe in hexagonal WC is compared to the literature concerning other transition metal carbides, which usually have cubic structures.

5:20 PM

(ICACC-S1-011-2020) How to improve the potential of high-temperature thermal energy storage technologies through ceramic matrix composites

Y. Shi*[1]; F. Kessel[1]; D. Koch[1]; V. Stahl[2]; T. Lanz[2]; P. Vetter[2]; W. Kraft[2]; W. Ding[3]

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2. DLR - German Aerospace Center, Institute of Vehicle Concepts, Germany
3. DLR - German Aerospace Center, Institute of Engineering Thermodynamics, Germany

The significant increase of world energy consumption raises the demand for imperatively clean energy resources. Since energy production methods have been greatly improved, one of the biggest challenges remains the development of high-performance energy storage systems. In this work, two newly developed thermal storage concepts with different storage materials, metallic phase change materials (mPCM) and molten chloride salts are investigated. Due to their unavoidable corrosion aggressiveness at high temperatures (HT), Ceramic Matrix Composites (CMCs) are used as housing material as they are well known for their high mechanical properties and damage tolerance at HT. The compatibility between storage materials and Liquid Silicon Infiltration based carbon fiber reinforced silicon carbide (C/C-SiC) composite has been firstly investigated via contact angle measurements. Then, C/C-SiC coupons were immersed separately in molten chloride salt and liquid mPCM at 700°C for several hundred hours. The results reveal that the C/C-SiC composite maintains its material properties after exposure in the both strongly corrosive storage materials. It is shown that the applicability of different thermal energy storage technologies at HT can be improved through C/C-SiC composite due to its excellent corrosion resistance and favorable material properties.

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

Advanced Coatings for Extreme Environments

Room: Ponce de Leon

Session Chairs: Peter Mechnich, DLR - German Aerospace Center; Eugene Medvedovski, Consultant

1:30 PM

(ICACC-S2-001-2020) Linking the Microstructural Evolution and Oxidization Behavior of Mixed Metal Diboride Coatings Deposited by Chemical Vapor Deposition

C. Romnes[2]; K. Canova[1]; Z. Tucker[1]; Z. Zhang[1]; J. R. Abelson[1]; J. A. Krogstad*[1]

1. University of Illinois at Urbana-Champaign, Materials Science and Engineering, USA
2. University of Illinois at Urbana-Champaign, Department of Nuclear, Plasma and Radiological Engineering, USA

Transition metal diboride protective coatings are of interest in automotive, aerospace, and manufacturing industries due to the potential to improve hardness, chemical inertness and increase wear resistance. Currently, use of diboride coatings is limited because transition metal diborides have poor oxidation resistance at intermediate temperatures. We report an innovative, low temperature, chemical vapor deposition process that results in a diboride film with improved oxidation resistance. These coatings consist of ternary alloy diborides in which the second metal is an effective oxide former. During annealing, a dense and protective surface oxide evolves to afford oxidation resistance, while the bulk transforms into a nanocrystalline phase. To better understand the annealing behavior, these coatings were investigated at various time

stages during isothermal annealing using transmission electron microscopy (TEM), scanning TEM, and energy dispersive x-ray spectroscopy. We report key insights on the relationship between microstructural evolution and oxidation resistance

1:50 PM

(ICACC-S2-002-2020) Relationship Between Bonding and Performance of Polymer-Derived Ceramic Thermal/ Environmental Barrier Coatings

K. McGarrity*[1]; H. Shulman[1]; P. Tumurugoti[2]; K. Ning[1]

1. Alfred University, Materials Science and Engineering, USA
2. Alfred University, USA

While their excellent mechanical strength and heightened thermal stability make covalent ceramics the subjects of much investigation for the increasingly extreme operating conditions required by the energy, automotive, and aerospace industries, oxidation, wear, gradual thermal decomposition, and machinability have limited their feasibility. Coatings for these prospective ceramic components which match or enhance their mechanical, thermal, and chemical resistance are therefore desirable. The present work focuses on several polymer-derived ceramics with varying structures in the Si-B-C-N-O system, as ceramics in this family have proven stable up to 2,000 C, and are capable of complex geometries and near-net-shape forming by virtue of their polymer-derived nature. Here the authors describe an empirical relationship between polymer precursor and resultant ceramic structure and chemistry, and further, thermomechanical properties observed in the resultant ceramic. The known decomposition mechanisms for these materials are used as a guide in the design of ceramic coatings of high density, thermal stability, and oxidation resistance. Hardness and practical scratch adhesion data are presented alongside the thermal and chemical.

2:10 PM

(ICACC-S2-003-2020) Influence of doping on the improvement of thermo-mechanical and chemical properties of a stabilised hafnia for an ultra-high temperature thermal and environmental barrier

L. Sévin*[1]; A. Julian-Jankowiak[1]; J. Justin[1]; V. Razafindramanana[1]; F. Mauvy[2]; F. Rebillat[3]

1. ONERA, DMAS, France
2. ICMCB-CNRS, France
3. University Bordeaux, Laboratory of thermostuctural composites, France

CNES has engaged the development of a new green propellant which induces the study of new combustion chamber materials due to harsh operating conditions. The design of such materials induced the development of a 3000 K oxides resistant ceramic as EBC/TBC. Thanks to its refractory and good thermal properties, the cubic-stabilised HfO_2 seems to be one of the most promising candidates. Thus, any phase transformation has to be avoided through doping with rare earth oxides[3]. However, doping generates oxygen vacancies leading to physicochemical properties variations. Thus, the influence of various natures (Gd_2O_3, Dy_2O_3, Y_2O_3, Er_2O_3, Yb_2O_3 and Lu_2O_3), rates (12-33 mol. %) and co-doping associations of rare earth oxides were investigated as a function of Thermal Expansion Coefficient (373-1773 K), ionic conductivity (860-1523 K) and finally Young's modulus. HfO_2 with the highest Y_2O_3 (YSH) doping rate, compared to the commonly used 8YSZ, exhibits decreasing of 3.10^{-6}/K for its TEC and 3 decades for its ionic conductivity. Furthermore, an improvement in Young's modulus is obtained. Finally, the co-doping approach affords to make positive synergistic effects to get better properties in comparison with the binary system which could be explained by the induced lattice distortion and the binding anharmonicity.

2:30 PM

(ICACC-S2-004-2020) Growth Behaviour of Thermally Grown Oxides Alumina and Silica in High-Temperature Protective Coatings

K. Chen*[1]

1. National Research Council Canada, Aerospace Research Centre, Canada

The atomistic transport theory combing ab initio thermodynamics (or density functional theory -DFT) was used to study the growth behaviour of alumina and silica oxide scales, where the former oxide scale grows in typical oxidation-resistant coatings and/or thermal barrier coatings, while the later oxide scale grows in environmental barrier coatings. The fundamental Wagner theory was applied to studying the growth kinetics of these two oxide scales. The oxygen potential gradient across the scales was evaluated using the combined ab initio thermodynamics, and the permeation of oxygen across the oxide scales was calculated in terms of diffusivities. The characteristics of the growth kinetics of these two oxide scales were analyzed and compared. The effect of selected reactive elements (REs) doping on oxygen permeability of these oxide scales was theoretically examined, and the physics behind the RE doping effect was explored. The effect of water vapour at high temperatures on the growth kinetics of these two oxide scales was also investigated.

3:10 PM

(ICACC-S2-005-2020) Erosion Studies of the Iron Boride Coatings for Protection of Tubing Components in Oil Production, Mineral Processing and Engineering Applications (Invited)

E. Medvedovski*[1]; M. Antonov[2]

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2. Tallinn University of Technology, Mechanical and Industrial Engineering, Estonia

Heavy oil production, oil sand and mineral processing require protection of steel components against sand erosion and erosion-corrosion. Boronized coatings on carbon steels obtained through thermal diffusion process were evaluated in the dry sand and slurry erosion conditions at low impingement angles commonly occurred in industrial applications. The specially designed system allowed testing either multiple sets of samples or full-size tubular sections with diameters of 180-220 mm. The boronized coatings consisted of dual iron boride layers FeB and Fe_2B with a total thickness of ~200 μm demonstrated significantly higher erosion resistance over carbon steel commonly used in industry. High performance of the boride coatings is defined by their high hardness, dual protective layer with a well-consolidated structure and diffusion related bonding with steels. The erosion wear mechanism is discussed based on examination of coating's structure and composition and on analysis of testing conditions. The components and tubing with inner or inner and outer protective iron boride coatings can be successfully employed in downhole oil production conditions, mineral processing and various engineering applications.

3:40 PM

(ICACC-S2-006-2020) Development of Materials of the Al_2O_3-Cr_2O_3-TiO_2 System for Surface Technologies

L. Berger*[1]; S. Conze[1]

1. Fraunhofer IKTS, Germany

For the manufacturing of ceramic coatings, in particular by thermal spraying, materials of the system Al_2O_3-Cr_2O_3-TiO_2 have an outstanding practical importance. However, the current use is limited to the single oxides and binary compositions. Coatings of each of the single oxides show certain drawbacks due to the specific material behavior in the spray process, which can be attenuated by the use of binary compositions. There are indications in the literature describing that the use of ternary compositions can result in

further improved properties. However, the knowledge-based development of corresponding thermal spray feedstock powders from this ternary system is strongly limited by the surprisingly much limited information about the phase equilibria, the synthesis conditions and stability of individual phases. Thus, with this work we follow a concept, to create some basic knowledge from sintered ceramic bodies in advance before feedstock powder preparation. By this reason, synthesis via SPS, phase composition, microstructure as well as hardness and specific electrical resistivity were investigated. Work was focused on alumina additions on Andersson-type phases of Cr_2O_3-TiO_2 and the influence of titania on the formation of the Al_2O_3-Cr_2O_3 solid solutions.

4:00 PM

(ICACC-S2-007-2020) Bioactive coatings obtained by cold spray and atmospheric plasma spraying

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Hydroxyapatite (HA)-based biomaterials has been widely used because it possesses almost the closest similarity in chemical composition with natural bone tissue. However, the poor mechanical properties such as low fracture toughness and low modulus of elasticity of HA it is not suitable for high load bearing implant. Plasma spraying (such as APS, VPS) still remains the most commonly used technique for HA coating on a Ti or Ti alloy substrate in the fabrication of artificial joint replacements. Cold-spray overcomes the shortcomings of thermal spraying, which involves melting and solidification of the splats. By this way one can be avoid phase transformation into other calcium phosphate phases such as α- or β- tricalcium phosphate, tetracalcium phosphate (TTCP) or calcium oxide (CaO) and the crystallinity of HA may also be lowered due to rapid solidification, which can negatively affect the mechanical properties The goal of the study presented here was to realize a morphological, microstructural and mechanical comparative study between atmospheric plasma spraying and cold spray hydroxyapatite coating on titanium alloy substrates. The coatings were investigated and characterized by Scanning Electron Microscopy (SEM), X-ray Diffraction (XRD), and pull off adhesion test.

4:20 PM

(ICACC-S2-008-2020) Advanced chemical vapor deposition routes for the production of SiO_2 barrier films at moderate temperature

K. Topka*[1]; S. Ponton[1]; H. Vergnes[1]; D. Samelor[2]; D. Sadowski[2]; V. Turq[3]; R. Laloo[3]; C. Genevois[4]; H. Lecoq[4]; C. Vahlas[2]; B. Caussat[1]

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2. CIRIMAT, Université de Toulouse, CNRS, France
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4. CNRS CEMHTI UPR3079, Université d'Orléans, F-45071, France

Amorphous SiO_2 films are used, among others, as barrier coatings for storage applications. They are frequently obtained from tetraethyl orthosilicate ($Si(OC_2H_5)_4$, TEOS) based chemical vapor deposition (CVD) processes, allowing for the deposition on the surface of complex, 3D parts. In the case of surface treatment of thermally sensitive substrates, such as plastics and glass, tuning of alternative, highly reactive chemical routes is required. In this presentation, we present results on the combined computational and experimental investigation of TEOS-based CVD processes involving O_2 and O_2/O_3 mixtures, and operating at atmospheric pressure in the 360-570°C temperature range. The TEOS-O_2-O_3 system proves to be highly reactive, yet at higher temperatures (>500°C) the addition of hexamethyldisilazane ($HN[Si(CH_3)_3]_2$, HMDS) yields threefold increase of growth rates. Films are systematically amorphous and exhibit a smooth surface morphology with low roughness. A cross-linked investigation of the structure and composition, involving FT-IR, Ellipsometry, TEM, IBA and nanoindentation, together with wet corrosion and wetting tests is underway. First results show that the hardness of the TEOS-O_2-O_3 films is ca. 8 GPa, to be compared with 3 GPa for those processed in the presence of HMDS, the latter being richer in hydrogen.

4:40 PM

(ICACC-S2-009-2020) Development of a kinetic model for the moderate temperature chemical vapor deposition of SiO_2 films from TEOS, O_2 and O_3

K. Topka*[1]; G. Chliavoras[2]; H. Vergnes[1]; D. Samelor[3]; D. Sadowski[3]; C. Vahlas[3]; B. Caussat[1]

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3. CIRIMAT, Université de Toulouse, CNRS, France

The use of SiO_2 films as barrier coatings for storage applications often requires deposition on the surface of complex 3D, thermally sensitive parts, at low to moderate temperatures. In this context, we make use of modelling and simulation of chemical vapor deposition of SiO_2 films from TEOS-O_2-O_3 to replace time-consuming trial-and-error screening aimed at process optimization. We perform deposition experiments to establish the thickness profiles of SiO_2 films and attempt to replicate them through elaboration of an innovative, apparent kinetic model and computational fluid dynamics calculations. Gas-phase and surface reactions are fitted in ANSYS Fluent simulation environment to achieve a good representation of deposition profiles and patterns. Updated kinetics calculations suggest that deposition of SiO_2 from this chemical system happens from reactions activated at specific temperatures: At low temperatures (<250°C) deposition is attributed mainly to the reaction between TEOS and O_3. At intermediate temperatures (~200-300°C) SiO_2 production is a result of the reaction between an intermediate, silicon-containing species and O_3, while at higher temperatures said intermediate reacts with O_2 instead. The proposed model is utilized for the computational investigation of optimal process conditions that yield uniform deposition profiles in complex 3D geometries.

5:00 PM

(ICACC-S2-010-2020) Development of the First SCO Molecular Ceramic by Cool-SPS

L. El Khoury*[1]; N. Daro[1]; M. Marchivie[1]; M. Josse[1]

1. ICMCB-CNRS, France

In recent decades, the development of new molecular materials exhibiting the Spin CrossOver phenomena (SCO) has aroused a growing interest. These compounds have the capacity to change their electronic state under external stimulus with important consequences on their structural and magnetic properties. The shaping of these molecular materials, remain almost unexplored, due to their thermodynamic fragility, Very recently, thanks to the development of Cool-SPS the first "molecular ceramics" were obtained at the ICMCB. The current work aims thus to develop new "molecular ceramics" using this approach but from functional SCO complexes. Beyond the evident interest of this new possibility of shaping, relationships between the microstructures obtained, their physical properties, and their switching behaviors will constitute a strong benefit to the understanding of the SCO phenomena from a fundamental viewpoint. In this context, the SCO complex $[Fe(Htrz)_2(trz)](BF_4)$ was synthesized using two different routes leading to two polymorphs. Several "molecular ceramics" were then produced by Cool-SPS using these starting powders. First results reveal a strong influence of sintering on SCO switching behaviour whose thermal amplitude was not expected. The optimal sintering conditions will be discussed and the influence of the sintering parameters (temperature, pressure, etc.) on the structural and morphological properties will be studied.

5:20 PM

(ICACC-S2-011-2020) Multifunctional ceramic layers of hexagonal boron nitride and graphite

C. S. Chari*[1]; K. Faber[1]

1. California Institute of Technology, Materials Science, USA

Hexagonal boron nitride (h-BN) and graphite have similar structures, with comparable lattice parameters and coefficients of thermal expansion, but vastly different electrical and oxidative properties. For example, h-BN is commonly used for its insulating properties and resistance to oxidation at temperatures up to 900 °C. In comparison, graphite is used for its conductive properties and superior processability, yet it is susceptible to oxidation at temperatures close to 600 °C. Despite their essential differences, it is possible to couple h-BN and graphite to form functional systems that can combine the distinct properties of each material. Such a layered system can be examined not only as an insulator-conductor pair, but also as an oxidation-resistant coated substrate, allowing for its multifunctionality. Previous studies have shown that it is possible to grow h-BN on the surface of graphite through the carbothermal reduction of boric acid in nitrogen, producing a coating of h-BN. This results in a layered system that is oxidation-resistant and insulating on the surface due to the h-BN coating, and compliant as well as conductive in the substrate due to the structural body of graphite. Here we will discuss the synthesis and characterization of this layered material, focusing particularly on the interface of the h-BN/graphite system to assess the chemical and mechanical adhesion of the layers.

S3: 17th International Symposium on Solid Oxide Cells (SOC): Materials, Science and Technology

Progress in SOFC and SOEC Technology

Room: Crystal

Session Chair: Narottam Bansal, NASA Glenn Research Center

1:30 PM

(ICACC-S3-001-2020) Overview of U.S. Department of Energy Office of Fossil Energy's Solid Oxide Fuel Cell Program (Invited)

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

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

The mission of the U.S. Department of Energy (DOE) Office of Fossil Energy National Energy Technology Laboratory (NETL) Solid Oxide Fuel Cell (SOFC) program is to enable the efficient generation of low-cost electricity for natural gas-fueled SOFC distributed generation (DG) systems and modular, coal-fueled systems in the near-term and coal or natural gas-fueled utility-scale systems with carbon capture and sequestration (CCS) in the long-term. R&D in the SOFC program is focused on maintaining cell development and core technology research to increase the reliability, robustness, and durability of cell, stack, and system technology; and providing the technology base to permit cost-competitive distributed generation applications. The program is a unique balance of strategically oriented research and development activities spanning fundamental research to prototype testing. This paper summarizes the status of the SOFC program's cell and core technologies R&D and integrated prototype systems tests.

2:00 PM

(ICACC-S3-002-2020) Development of Solid Oxide Fuel Cells in China (Invited)

M. Han*[1]

1. Tsinghua University, State Key Laboratory of Power Systems, Department of Energy and Power Engineering, China

Solid oxide fuel cell (SOFC) develops very fast in the world recently. This work details the growth and evolution of SOFC technology from fundamental science to engineering in China, especially in Tsinghua group. Supported by the National "973" Project of MOST (Ministry of Science and Technology of China) from 2011 to 2016, fundamental researches have been carried out to clear the scientific issues, including the construction of SOFC structure and its electrochemical behavior, the coupled mechanism of multi-physics and multi-scale, as well as the energy management and analysis of SOFC system. Now, the degradation mechanism and lifetime extending of SOFC, supported by National Key R&D Project of Most from 2019 to 2023 is engaging. Meanwhile, the SOFC industry chain from powder to power has been developed to solve the engineering issues. The first 200,000 SOFC production line has been put into service in Xuzhou, Jiangsu Province since August, 2019. Based on the scale-up cells and stacks technology, a coal-based fuel cell integrated demonstration system has been built in a Shanxi Province in 2018. Another project aims to "Coal Gasification Power System with Near Zero CO2 Emission", which was supported by MOST from 2017 to 2021. All the works will support the real application of the SOFC technology in China, providing contributions to the future clean and high efficiency power plants using coal or other fuels.

2:30 PM

(ICACC-S3-003-2020) SOC Progress in Germany and EU (Invited)

M. Kusnezoff*[1]; A. Michaelis[1]

1. Fraunhofer IKTS, Germany

New legislation supporting the reduction of GHG emissions in Germany and in increasing number of EU countries stimulates the development of fuel cells and electrolyzers as important components of future cleaner energy system. Fuel cells and electrolyzers in general have achieved the customer acceptance level needed for product commercialization. Especially SOFC with high technology readiness level is entering first mass markets and is struggling with production scalability, cost and robustness / durability issues. Germany and European Commission strongly supports SOC development seeing in this technology an option for high effciency utilization of energy coming from fossile fuels and at the same time as technology able to utilize excess heat, CO_2 and provide highly energy efficient path to climate neutral hydrogen, ammonia and hydrocarbons production. Especially such companies like sunfire, Bosch and SolidPower as well as gas companies like Air Liquide are expanding their activities in fuel cell and/or electrolysis area. Although low temperature fuel cells and electrolyzers dominate the markets today, SOFC / SOEC is considered as most promising option for hydrocarbons utilization and chemicals synthesis or carbon capture and utilization correspondingly.

3:00 PM

(ICACC-S3-004-2020) High temperature proton- and oxide-conducting fuel cells and electrolyzers in ARPA-E portfolio (Invited)

G. L. Soloveichik*[1]

1. Advanced Research Projects Agency - Energy (ARPA-E), USA

Development of high temperature proton- and oxide-conducting fuel cells and electrolyzers for fast growing zero-carbon transportation and grid-scale energy storage is supported in part by the Advanced Research Projects Agency (ARPA-E), which funds high

risk, high reward transformational research to reduce energy related emissions, reduce energy imports, improve energy efficiency, and ensure US technological lead in advanced energy technologies. Reliable Electricity Based on ELectrochemical Systems (REBELS) program pursued alternate fuel cell materials and operating conditions for natural gas fueled fuel cells that could enable system cost reductions. The Renewable Electricity to Fuels through Utilization of Energy-dense Liquids (REFUEL) program funds the development of transformational technologies to reduce the barriers to widespread adoption of intermittent renewable energy sources by enabling the conversion of energy from these sources, water and air to energy-dense carbon neutral liquid fuels using high temperature electrolyzers and their conversion back to electricity using fuel cells. Development of liquid fueled and regenerative fuel cells for energy storage is also being funded via OPEN program. Possible future ARPA-E program targeting the use of ceramic fuel cells fed by CNLFs for hybrid transportation (in range extenders, APUs, etc.) will be also discussed.

SOC Stacks and Their Integration in the Systems

Room: Crystal
Session Chair: Patcharin Burke, Pennsylvania State University

4:00 PM

(ICACC-S3-005-2020) Demonstration of an industrial-size biogas-fed SOFC system: Experience, degradations, economy (Invited)

M. Santarelli*[1]; M. Gandiglio[2]; T. Hakala[2]; M. Rautanen[3]; M. Acri[4]; A. Hawkes[5]

1. Politecnico di Torino, Energy, Italy
2. CONVION Oy, Finland
3. VTT Technical Research Centre of Finland, Finland
4. SMAT spa, Italy
5. Imperial College, United Kingdom

The EU-funded DEMOSOFC project (www.demosofc.eu), still running, has spent the last 3 years in demonstrating the technical and economic feasibility of operating an industrial-size (174 kW_e) SOFC system in a wastewater treatment plant. The fuel for the SOFC modules is biogas. A heat-recovery loop allows to recover useful thermal energy from the hot SOFC exhaust gases (90-100 kW_{th}), transferred through a water loop to the sludge. The integrated biogas-SOFC plant includes three main units: 1) the biogas clean-up and compression section; 2) the three SOFC power modules, and 3) the heat recovery loop. Main advantages of the proposed layout is the net electric efficiency of the SOFC, which is in the range 50-55%, and the near-zero emissions in terms of NO_x, SO_x, VOC, PM. The talk will present the results from the first 3 years of operation of the system, by showing results in terms of: 1) Operation: integration with the existing plant and loads coverage, plant electrical production and thermal recovery, performance of the biogas purification unit, day-by-day experiences of management and maintenance. 2) Degradation: the data analysis allows a first evaluations of criticalities of biogas-fed SOFC in industrial plants, with insight in observed degradation issues. 3) Economy: economic and business perspective are presented for this plant concept.

4:30 PM

(ICACC-S3-006-2020) SOFC Stack and System Development for Automotive Use

T. Shiomi*[1]; M. Kamijo[1]

1. Nissan Motor Co., Ltd., EV System Laboratory, Japan

SOFC is generally considered for residential and industrial power supply, but in recent years the research and the development of SOFC powered application has been spreading to power supply for portable device, datacenter, truck APU and so on. Under these circumstances, Nissan has been researching SOFC-powered system more than fifteen years and demonstrated SOFC-powered range extender electric vehicle in 2016. This SOFC-powered vehicle gives the cruising range similar to gasoline-powered cars as well as electric vehicle's distinct features of silent drive. After the demonstration in 2016, Nissan has been developing SOFC-powered range extender system to apply various types of automotive use. The requirement of SOFC-powered system for automobile use is unique compared with that of stationary use because operating environment for automotive application requires high power output, rapid start-up and a large number of start and stop cycles with thermal and redox cycling, etc. Among many technical challenges from system level to cell level, the compactness of the entire SOFC system is one of key aspects to be able to fit in limited vehicle cocoon. Therefore, both the improvement of cell/stack performance by changing material/process and proper temperature control at stack/system level are essential. In the presentation, their challenges, progress and future expectations are discussed with SOFC requirement for automotive use.

5:00 PM

(ICACC-S3-007-2020) Development and operation of Saint-Gobain's all-ceramic Solid Oxide Fuel Cell Stack (Invited)

J. Pietras*[1]; Y. Takagi[1]; B. Feldman[1]; S. Megel[2]; J. Schnetter[2]; S. Hielscher[2]; G. Ganzer[2]; M. Kusnezoff[2]

1. Saint-Gobain, USA
2. Fraunhofer IKTS, Germany

Energy generation through solid oxide fuel cells (SOFCs) is a long-term strategic project for Saint-Gobain, a global leader in ceramic materials and components. High electrical efficiency and operational durability are two key aspects for successful commercialization of the technology. Our unique all-ceramic SOFC stacks are designed to meet the reliability and cost targets for distributed energy applications in the small to medium power scale. Significant operational stability improvement and manufacturing cost reduction is achieved through extensive engineering of ultra-thin ceramic interconnects, simplified monolithic stack-supported design and a multi-cell co-firing process. Improvements in performance, stability and size of Saint-Gobain's stack/hot box designs will be presented.

5:30 PM

(ICACC-S3-008-2020) Power to fuels: A giga-market channel for renewable energy

J. Hartvigsen[1]; S. Elangovan*[1]; S. Nigarura[2]

1. OXEON ENERGY LLC, USA
2. Global Tungsten Powders, USA

Decades of investment in solid oxide fuel cell development have been leveraged by solid oxide electrolysis (SOXE) developers. Thermodynamics favor electrolysis at high temperatures. A SOXE can produce ~50% more hydrogen per MW-h than water electrolysis. SOXE devices can also co-electrolyze steam and CO_2 to produce synthesis gas for conversion to sustainable, non-fossil, drop-in fit for purpose liquid fuels. The electric demand to completely displace fossil fuels is several times that of the conventional electric utility grid market size. Power to fuels plants create an outlet for enormous blocks of low-cost clean power, bridging renewable energy into the transportation and chemicals sectors. Climate change is an environmental, economic, and security threat. OxEon Energy's mission is to implement the vision of using renewable energy to produce synthetic fuels. OxEon, with Plansee Group teams in Reutte Austria and Towanda PA (Global Tungsten Powders) have collaborated to create SOXE devices for NASA, such as the MOXIE (Mars Oxygen ISRU Experiment) that will fly on the Mars rover launch in mid-2020. As the MOXIE flight hardware was nearing delivery, the OxEon and Plansee-GTP teams collaborated to apply lessons learned from MOXIE and scale it for both manned Mars mission and terrestrial commercial power to fuels applications. The case for power to fuels and the progress toward achieving this objective will be presented.

S4: Armor Ceramics - Challenges and New Developments

Terminal Ballistics I & II

Room: St. Johns
Session Chairs: Jerry LaSalvia, U.S. Army Research Laboratory; Brian Schuster, U.S. Army Research Laboratory

1:30 PM

(ICACC-S4-001-2020) Ballistic Testing and Characterization of Ceramic Armor Materials (Invited)

E. Strassburger*[1]

1. Fraunhofer Institute, High-Speed Dynamics, Germany

The challenge to improve the efficiency of ceramic armor has always been approached from two sides. Extensive ballistic testing of promising material combinations has been the traditional approach. The development and use of diagnostic tools like high-speed cameras, flash X-ray techniques and interferometric methods in combination with pressure and strain measurements has led to new insights into the processes during the penetration of projectiles into ceramic materials. These results have significantly improved the efficiency of this approach. On the other hand, methods for dynamic material characterization have been developed and refined to understand the physics of the material behavior under the extreme conditions of a ballistic event. For brittle materials, ballistic impact always generates fracture, which propagates at high velocity in front of the penetrating projectile. Therefore, the properties of the damaged material are also decisive for its penetration resistance. This paper reviews the investigations on lightweight ceramic armor at EMI with a focus on damage in ceramic and glass. A new method will be presented, which is designed to improve the understanding of the influence of pre-damage on strength in glass. The procedure is based on three steps: defined initiation of damage, quantification pre-damage and strength measurement. First results with soda-lime glass will be discussed.

2:00 PM

(ICACC-S4-002-2020) Ballistic evaluation of boron carbide ceramic tiles (Invited)

G. Toussaint*[1]; B. Koch[2]; J. D. Hogan[2]

1. Defence Research and Development Canada, Canada
2. University of Alberta, Edmonton, Mechanical Engineering, Canada

The continuing development of novel weapons technologies requires improving the ballistic performance of personal protection equipment as well as vehicle add-on armour. For this purpose, advancements of amour ceramics play an important role and over the past few years, boron carbide has gained interest in defence applications due to its light density and high hardness compared to traditional ceramics. In this study, fracture and fragmentation behavior of boron carbide ceramics from different manufacturers were investigated. Characterization of the ceramics as well as ballistic tests were conducted. The ballistic performance of boron carbide tiles was evaluated using the depth of penetration test method using 7.62 mm armour piercing (AP M2) projectile impacting boron ceramics bonded to a polycarbonate 20% glass filled backing material. The results demonstrated that for the generic boron carbide ceramic, quality of the ceramic is of prime importance when selecting a ceramic for armour systems. Moreover, it demonstrates the importance to characterize the ceramics to determine the properties to be used in the constitutive models of the finite element models.

2:30 PM

(ICACC-S4-003-2020) Incipient Fracture of Ceramics Under Impact (Invited)

B. Schuster*[1]; A. Tonge[1]; P. Jannotti[1]; T. Scharf[2]; N. Lorenzo[3]

1. US Army Research Laboratory, FCDD-RLW-B, USA
2. University of North Texas, Department of Material Science and Engineering, USA
3. US Army Research Laboratory, SURVICE Engineering, USA

Boron carbide is a candidate in many armor applications because it has an exceptional specific strength (hardness approaching 30 GPa and low density of ~2.5 g/cc). In practice, wider use of this material has been limited because it is well-known to show a loss of shear strength at pressures exceeding 20 GPa. Here, the large-scale ballistic response was investigated using the High voltage In-situ Diagnostic Radiography Apparatus (HIDRA) at ARL. The penetrator-target interactions and activated damage modes were observed using radiography, high speed imaging and velocimetry. The small-scale impact and shock response was investigated using propagation phase contrast imaging (PCI) and X-ray diffraction under shock loading at the Dynamic Compression Sector (DCS, 35-ID-E) at the Advanced Photon Source. At the lowest peak stresses investigated, experiments at both length scales show the penetrator undergoes dwell and the target response is consistent with cone crack formation at the impact site. At higher striking velocities there is a distinct transition to massive fragmentation leading to the onset of penetration. Imaging and diffraction suggest that the loss of shear strength at high pressures can be attributed to fine-scale brittle fracture above the Hugoniot Elastic Limit (HEL). Finally, we will discuss how these small-scale impact experiments are used for validation of meso and continuum-scale computational models.

3:20 PM

(ICACC-S4-004-2020) Using Laser-Driven Projectiles for Ceramic Armor Characterization

D. D. Mallick*[1]

1. US Army Research Laboratory, USA

A variety of projectiles can be accelerated using pulsed lasers, from planar metal flyer foils to spherical projectiles. This selectability of impactor geometry offers adjustable loading conditions in stress space. Here we explore the spall failure, changes in hugoniot elastic limit, shear banding, and fracture of advanced protection ceramics (Boron Carbide, Silicon Carbide, and Diamond-SiC Composites) under loading from various projectile geometries at velocities from 800 to 2000 meters-per-second. High speed imaging at 10 million frames-per-second allows us to characterize the flyer geometry and identify active failure mechanisms in the target. Photon Doppler Velocimetry provides nanosecond resolution of the target free surface velocity history and thus the internal stress state during failure.

3:40 PM

(ICACC-S4-005-2020) Impedance Matching Ceramic Matrix Composites for Better Armour Ceramics

J. Teo*[1]; E. Saiz Gutierrez[1]; L. J. Vandeperre[1]

1. Imperial College, Materials, United Kingdom

The use of impedance matching to design better add-on-armour modules is well established. The design strategy of impedance matching dictates that a ceramic should be backed by a sonically harder material to prevent tensile reflections from occurring at the ceramic-backing interface. Tensile reflections are particularly problematic for ceramics as their tensile strengths are orders of magnitude poorer than their compressive strengths. In this work, we present an alternative armour ceramic design based on the impedance matching. It is stipulated that since the main cause of

ceramic failure arises from the backend reflection, engineering a series of small tensile reflections before the compressive wave hit the backend would help reduce the magnitude of that large tensile wave return. A review of some of the ballistics work done by other groups was conducted, primarily using a multi-layer wave propagation programme to predict the effectiveness of the ballistic ceramic which showed positive results. This will be presented alongside some proposed designs to fabricate "better" ceramics for armour applications.

4:00 PM

(ICACC-S4-006-2020) Ceramic Components and Composites and their Application in Ballistic Protection (Invited)

R. Gadow*[1]

1. Institute for Manufacturing Technologies of Ceramic Components and Composites, University of Stuttgart, Germany

Hard ceramic bulk materials are the preferred material solution for ballistic protection of various systems. Besides the mentioned bulk ceramics, ceramic layer composites developed at IFKB proved their strength and performance for protective systems. Particularly in personal protection but also in armor of motor vehicles and helicopters, these material systems have largely displaced the armor steels used so far. The main advantage of ceramic armor is the significant lower area weight compared to high strength armor steel plates. Ceramic composite armoring protect against a wide spectrum of hostile threads including direct gunfire, mortar and artillery shatter as well as ground mines. The protective panels can only fulfill their task safely if the material system is correctly tailored. For this purpose, these systems have to fulfill the requirements, like high hardness, fracture toughness and stiffness. Lightweight engineering potentials are not to be neglected in order to lower the overall masses of armored vehicles, improve handling in critical situations and enable airborne operations. Multihit resistivity is a further issue. During this lecture detailed manufacturing technologies like supersonic fast liquid fuel driven thermal spraying with powders and agglomerates (HVOLF) including relevant test results are presented as well as current application examples for modern military purposes.

4:30 PM

(ICACC-S4-007-2020) Composite armor based on borides and carbides

T. Prikhna*[1]; R. A. Haber[2]; P. Barvitskyi[1]; A. Neshpor[3]; V. Moshchil[1]; C. Hwang[2]; A. Maznaya[4]; A. Kozyrev[1]; V. Muratov[4]; L. Devin[1]; M. Karpets[1]; S. Dub[1]; E. Prysiazhna[1]; A. Lokatkina[1]

1. Institute for Superhard Materials of the National Academy of Sciences of Ukraine, Ukraine
2. Rutgers University, Department of Material Science and Engineering, USA
3. Institute for Problems in Material Sciences of the National Academy of Sciences of Ukraine, Scientific and Technical Center "Composite Materials", Ukraine
4. Institute for Problems in Material Science, NAS Ukraine, Ukraine

The presentation compared the structure and properties of composite light-weight armor with high ballistic performance. The armor plates contained cylindrical or plate-like ceramic elements from hot-pressed $AlB_{12}C_2$-TiB_2, B_4C-SiC or pressureless sintered self-bonded SiC composites with improved mechanical characteristics placed on duralumin alloy support, impregnated in polyurethane and wrapped in Kevlar. The hot pressing at 30 MPa and 1950 °C of α-AlB_{12} nanopowder with TiC addition can result in formation of ceramic elements having according x-ray diffraction analysis 74wt.%$AlB_{12}C_2$-22wt.%TiB_2-4wt.%Al_2O_3 composition and containing according to SEM EDX analysis $Al_{0.17}B_{4.55}C_2O_{0.04}N_{0.21}$, $TiB_{2.58}C_{0.8}Al_{0.02}$ and $Al_2O_{3.79}B_{2.04}C_{0.8}$ phases, which demonstrated s_{cs}=795 MPa compression and s_{bs}=646 MPa bending strengths, H_V(49 N-load)= 37.7 GPa hardness, K_{1C}(49 N-load)=5.2 MPa•$m^{0.5}$ fracture toughness and r=3.12 g/cm^3 density. Manufactured from B_4C and SiC powders composite material containing 78 wt.% B_4C (with some dissolved Si: $B_{3.64}CSi_{0.01}$) and 22 wt.% SiC ($SiC_{1.07}$) demonstrated s_{cs}=1878 MPa, s_{bs}=474 MPa, H_V(49 N)=29.6 GPa and H_V(4.9 N)= 51.7 GPa, K_{1C}(49 N)=5.4 MPa•$m^{0.5}$ r=2.63 g/cm^3 and high level of protection from B32 bullets of 7.62 caliber. The armor from pressureless sintered self-bonded SiC which can protect from B-32 bullets of 12.7 and 14.5 mm calibers has surface density 59-61 kg/m^2 and 91-96 kg/m^2, respectively.

4:50 PM

(ICACC-S4-008-2020) A Unified Analytical Model for the Dynamic Response of Armor Ceramics to Impact and Penetration

S. Bavdekar*[2]; S. Satapathy[1]; G. Subhash[2]

1. US Army Research Laboratory, FCDD-RLW-PB, USA
2. University of Florida, Mechanical and Aerospace Engineering, USA

A new unified model for dwell and penetration during the impact of long projectiles on thick ceramic targets has been developed using the principles of conservation of mass and momentum. The target response is assumed to occur in a hemispherical region containing nested comminuted, cracked and elastic regions of deformation. A dynamic expanding cavity model is used to capture the stress fields in these regions. The material constitutive behavior is predicted using the extended Mohr-Coulomb (SS-EMC) model, which captures the pressure-dependent shear strength of ceramics with a single set of model constants that are applicable to a wide range of ceramics. Hence, the model can predict important ballistic parameters like the depth of penetration and the penetration resistance of ceramics, and can be used to quantify the benefits of superior mechanical properties. The predictions of the new model are in good agreement with experimental results obtained from long-rod impact tests published in literature. The results of the analysis reveal that the properties of the comminuted material have a greater influence on the ballistic performance of ceramics than those of its intact state. The model is also used to quantify the effect of amorphization on boron carbide.

Quasi-Static and Dynamic Behavior I

Room: St. Johns
Session Chairs: Sikhanda Satapathy, Army Research Laboratory; Ghatu Subhash, University of Florida

5:10 PM

(ICACC-S4-009-2020) Experimental analysis of thermally shocked ceramics as models of damaged behavior

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

1. University of Alberta, Edmonton, Mechanical Engineering, Canada
2. Weapons and Materials Research Directorate, Combat Capabilities Development Command Army Research Laboratory, USA

Experimental studies of brittle failure mechanisms are primarily concerned with testing pristine materials to failure, but the failure process can be broken down into three distinct stages: intact, cracked but not fragmented, and fragmented. In the field of advanced ceramics it is easy to assume that the cracked but not fragmented stage occurs too quickly to contribute to the overall behavior, but from the study of planetary sciences it is known that damage accumulation in fault lines does not follow a monotonic response of degree of cracking to changes in strength and elastic properties. In this study, we explore the mechanical behaviors of advanced ceramics subjected to thermal shocking as a model material for the intermediate damage stage. Mechanical experiments using dynamic and quasi-static methods are combined with digital image correlation and ultra-high-speed photography to produce two dimensional stress-strain profiles to discern the effects of the damage. The use of X-ray computed tomography upon these samples allows for the quantification of the degree of damage actually present, and further

these model thermal shocked materials are compared to mechanically damaged materials. Mechanical response is then compared to quantified damage and correlations between the two are drawn to produce new insights into the behavior of damaged materials.

5:30 PM

(ICACC-S4-010-2020) Dynamic Fragmentation Behavior of Confined Ceramics

J. J. Pittari*[1]; T. R. Walter[1]

1. CCDC Army Research Laboratory, Material Response and Design Branch, USA

A comprehensive understanding of the mechanics of failure and dynamic fragmentation behavior of ceramics during impact is sought to facilitate improvements in current simulation techniques for these types of interactions, which primarily rely on calibration fits of penetration data. Furthermore, quantifiable shape descriptors of the comminuted material, such as morphology, size distribution, and solid fraction, are necessary inputs into mesoscale models to investigate the granular flow behavior of failed ceramics under these extreme conditions. The objective of this research is to develop techniques that can generate and capture the experimental data quantifying the behavior of ceramics subjected to triaxial loading states, both fully-dense and comminuted alike, to feed constitutive models. Several preliminary studies of confined alumina rods have been conducted with promising results. The specimens are uniaxially loaded to failure in order to mechanically fail the ceramic and capture the fragments in situ. The specimen is then scanned via microcomputed tomography to quantify the fragmentation behavior of the ceramic, and, subsequently, reloaded to examine the granular flow behavior of the comminuted ceramic material. The objective is to extrapolate this developed methodology towards future studies investigating the presence of rate effects on damage evolution and comminuted behavior of strategic ceramic materials.

S5: Next Generation Bioceramics and Biocomposites

Next Generation Bioceramics I

Room: Coquina Salon C

Session Chairs: Akiyoshi Osaka, Henan Univ of Sci & Tech; Masanori Kikuchi, National Institute for Materials Science (NIMS)

1:30 PM

(ICACC-S5-001-2020) Biomedical Applications of Cerium Oxide Nanoparticles: Applications, Mechanisms and the Road Ahead

A. Karakoti*[1]

1. University of Newcastle, Global Innovative Center for Advanced Nanomaterials, Australia

Biomedical applications of cerium oxide nanoparticles – 'nanoceria' has observed a burgeoning interest from its first reported use in 2005. Since then it has been used as a novel antioxidant that mimics the function of Reactive Oxygen Species (ROS) scavenging enzymes such as superoxide dismutase, catalase, and glutathione etc. to maintain redox homeostasis. The antioxidant property has been exploited in prevention and treatment of multiple diseases mediated by oxidative stress. The ability to reversibly switch its oxidation state between Ce^{3+} and Ce^{4+} is fundamental to these applications. High surface energy due to small size of particles creates surface oxygen vacancies that promote the enzymatic activity by increasing Ce^{3+} concentration. Oxidation state of cerium ions and its reversibility was found to be more important for its antioxidant activity as compared to oxygen vacancies. This talk will summarize various applications of cerium oxide nanoparticles in biomedical applications with emphasis on possible mechanism of its interaction with hydrogen peroxide and superoxide. It will also discuss the importance of oxidation state and presence of oxygen vacancies on its surface for preventing ROS mediated damage. At the end it will discuss the current trends and applications of nanoceria with role of ligands in manipulating its redox activity for carrying out enzymatic reactions.

1:50 PM

(ICACC-S5-002-2020) Preparation of Gentamicin-Sulfate Loaded Hydroxyapatite/collagen Bone-Like Nanocomposite for Antibacterial Bone Void Fillers (Invited)

M. Kikuchi*[1]; S. Oshima[3]; K. Ozeki[3]; M. Honda[2]

1. National Institute for Materials Science (NIMS), Bioceramics Group, Japan
2. Meiji University, Japan
3. Ibaraki University, Japan

Protection of infection during surgical operation is important for good prognosis; therefore, surgical operation room, surgeons and nurses control their aseptic conditions. However, aseptic environment can difficult to control sometimes in developing countries and/or in fields including battlegrounds; thus, antibacterial property would help to control infections during surgery. Further, implants for bone defect repair with good substitution property with bone reduce a risk of biofilm formation by gradual disappearance of implant itself, a possible place for infection focus. Hydroxyapatite/collagen bone-like nanocomposite (HAp/Col) is one of the excellent materials for bone repair because it is incorporated into bone remodeling process to be changed into newly formed bone. In the lecture, adsorption and desorption properties of gentamicin sulfate on the HAp/Col and good antibacterial properties were presented.

2:10 PM

(ICACC-S5-003-2020) Engineering of silicone-based mixtures for the digital light processing of akermanite scaffolds (Invited)

A. Dasan[3]; H. Elsayed[1]; J. Kraxner[3]; D. Galusek[2]; P. Colombo[1]; E. Bernardo*[1]

1. University of Padova, Department of Industrial Engineering, Italy
2. IIC SAS, Joint Glass centre, Slovakia
3. University of Trenčín, FunGlass (Centre for Functional and Surface Functionalized Glass), Slovakia

Preceramic polymers and oxide fillers have been recently established as precursors for several bioactive glass-ceramics. CaO and MgO easily react with silica deriving from the pyrolysis, in air or in nitrogen, of silicones. The phase purity is enhanced by the introduction of borate salts, providing liquid phase upon firing at 900-1200 °C, in turn promoting ionic interdiffusion. A key point is represented by the coupling of synthesis and shaping of bioceramics, since highly porous bodies are first manufactured with silicones in the polymeric state, at low temperature, and later subjected to ceramic transformation. After successful application of direct ink writing of silicone-based pastes, for the obtainment of akermanite ($Ca_2MgSi_2O_7$) reticulated scaffolds, the present study illustrates the tuning of mixtures, for the same bioceramic, in order to form scaffolds by digital light processing. This implied the selection of commercial silicones, to produce stable and homogeneous blends with a photocurable resin and also enabling the manufacturing of defect free printed scaffolds, before and after firing, without fillers. The blends are further refined with the introduction of fillers, followed by firing at 1000 °C, in air. Optimized blends (from H44 resin) including reactive fillers (up to 4.5wt% borax), lead to crack-free, phase-pure scaffolds, with microporous struts.

2:30 PM

(ICACC-S5-004-2020) Suspension Flame Sprayed Metal Doped Calcium Phosphate Coatings with Antibacterial Properties for Infection Prophylaxis (Invited)

R. Gadow*[1]; A. Killinger[1]; A. Bernstein[2]; M. Blum[1]

1. Institute for Manufacturing Technologies of Ceramic Components and Composites, University of Stuttgart, Germany
2. Musculoskeletal research lab, Clinics of Orthopedics and Trauma Surgery, Germany

High Velocity Suspension Flame Spraying (HVSFS) has been successfully employed to produce a wide variety of bioceramic coatings for prosthetic devices and bone implants. The HVSFS process has proven to be capable to process biomaterials resulting in dense and well adherent coatings on various types of metal and ceramic substrates. Degradable bioceramic coatings offer a faster osseointegration of endoprosthetic structures. A common problem that occurs after the application of all types of implants is the risk of infection due to the presence of bacteria which can result in severe post operative inflammation reactions associated with a high risk of losing the implant. In a novel approach, metals with known antibacterial properties are incorporated into the coating as a nanosize dispersion dopant to reduce the risk of inflammation. Metal doped coatings based on bioceramics were suspension flame sprayed using modified suspensions containing additional metals or metal salt based precursors. These coatings were evaluated regarding their microstructure and phase composition, as well as their in-vitro behavior. The presence of metal and metal oxide particles in the coating were characterized using micro-Raman and SEM. To evaluate the biocompatibility, a live/dead-assay study based on MG-63 cells was performed. Results showed no evidence for any cytotoxic reaction.

3:10 PM

(ICACC-S5-005-2020) Lithography-based additive manufacturing of zirconia for dental and medical applications (Invited)

M. Schwentenwein*[1]; J. Rabitsch[1]; T. Konegger[2]; J. Homa[1]

1. Lithoz GmbH, Austria
2. TU Wien - Vienna University of Technology, Institute of Chemical Technologies and Analytics, Austria

Lithography-based additive manufacturing (AM) techniques show great promise for biomedical applications because of their high precision and resolution. This contribution focuses on ceramics made by lithography-based ceramic manufacturing (LCM) for a demanding application area: zirconia ceramics for dental or medical use. This contribution focuses on the mechanical and biological properties of the 3D printed ceramic materials and shows that the achievable properties are fully in line with the characteristics of traditionally fabricated components (>99.5% rel. density, 4 point bending strength >950MPa). Beside materials characterization including mechanical, biological as well as optical properties, this paper will also present first showcases of 3D printed components in biomedical applications. Zirconia as bioinert and exceptionally tough ceramic allows the production of permanent bone replacements and also dental replacements like implants, crowns, bridges and orthodontic products like brackets. Other examples of permanent ceramic implants include osteosynthesis materials like plates and screws but also implantable blood pumps.

3:30 PM

(ICACC-S5-006-2020) Hydroxyapatite nanoparticles due to direct conversion of calcium carbonate in phosphate solutions (Invited)

Y. Sun[2]; A. Osaka*[1]; G. Wang[2]

1. Okayama University/Henan Univ of Science & Tech, Japan
2. Henan University of Science & Tech, School Mat Science & Engineering, China

We have studied the converstion of CC nanoparticles to Ca-P particles when in contact with alkali hydrogenphsophate (R2HPO4: R=Na, K) solutions of varied concentrations and temperature. Scanning electron microscopy showed that the particles took a core-shell type structure at the earlier stages while they were assemblies of petal like crystallites, and sometimes they took a hollow microstructure. X-ray diffraction (XRD) studies indicated that hydroxyapatite diffractions appered 1 h after the contact, and the conversion finished in 1 d when 0.15 M solution was employed while it took 2 d with 0.1 M solution. Magic Angle Spinning Solid State NMR spectra confirmed that the Ca-P particles constituted of HCA. In addition, the CC partilces developed on bioinert substrates were also converted to form HCA layers. The effects of pH and the presence of smaller physiologically active substances on the conversion mechanisms will be discussed as well as the application of this procedure for bone substituting/filling materials.

3:50 PM

(ICACC-S5-007-2020) Micro-to-nano surface roughening of 3Y-TZP for improved osseointegration and bacteria reduction (Invited)

A. Kocjan*[1]; J. Moritz[2]; A. Abram[1]; A. Dakskobler[3]; K. Ivicak-Kocjan[4]

1. Jozef Stefan Institute, Slovenia
2. Fraunhofer IWS, Germany
3. Vall-Cer d.o.o., Slovenia
4. National Institute of Chemistry, Slovenia

Ti implants are widely used in dentistry but the increasing medical and aesthetical concerns have set 3Y-TZP with superior biocompatibility and appearance as an all-ceramic, metal-free alternative. The tissue response can be enhanced if the micro-to-nano surface roughening is provided. Micro-roughening can be achieved via sandblasting, which also improves strength and ageing resistance of 3Y-TZP, and is readily adopted in everyday dental laboratory practice. However, nano-roughening can only be achieved by complex nanostructures deposition or aggressive acid etching, where the latter is problematic due to the brittle nature of ceramics and ageing (in)stability. This work employed a simple, non-invasive approach for nano-roughening of micro-roughened 3Y-TZP achieved by sandblasting by the application of a nanostructured alumina coating. Such surfaces were inspected for their morphology and composition, roughness, wetting, and zeta potential and correlated to the behaviour of human osteoblast cells and Staphylococcus aureus bacteria. The combination of micro-to-nano-roughening was beneficial for initial cell attachment and differentiation while limiting bacterial adhesion. The presented strategy that is already available in dental laboratories could represent an effective solution for modifying the surfaces of 3Y-TZP dental implants.

4:10 PM

(ICACC-S5-008-2020) Processing and Characterisation of Multifunctional Pressureless Sintered Al_2O_3-$CaTiO_3$ Nanocomposites (Invited)

P. K. Mallik*[1]; J. K. Sahoo[1]; S. Mallick[1]; S. Patnaik[1]

1. Indira Gandi Institute of Technology Sarang, Metallurgical and Materials Engineering, India

Despite being highly wear resistance, bioinert and biocompatible, some of the limitations like poor fracture toughness, lack of bioactivity and electrical conductivity properties restrict the use of monolith alumina as bone replacement material. In this paper,

we address one such issue and will demonstrate how $CaTiO_3$ (CT) addition enhances bioactivity, physical and electrical properties like dielectric constant and electrical conductivity etc. Therefore, the strategy in the current research is to develop dense alumina Al_2O_3-CT biocomposites using conventional pressureless sintering technique (1200 °C, 2 hours) that can be enhanced the functional properties compared to that of monolith alumina. The microstructural as well as functional characterizations were carried out by using XRD, SEM and impedance analyser. As results due to the effects of nanosized of Al_2O_3 powders, XRD results indicate the strong peaks of Al_2O_3, CT and the trace of weak peaks of $CaAl_2O_3$. A SEM result indicates the average grain size is in nanometer scale. Due to nanocomposite, the dielectric constant and electrical conductivity increases with function of frequency and temperature was higher compared to other composites. Finally, it is expected that effect of electrically bioactive Al_2O_3-CT nanocomposite will be potential candidate materials for electronic interfacing materials for biomedical application.

S6: Advanced Materials and Technologies for Rechargeable Energy Storage

All Solid State Batteries

Room: Tomoka A

Session Chairs: Palani Balaya, National University of Singapore; Olivier Guillon, Forschungszentrum Juelich

1:30 PM

(ICACC-S6-001-2020) Operando Analysis of All-Solid-State Battery Cathodes Using X-Ray Absorption Spectroscopy Measurements (Invited)

Y. Kimura[1]; T. Nakamura[1]; K. Amezawa*[1]

1. Tohoku University, IMRAM, Japan

Recently all-solid-state lithium-ion batteries (ASSLIBs) attract attentions because of their potential of high power density as well as safety operation. In ASSLIB electrodes, particles of electrode active material and solid electrolyte are randomly mixed and paths for ion and electron transfer are constructed three-dimensionally. The complicated paths for the charge carriers' transfer may cause an inhomogeneous charge/discharge reaction in the electrode, resulting in the capacity losses. For the appropriate design of the electrode microstructure, which can suppress such an inhomogeneous reaction, it is important to understand how the reaction proceeds and what controls the reaction rate in the electrode. In this work, we developed various operando analytical techniques to investigate the charge/discharge reaction in ASSLIB electrodes. For instance, the computed-tomography X-ray absorption fine structure (CT-XAFS) technique was applied to investigate the propagation of the charge/discharge reaction in ASSLIB electrodes. This technique enables us to visualize the spatial distribution of the state of charge (SOC) in the battery electrodes under the operation. In the presentation, our recent results obtained with operando CT-XAFS and other types of XAFS measurements of oxide-type ASSLIBs will be introduced.

2:00 PM

(ICACC-S6-002-2020) Interphase Formation and Chemo-Mechanics in Ceramic Electrolytes for Solid-State Batteries (Invited)

M. McDowell*[1]

1. Georgia Institute of Technology, Mechanical Engineering, Materials Science and Engineering, USA

Most solid-state electrolyte (SSE) materials are unstable in contact with lithium metal, and reactions between SSEs and lithium result in the formation of an interphase region. Understanding the dynamics of this process is key for controlling solid-state interfaces. Here, we use a variety of in situ experiments to reveal how the formation of the interphase in NASICON and sulfide-based solid-state batteries is linked to cell degradation. In situ transmission electron microscopy (TEM) shows that the reaction of $L_{1+x}Al_xGe_{2-x}(PO_4)_3$ (LAGP) with lithium is similar to a conversion reaction, in which lithium insertion causes amorphization and volume expansion of ~130%. In situ X-ray tomography experiments of operating LAGP-based cells reveal that the growth of the interphase causes fracture of the SSE, and quantification of the crack network shows that the extent of fracture is directly correlated to impedance within the cell. We have furthermore found that interphase growth trajectories can be modulated through the deposition of interfacial protection layers, which can extend the cycling stability of symmetric cells from ~30 hours with unprotected SSEs to >1000 hours with protected materials. Overall, these results show that control over interfacial transformation processes could enable a wider variety of materials to be used in solid-state lithium metal batteries.

2:30 PM

(ICACC-S6-003-2020) Room-temperature all-solid-state sodium batteries with robust ceramic interface between electrolyte and electrode materials

T. Lan[1]; Q. Ma[1]; C. Tsai[1]; F. Tietz[1]; O. Guillon*[1]

1. Forschungszentrum Juelich, IEK-1, Germany

All-solid-state sodium batteries (Na-ASSBs) are regarded as an ecologic and economical alternative to their Li congeners for stationary applications. Ceramic-based Na-ASSBs benefit from the high conductivity of the oxide Na-ion conductors used as electrolyte, as well as from their good mechanical, chemical and thermodynamic stability. However, so far Na-ASSBs suffer from severe contact problems in the positive electrodes and subsequently unsatisfactory cell performance. In this report, the inter-ceramic contact problems are solved by combining the infiltration of a porous electrolyte scaffold by precursor solution with in situ synthesis of electrode active material (chemical infiltration) to fabricate model cells using $Na_3V_2P_3O_{12}$ (NVP), $Na_{3.4}Zr_2Si_{2.4}P_{0.6}O_{12}$ (NZSP) and Na as the positive electrode, electrolyte and negative electrode materials, respectively. Optimized interface between NVP nano layer and NZSP backbone was formed, providing effective ion transfer and minimizing the stress caused by volume change of electrode active material during charging and discharging processes. The interface resulted in a highly stable battery performance at 25 °C. The performances, achieved without using any liquid or polymer phase as an accommodation medium, suggest a promising future for chemical infiltration as fabrication process for ceramic-based ASSBs.

3:10 PM

(ICACC-S6-004-2020) Synchrotron X-ray imaging of all solid state batteries and interfaces (Invited)

J. Wang*[1]

1. Harbin Institute of Technology, China

In recent years, there has been a dramatic increase in research efforts related to solid state batteries for advanced energy storage application. Deep understanding of solid state batteries and their interfaces, which is an important factor for the development of next-generation energy systems, has proven considerably insightful. Elucidating these issues necessitates advanced characterization methods, in particular imaging techniques enable spatial visualization of electrochemical reaction, considering the technological difficulties and in operando experimental complexities in this complex energy system. Taking advantages of high-brilliance and high-coherence synchrotron X-ray source, considerable progresses have been achieved in synchrotron X-ray imaging techniques to provide invaluable insights into electrochemical reaction mechanism. Based on our successful experience in developing X-ray nanotomography techniques for energy materials, in this talk, we will discuss a few newly developed synchrotron X-ray imaging methods and highlight the X-ray tomography with

in-operando research capability for solid state battery research. Instead of the technical principle, we mainly discuss how this technique can advance our fundamental understanding the complex solid state electrochemistry. Challenges and opportunities of operando X-ray imaging technology for solid state battery research will be also discussed.

3:40 PM

(ICACC-S6-005-2020) Ion conducting polymers that emulate LiPON. Towards all solid-state batteries (ASBs) (Invited)

R. M. Laine*[1]; E. Temeche[1]; X. Zhang[1]

1. University of Michigan, Materials Science and Eng., USA

Cycling lithium and sodium batteries containing liquid electrolytes often leads to uneven plating of metal on charging that generates dendrites that penetrate the polymer membranes used to separate anodes from cathodes that are coincidentally permeable to dissolved electrolytes. The resulting short circuiting leads to cell failure prompting investigation of ceramic electrolytes thought to mechanically block dendrites from bridging. The search for ceramic electrolytes that offer Li^+ conductivities of 0.1-1 mS/cm has focused on LATP and c-LLZO. Unfortunately, LATP undergoes irreversible reduction of Ti^{4+} during cycling and c-LLZO is susceptible to Li dendrite penetration along grain boundaries. One solution has been to use very thin films (5-200 nm) of gas phase deposited, amorphous LiPON to block this behavior. Unfortunately LiPON thin films offer Li^+ conductivities of 10^{-3} to 10^{-5} mS/cm that mandate the use of thin films. We have successfully synthesized LiPON-like polymers in an effort to explore their utility in the assembly of ASBs. We report here that these polymers show ambient conductivities of up to 1 mS/cm and can be used to assembly ASBs with lithium sulfur cathodes and Li anodes that offer cyclability with energy densities of ≥ 750 mAh/g_{sulfur} over 200 h and 100 cycles at 0.5C with > 90 % efficiency.

4:10 PM

(ICACC-S6-006-2020) Ceramic cathodes for all-solid-state batteries: Co-sintering of Li conductive oxide and active material

K. Waetzig[1]; J. Schilm[2]; J. Beaupain[1]; H. Auer[1]; K. Nikolowski[1]; M. Wolter[1]; M. Kusnezoff*[1]

1. Fraunhofer IKTS, Germany
2. Fraunhofer IKTS, Materials and components, Germany

In all solid-state batteries the conventional liquid electrolyte is substituted by Li conductive solids. An innovative battery concept is discussed with a ceramic cathode, a solid electrolyte as separator and a Lithium metal-based anode. In this concept, the cathode consists of a solid electrolyte for Li conductivity and an active material as main component. The aim is to study the reaction of the Li conductive oxide LATP ($Li_{1.3}Al_{0.3}Ti_{1.7}(PO_4)_3$) with high capacitive cathode material NCM622 ($LiNi_{0.6}Mn_{0.2}Co_{0.2}O_2$) by co-sintering to produce a full ceramic cathode. The start of reactions between LATP and NCM in the range of 550 and 600 °C was indicated by X-ray diffraction. As reaction products Li_3PO_4 and oxides with elements of the cathode material are detected. The analysis of the reaction products is suggesting a Li diffusion from the cathode material to the electrolyte, whereas the NCM is changing the layered structure to two types of spinel and to mixed oxides without Li at higher temperatures. The influence of coatings on the cathode material will be discussed in comparison to the uncoated powder. The compatibility of the active material with the solid electrolyte is studied by phase and microstructure analysis of the sintered composites. The co-sintered cathode is characterized electrochemically with a conventional liquid-infiltrated separator compared to polymer.

4:40 PM

(ICACC-S6-007-2020) Investigation of scalable solid-state battery components

D. Dornbusch*[1]; R. Viggiano[1]; F. Dynys[1]

1. NASA Glenn Research Center, USA

Development of solid-state electrolytes for next generation batteries is critical for enabling future technologies such as electric aviation with the promise of higher energy, higher power, and improved safety by replacement of flammable organic liquid electrolytes. However, a large challenge preventing the adoption and commercialization of solid-state batteries is due to their difficulty in manufacturability and scalability. Tape casting is a commonly applied technique in current state-of-the-art lithium-ion batteries and can be adapted for the scalable manufacturing of solid-state batteries. The production of solid-state battery components through this method is demonstrated. Tape-casting is capable of producing solid-state battery components with desirable morphologies and thicknesses that can transition into larger scale production. Active material and electrolyte materials were tested to determine their compatibility with this production method by analyzing their stability with slurry processing, solvent compatibility, and retention of electrochemical parameters.

5:00 PM

(ICACC-S6-008-2020) Electrochemical properties of $Na_2FeP_2O_7$ glass-ceramic cathode in all-solid-state battery

T. Honma*[1]; H. Yamauchi[2]; J. Ikejiri[2]; F. Sato[2]; T. Komatsu[3]

1. Nagaoka Univeristy of Technology, Department of Materials Science and Technology, Japan
2. Nippon Electric Glass Co., Ltd, Japan
3. Nagaoka University of Technology, Japan

The expansion of demand for lithium ion batteries has resulted in soaring prices of the constituent resources. From the viewpoint of safety, studies on all-solid-state batteries are actively being carried out. In recently Yamauchi et al. succeeded in driving all-solid-state batteries derived from nontoxic oxide glasses at room temperature. The main structure of the ceramic batteries with a simple structure in which $Na_2FeP_2O_7$ crystallized glass and β″-alumina solid solution are joined by pressureless cofiring at 550°C. In this study, we examined crystallization behavior and its electrochemical properties of $Na_2FeP_2O_7$ cathode in oxide base all-solid-state battery. During the crystallization of Na_2O-Fe_2O_3-P_2O_5 glass, fusion with the β″-alumina solid solution is achieved. Reversible charge and discharge of 80 mAh/g were achieved at room temperature. It is not necessary to apply pressure during cell preparation or the use of the batteries. Furthermore, the strong junction at the cathode and electrolyte interface does not peel off during charge and discharge over a long period of 623 cycles. Surprisingly, ex situ X-ray photoelectron spectroscopy revealed partial Fe^{4+} induction and a reversible charge and discharge reaction even after overcharging to 9 V. It was demonstrated that $Na_2FeP_2O_7$ is very stable against overcharging to 9 V.

5:20 PM

(ICACC-S6-009-2020) Ion conducting polymers that emulate LiPON. Precursor/PEO solid solutions as solid electrolytes: Towards all solid-state batteries (ASBs)

R. M. Laine*[1]; E. Temeche[1]; X. Zhang[1]

1. Dept of Materials Science and Eng, USA

Cycling Li^+ and Na^+ batteries containing liquid electrolytes can lead to uneven plating of metal that generates dendrites that penetrate polymer membranes used to separate anodes from cathodes causing short circuits and cell failure. This has prompted efforts to use ceramic electrolytes to mechanically block dendrites from bridging. Ceramic electrolytes that offer Li^+ conductivities commensurate with liquid electrolyte systems with 0.1-1 mS/cm has focused on LATP

and c-LLZO. Unfortunately, LATP undergoes irreversible reduction of Ti^{4+} during cycling and c-LLZO is susceptible to Li dendrite penetration along grain boundaries. Such problems can be resolved using very thin films (5-200 nm) of gas phase deposited, amorphous LiPON to block dendrite penetration. Unfortunately LiPON thin films offer Li^+ conductivities of 10^{-3}-10^{-5} mS/cm mandating use of thin films. We have successfully synthesized LiPON-like polymers in an effort to explore their utility in the assembly of ASBs. We report here that these polymers as solid solutions with PEO show ambient conductivities of up to 1 mS/cm and can be used to assembly ASBs. These results contrast with most reported PEO systems that only offer such levels of conductivity at ≥ 60 °C. We will present data on ASB cycling with a variety of cathode materials and Li anodes.

S7: 14th International Symposium on Functional Nanomaterials and Thin Films for Sustainable Energy Harvesting, Environmental, and Health Applications

Nanomaterials for Energy Conversion and Storage and Catalysis I

Room: Flagler A
Session Chair: Yakup Gönüllü, University of Cologne

2:00 PM

(ICACC-S7-001-2020) Multifunctional materials for emerging technologies (Invited)

F. Rosei*[1]

1. INRS, Canada

As the age of fossil fuels is coming to an end, now more than ever there is the need for more efficient and sustainable renewable energy technologies. This presentation will give an overview on recent developments in solar technologies that aim to address the energy challenge. In particular, nanostructured materials synthesized via the bottom–up approach present an opportunity for future generation low cost manufacturing of devices. We demonstrate various multifunctional materials, namely materials that exhibit more than one functionality, and structure/property relationships in such systems, including new strategies for the synthesis of multifunctional nanoscale materials to be used for applications in photovoltaics, solar hydrogen production, luminescent solar concentrators and other emerging optoelectronic technologies.

2:30 PM

(ICACC-S7-002-2020) Interfacial properties determine the functional behavior in composite nano-systems for energy harvesting (Invited)

A. Vomiero*[1]; I. Concina[1]; G. Solomon[1]; P. Ghamgosar[1]; M. Gilzad Kohan[1]

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

Composite nanostructures can be efficiently applied for Sunlight detection and conversion. In most of the applied systems, like photodetectors, excitonic solar cells and (photo)-electrochemical cells to produce solar fuels, nanomaterials can play a critical role in boosting photoconversion efficiency by ameliorating the processes of charge photogeneration, exciton dissociation and charge transport. Critical role in such processes is played by the structure and quality of the interface, which needs to be properly assembled to obtain the desired functionality. Several strategies can be pursued to maximize energy harvesting and storage, including broadening of light absorbance to reduce solar light losses, fastening exciton dissociation and charge injection, reducing charge recombination. A few examples of application of nanocomposites will be thoroughly discussed, including all-oxide coaxial p-n junction nanowire photodetectors and solar cells, core-shell quantum dot fluorophores for high-efficiency lumi-nescent solar concentrators and composite sulfides for hydrogen generation. Emphasis will be given to the role of interface engi-neering in improving the efficiency of energy conversion in different systems, spanning from electric power generation from Sunlight, to chemical fuel production, to conversion of heat lost through thermoelectric materials.

Nanomaterials for Energy Conversion and Storage and Catalysis II

Room: Flagler A
Session Chair: Alberto Vomiero, Lulea University of Technology

3:20 PM

(ICACC-S7-003-2020) Copper-nickel nanoalloys supported on mesoporous TiO_2 as efficient photocatalysts for H_2 production through water splitting reaction

S. Neatu*[1]; K. Belfaa[2]; A. Gardi[2]; M. M. Trandafir[1]; M. Florea[1]; F. Neatu[1]

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2. Unite de Researche Electrochimie, Materiaux et Environnement (UREME), Faculte de Sciences de Gabes, Universite de Gabes, Tunisia

Hydrogen economy achieved by water splitting process is one of the most studied reactions, with high impact on social life. Photocatalysis is a key method in water splitting reaction and implies the succession of a series of photophysical, photochemical and elec-trocatalytic processes. The development of robust, safe, cost-effective and efficient photocatalytic systems for water splitting should take into account the presence of a proper and powerful photon absorber and an efficient, low-cost and earth-abundant electrocatalyst to perform the reaction at high conversions. In this study, Cu-Ni/TiO_2 materials with high photoactivity for water splitting under UV-Vis (no NIR and IR) irradiation were successfully prepared via a simple low-cost deposition route. Thus, 0.5, 1.0 and 1.5 wt. % of different Cu-Ni ratios (1:0, 1:1, 1:2, 2:1, and 0:1) were deposited on meso-porous TiO_2 prepared by an environmentally friendly hydrothermal method. The best Cu-Ni/TiO_2 photocatalyst generates, under certain experimental conditions, a H_2 production rate above 50 mmols $g^{-1}h^{-1}$, which is among the highest values obtained on both Cu and Ni-based photocatalysts. The obtained results open new opportunities in the preparation of very active materials for H_2 production through water splitting based on the optimization of three-component structures.

3:40 PM

(ICACC-S7-004-2020) New Aspects For Thermoelectricity "Hybrid and 3D Thermoelectric Materials" (Invited)

S. Ballikaya*[1]; E. Çelik[2]; M. S. Toprak[3]

1. Istanbul University, Physics, Turkey
2. University of Miami, Aerospace and Mechanical Eng., USA
3. KTH Royal Institute of Technology, Dept. of Applied Physics, Sweden

Thermoelectric (TE) materials have attracted great attention in the past decade years due to their potential application for both power generator and cooling applications. 3D printed and hybrid TE materials have gained new aspects in TE fields. In this study, we reported high temperature TE properties of Bi_2Te_3 materials that were prepared by 3D additive manufacturing technique. Their thermoelectric properties were investigated depending on different sintering temperature. High temperature TE properties of Bi_2Te_3 showed that the electrical conductivity increases with increasing sintering temperature likely due to increase grain size and crystallinity of samples. The maximum ZT value of 0.55 was obtained in sample sintered 500 °C likely due to high power factor and low thermal conductivity in this sample. Magnetic nano particle included conducting polymer (PEDOT:PSS) were prepared by the spin

coating process. The Seebeck coefficient sign indicates that holes are the main charge carriers in all samples. The electrical conductivity increases as the content of nanoparticles increase up to about 2% and then decreases as more nanoparticles are added. In the presence of a magnetic field, the electrical conductivity increases dramatically while there is no significant change in the Seebeck coefficient. This may possibly be due to magnetically ordered nanoparticles assisting in aligning the polymer chains.

4:10 PM

(ICACC-S7-005-2020) Parallelizable electrocatalytic oxygen evolution reactions on metal phosphides

M. Je*[1]; H. Choi[1]

1. University of Cologne, Germany

Mixtures of multiple solid phase often exhibit high electrocatalytic oxygen evolution reaction (OER) activities even when each phase has poor catalytic activity. Our recent theoretical and experimental works have proved our original concept of catalyst design for OER, 'parallelism' for various electrocatalytic materials, such as metal sulfide and metal borides. Similarly, many recent experimental works are reporting that metal phosphides electrocatalysts also have highly increased OER activities when multiple phases are mixed. In this study, using density functional theory (DFT) calculations, we systematically investigated the roles of phase boundary and possibility of OER parallelization of Ni_2P-Fe_2P mixed system. Interestingly, we found that each metal phosphide phase boundary has different adsorption energies of OER intermediate species than Ni_2P and Fe_2P, and behave like a new material phase, leading to efficient conversion of serial OER to parallelized reaction.

4:30 PM

(ICACC-S7-006-2020) Piezoelectrics go lead-free: Potassium Sodium Niobate nanofibers for energy harvesting applications

A. Ichangi*[1]; A. Gomez[2]; N. Panayanthatta[3]; A. Verma[1]; T. Fischer[4]; S. Mathur[4]

1. University of Cologne, Institute of Inorganic Chemistry, Germany
2. Institut de Ciència de Materials de Barcelona, Spain
3. Institut IMEP-LaHC, France
4. University of Cologne, Institute of Inorganic Chemistry, Germany

In our daily lives, we are surrounded by systems which run on different sources of energy, and without any exception, a portion of this energy goes unutilized as waste energy—for example, the vibrations and heat emitted by a cars' engine. On a large scale, energy is mainly harvested by solar, geothermal and wind turbines; however, for harvesting energy on a microscale for low power applications, piezoelectric materials have been among the most promising methods. Currently, in most piezoelectric applications, the Lead Zirconate Titanate (PZT) finds usage owing to its high piezoelectric constant. However, as the lead is a highly toxic material, the search for an alternative biocompatible lead-free piezoelectric material has become a priority. Among the many alternatives, the Potassium Sodium Niobate (KNN) is at the forefront intended to replace the lead-based piezoelectric materials. The KNN is a solid solution having a perovskite structure--$(K_{0.5}Na_{0.5})$ NbO_3. Along with its piezoelectric behavior, the KNN is also photovoltaic and pyroelectric. In this work, we present the nanofibers of KNN for their suitability in hybrid scavenging of energies available within the car—namely the heat and the vibrational energies. The nanofibers were prepared by electrospinning technique and later modified with A-site and B-site cation dopants to enhance the piezoelectric properties.

4:50 PM

(ICACC-S7-007-2020) Nanocrystalline, transition metal oxide/oxy-chalcogenide nanostructures for high-current hydrogen evolution electrocatalysis

G. Giuffredi*[1]; A. Mezzetti[1]; A. Perego[1]; G. Tirelli[2]; P. Mazzolini[1]; F. Fumagalli[1]; F. Di Fonzo[1]

1. Istituto Italiano di Tecnologia, Italy
2. Politecnico di Milano, Italy

The anisotropic distribution of active sites and limited electrical conductivity hinder the maximum Hydrogen Evolution Reaction (HER) performance for Transition Metal Chalcogenides (TMDs), thus research is focused in overcoming the drawbacks by incorporating conductive species in the material or by increasing the active site density. In this contribution we exploit oxygen incorporation and morphology control for self-supported amorphous TMDs to improve their HER performance. We modify the composition of the TMDs by electrochemical methods, releasing excess chalcogen from the structure and incorporating oxygen to form sub-stoichiometric metal oxide phases with high electrical conductivity. Employing Pulsed Laser Deposition (PLD) for synthesis, we precisely control the nanostructuring and morphology of the materials down to the nanoscale. The increased electrical conductivity allows to reach HER performances among state-of-art for TMDs, regardless of their support or crystalline structure, with a Tafel slope of 35 mV dec^{-1} and a -100 mA cm^{-2} overpotential of 170 mV for amorphous molybdenum sulfide and a -10 mA cm^{-2} overpotential for amorphous tungsten selenide of 184 mV. By controlling the morphology, moreover, we relate the structure of the materials to their HER activity descriptors like charge transport, capacitance and onset potential for the reaction.

S8: 14th International Symposium on Advanced Processing and Manufacturing Technologies for Structural and Multifunctional Materials and Systems (APMT14)

Advanced Sintering Technologies I

Room: Coquina Salon A
Session Chairs: Richard Todd, University of Oxford; Hisayuki Suematsu, Nagaoka University of Technology

1:30 PM

(ICACC-S8-001-2020) High and Ultra-High Temperature Ceramic Matrix Composites Fabricated by Rapid Chemical Vapour Infiltration (Invited)

J. Binner*[1]; V. Venkatchalam[1]; M. Porter[1]

1. University of Birmingham, Metallurgy and Materials, United Kingdom

Better materials and manufacturing methods are needed to deliver the next generation of aerospace propulsion systems for both high temperature (e.g. engine parts) and ultra-high temperature (e.g. rocket nozzles & leading edges) applications. SiCf/SiC & Cf/HfB_2 or Cf/ZrB_2 are potential candidates, however, fast & economic manufacturing methods are still needed. Chemical vapour infiltration (CVI) is an effective route capable of creating near fully dense components with an extremely refined microstructure, little or no preform degradation and minimal residual stresses. However, the conventional approach requires production times of 2 – 3 months meaning that the costs are very high and the product expensive. Microwave energy (MCVI) is a potential solution to heat the SiC fibre preform I, whilst radio frequency heating (RFCVI) can be used to heat C fibre preforms. Both produce an inverse temperature profile, meaning the temperature is hottest at the centre of the component in contrast to conventional CVI.

This initiates densification at the centre of the sample, thus avoiding surface porosity closure. It has been shown that MCVI can yield SiCf/SiC composites in under 100 hours of processing time, whilst RFCVI can yield Cf/ZrB_2 and Cf/HfB_2 composites in as little as 25 hours. The results will be presented.

2:00 PM

(ICACC-S8-002-2020) Review of the total bond order density concept for structural and multifunctional materials with applications to material design and processing (Invited)

W. Ching*[1]

1. University of Missouri-Kansas City, USA

Over the past several years, I have been advocating in using the total bond order density (TBOD) as a single quantum mechanical metric in assessing materials properties, especially the mechanical properties. In this presentation, I review a large amount of data we have collected for structural and multifunctional materials. The systems include high entropy alloys, ceramic crystals and composites, metallic glasses, insulating glasses, C-S-H cements, chalcogenide crystals, MAX phases and much more. From the careful analysis of these different classes of materials, and the connection between physical properties to the TBOD and its partial components (PBOD), we suggest strategies for potential rational materials design and its processing.

2:30 PM

(ICACC-S8-003-2020) Synthesis and mechanical properties of highly porous ultrafine-grain Si_3N_4 ceramics via carbothermal reduction-nitridation (Invited)

J. Yang*[1]; Q. Zhi[1]; B. Wang[1]

1. Xi'an Jiaotong University, State Key Laboratory for Mechanical Behavior of Materials, China

In order to improve the mechanical properties of Si_3N_4 ceramics with high porosity, it is necessary to lower the grain diameter and increase the aspect ratio of Si_3N_4 grains. In this work, a novel carbothermal reduction-nitridation (CRN) reaction between carbon nanotubes and SiO vapor in N_2 atmosphere at temperatures ranging from 1550 to 1650 °C was proposed to synthesize highly porous ceramics consisting of fibrous β-Si_3N_4 nanograins derived from green bodies containing 49.0wt% CNT and 40.4wt% α-Si_3N_4 powder, using SiO powder as precursor of gaseous SiO and Y_2O_3 as sintering aid. The as-received porous β-Si_3N_4 ceramic achieved average grain diameter of approximately 290 nm, and average aspect ratio of as high as 20. The porosity of the as-received Si_3N_4 ceramics was up to 73% even if no pore forming agent was used, and the bending and compressive strength were up to 62 MPa and 82 MPa, which are higher than the values in the literatures. It should be noted that the as-received Si_3N_4 ceramics exhibited a large strain to failure and 'pseudo-plastic' deformation behavior during the compressive test, which is attributed to the high porosity.

3:20 PM

(ICACC-S8-004-2020) Closed Loop Recycling of Ceramics (Invited)

R. Riman[1]; D. Kopp*[1]

1. Rutgers University, Materials Science & Engineering, USA

Utilization of carbon dioxide (CO_2) by converting it into valuable products, such as fuels, chemicals, plastics, and building material is thought to be necessary for sufficient carbon management. A breakthrough process invented and patented by Rutgers University called gas-assisted reactive hydrothermal liquid phase densification (g-rHLPD) has been shown as effective in utilizing CO_2 by curing/densifying synthetic and mineral silicates at temperatures below 100°C. In the cement industry, this process has demonstrated a carbon footprint reduction of 65% by sequestering CO_2 during the curing process. This presentation will describe another breakthrough technology that enables the closed-loop recycling of carbonate-cured ceramics and can result in >50% of additional carbon footprint reduction. This presentation will also detail the closed-loop recycling process and how it applies to a myriad of composite structures and provide a perspective on how its contribution to sustainability can impact a broad spectrum of business in performance enhancing products while greatly diminishing CO_2 emissions.

3:50 PM

(ICACC-S8-005-2020) Advanced ceramic foams from metakaolin-based aqueous suspensions actived with inorganic and organic bases (Invited)

A. Rincon[2]; H. Elsayed[1]; F. Dogrul[2]; E. Bernardo*[1]

1. University of Padova, Department of Industrial Engineering, Italy
2. University of Trencin, FunGlass (Centre for Functional and Surface Functionalized Glass), Slovakia

Air may be easily incorporated by vigorous mechanical stirring, with the help of surfactants, of activated geopolymer-yielding suspensions. The cellular structure is stabilised by the viscosity increase caused by curing reactions, configuring an 'inorganic gel casting'. The approach is so flexibile that it may be applied to mixtures embedding fillers, such as reactive γ-Al_2O_3 powders, playing a fundamental role upon ceramic conversion, at high temperatures. After successful development of mullite foams, the present study is dedicated to recent extensions to highly porous cordierite and SiAlON foams, obtained starting from Na-based geopolymer-yielding mixtures added with talc or carbon black, and fired in air (at 1250 °C) or nitrogen (at 1500 °C), respectively. A key intermediate step is represented by the removal of Na^+ ions from 'green' foams, by ion exchange in solution (0.1 M) of ammonium nitrate, for 24-48 h, before ceramization. We will show, however, that direct ceramization is feasible, once the gelation of metakaolin suspensions is not achieved by reaction with NaOH (implying geopolymerization), but with tetramethylammonium hydroxide. The new gels are effective in yielding phase pure cordierite (a limited glass phase is due to the practical absence of any sodium oxide contamination) as well as β/X-SiAlON with minor corundum impurity.

4:20 PM

(ICACC-S8-006-2020) Manufacturing of Ceramic Fiber reinforced Light Metals via Spark Plasma Sintering

M. Jiménez Martínez*[1]; R. Gadow[2]; F. Kern[1]

1. University of Stuttgart, Institute for Manufacturing Technologies of Ceramic Components and Composites, Germany
2. Institute for Manufacturing Technologies of Ceramic Components and Composites, University of Stuttgart, Germany

Spark Plasma Sintering (SPS) is a pulsed current activated, pressure-assisted sintering technique. In comparison to hot pressing (HP), SPS offers a better controlled and more efficient heat transfer in the green body and faster consolidation cycles. In the field of Metal Matrix Composites (MMC), SPS technique has been used so far for the manufacture of ceramic particle and short fiber reinforced alloys. In this work, SPS technique is employed for the first time to produce continuous fiber reinforced metals. For this purpose, MMC prepregs with aluminum as a surface coating on carbon fiber textiles are manufactured by twin arc wire spraying and subsequently consolidated by SPS in a graphite die in semisolid temperature range of the alloy. Shear thinning rheological behavior of the metal alloy at temperatures between solidus and liquidus enables the infiltration of fiber rovings under reduced forming loads. The stable process management of modern SPS presses enables an accurate control of main densification parameters: temperature and pressure. Fully densified samples with no porosity prove the suitability of SPS for densification of continuous fiber reinforced metals. Pulse activated sintering process leads to a quite strong fiber/matrix adhesion with low carbide formation. This presentation focuses on the manufacturing process and the characterization of consolidated samples.

4:40 PM

(ICACC-S8-007-2020) Origin of high interfacial resistance in solid-state batteries: Interdiffusion and amorphous film formation

L. Stanciu*[1]

1. Purdue University, Materials Engineering, USA

The large interfacial resistance between electrolyte and electrode has become a significant roadblock for the commercialization of all-solid-state batteries. Understanding the mechanism of interphase formation, along with investigating its effect on ionic conductivity, could lead to the discovery of avenues towards designing high performance all-solid-state Li ion batteries. In this work, we studied the interphase formation in the perovskite electrolyte $Li_{0.33}La_{0.57}TiO_3$(LLTO) and spinel cathode $LiMn_2O_4$ (LMO) pair by co-sintering experiments via Spark Plasma Sintering (SPS), as well as conventional sintering. While the processing method had an influence on the quality of the electrode/electrolyte contact, the formation of an interphase could not be avoided. At the LLTO/ LMO interface, we observed both an interphase formed by interdiffusion, as well as a complexion-like amorphous layer. We directly characterized the complexion layer morphology via HRTEM. Analytical TEM and SEM where used to reveal the elemental composition of the interphase. Furthermore, we used impedance spectroscopy to measure the electrical properties of the LLTO/LMO interphase and identified the interfacial resistance from the interdiffusion induced interphase to be larger than the individual phases about by a factor of 40, while the amorphous layer was not visible in the impedance.

5:00 PM

(ICACC-S8-008-2020) Effect of sintering aids, phase interface and grain boundaries on thermal conductivity of SiC ceramics fabricated by spark plasma sintering

Z. Chai*[1]; P. Xiao[1]

1. University of Manchester, Materials, United Kingdom

In this study, SiC ceramics were fabricated by SPS to examine the effect of sintering aid content and sintering temperature on thermal conductivity of SiC ceramics and its correlation with microstructure. SiC ceramics with relative density of over 97% were fabricated at 1650-1850°C by SPS with 3-10wt.% Al_2O_3-Y_2O_3 as a sintering aid. Although relative density was independent of the sintering aid content and sintering temperature, variation of the sintering temperature led to difference in grain size of the sintered SiC. With the grain size larger than 700nm, effect of grain size on thermal conductivity was relatively small. It is believed that the presence of the sintering aids has shown little effect on change of thermal conductivity. However, dissolution of alumina in SiC during sintering significantly decreased the thermal conductivity of SiC. Increase in sintering temperature led to less grain boundary segregation in SiC, resulting in higher thermal conductivity of the SiC.

5:20 PM

(ICACC-S8-009-2020) Agar gelcasting of Al_2O_3 beads with different sizes through combination of dripping and novel rapid drying

K. Lertwittayanon*[1]; C. Phetkanchanamala[1]

1. Prince of Songkla University, Materials Science and Technology, Thailand

Al_2O_3 beads with different sizes between1 and 5 mm were rapidly fabricated by two combined processes of dripping into a cooled oil and soaking in acetone. The dripping sizes were controlled by different sizes of dropper tip. Mixture between Al_2O_3 slurry and agar solution was first prepared and maintained at 70°C prior to dripping into soy bean oil cooled at ~ 2°C. Subsequently, the mixture tranformed into sphere and gel due to surface tension and oil coolness, respectively. After the transformation, the rapid drying of transformed mixture was performed by soaking in acetone. for 15 min to allow interdiffusion between the water in spherical gel and the surrounding acetone. Finally, the acetone-rich transformed mixture was dried within 10 min due to the acetone evaporation at RT prior to sintering. The roundness of Al_2O_3 beads decreased when the dripping sizes increased resulting from the dominant weight of gelled mixture. However, the increases in surface tension of the transformed mixture and the viscosity of cooled oil were used to obtain the enhanced roundness percentage of increasing dripping sizes.

5:40 PM

(ICACC-S8-010-2020) Variation in Densification behavior of Yttria Stabilized Zirconia Processed by Spark Plasma Sintering

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

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

The rise of spark plasma sintering (SPS) for rapid consolidation of powder-based materials has occurred without a complete understanding of the underlying mechanisms. As a result, there is sometimes little consensus on the optimal processing parameters needed for various materials. For example, ionic conductors, such as yttria stabilized zirconia (YSZ), have exhibited highly variable electrical and physical properties when sintered by SPS using different parameters. In this study, the variability in processing YSZ using a given set of SPS parameters was investigated. Identical processing conditions were used to sinter YSZ in two different configurations. One configuration used a conductive Ni layer to cap the YSZ during sintering, while the YSZ directly contacted the graphite of the sintering die in the other. Micro-indentation was used to measure the variation in hardness between and within samples produced in each configuration. A significant variation in hardness was observed for bare YSZ samples that also experienced different degrees of reduction. The capped samples exhibited less variability in properties and lower likelihood of reduction during processing as well as reduced hardness overall. These results have several possible implications for developing a better understanding of the mechanisms involved in SPS and improving the processing variability for the technique.

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

Ceramics : Powder, Bulk and Characterization

Room: Tomoka B

Session Chairs: Byung-Koog Jang, Kyushu University; Sungwook Mhin, Korea Institute of Industrial Technology

1:30 PM

(ICACC-S11-001-2020) Control of morphology and oxidation state of metal oxides, using ionic liquids/deep eutectic solvents assisted synthesis (Invited)

O. Gomez Rojas*[1]; T. Nakayama[1]

1. Nagaoka University of Technology, Japan

Ceramics functionality differs from composition, chemical state, as well as from the resultant morphology exhibited by the material. The ionic liquid (& solids)/deep eutectic solvents assisted synthesis has proven efficiency in the production of diverse metal oxides, reducing the overall calcination temperature and lowering cost/time consumed in the synthetic process. However, the synthetic procedure needs further exploration, such as which factors crucially provide control over the resultant morphology (size and shape). On the other hand, a ceramic composed by the same crystal composition

but, changing the valence state of elements contained in the material, provides variations in properties and crystal structures. Therefore, it is of crucial importance to be able to gain exquisite control over the final oxidation state of the resultant material, in addition to simplify and speed up the process.

2:00 PM

(ICACC-S11-002-2020) Nanoparticle synthesis of borides by pulsed discharge of compacted powder

H. D. Nguyen*[1]; M. Ngo[1]; Y. Tokoi[2]; T. Do[1]; T. Nakayama[1]; H. Suematsu[1]; K. Niihara[1]

1. Nagaoka University of Technology, Japan
2. National Institute of Technology, Oyama College, Japan

Borides have various applications because of their excellent mechanical properties. They are usually extremely hard and anti-corrosive in high temperature, as a result, they are being utilized widely in material industries. Moreover, based on Hall-Petch effect, materials as nanosized powders, are expected to increase the strength. In this study, a method to synthesize boride nanoparticles is introduced. By discharging, a large pulsed current went through B micron powder as a raw material which had been compressed by two pieces of electrode of various materials (Fe, Ti, Mo, W). Due to Ohmic heating, B micron powder and electrodes' material contacting B micron powder were heated, melted, vaporized, and reacted with each other to form nanoparticles of borides. These reactions were occurred in Ar gas at 100 kPa. Pulsed current was discharged after charging three banks of capacitor (capacity of each one is 10μF) to 6.2 kV. By TEM bright field images, prepared powder was spherical nanoparticles with size less than 100 nm. With Ti electrodes, after the phase identification by X-ray diffraction, the nanoparticles were mostly of TiB_2.

2:20 PM

(ICACC-S11-003-2020) Development of rheological techniques to analyse the flow of highly filled injection moulding compounds

A. Edwards*[1]

1. University of Birmingham, Chemical Engineering, United Kingdom

Highly filled materials are extensively used in various shape forming processes including injection moulding. Typically, organic or inorganic filler particles are dispersed within a binder matrix to form a feedstock, which is then injected via a plunger or screw type mechanism into a mould pattern of the desired geometry. Determining the fundamental rheological properties of injection moulding feedstock can facilitate in helping to reduce the prevalence of flow defects. The requirement of producing a dimensionally stable geometry means that the filler fraction is often close to the critical solids loading of the binder matrix. The low binder content of the feedstock means that rigid viscoplastic behaviour is observed, this makes trying to determine material flow properties such as a yield stress or viscosity very problematic. Traditionally such material flow properties are determined by employing the use of rotational or capillary rheometers, the idea being that both instruments should cover the range of shear rates that could occur under different processing conditions. Sample preparation is key with such lab based techniques, where sample inconsistency can have a detrimental effect on the accuracy and repeatability of results. Pre forming the sample prior to testing is a solution that can help to mitigate some issues inherent to conventional rheological test methods.

2:40 PM

(ICACC-S11-004-2020) Study about graphitization behavior of furan resin at the interface during C/C composite production

K. Nishimura*[1]; T. Irisawa[2]; T. Yamamoto[3]; Y. Tanabe[2]

1. Nagoya University, Engineering, Japan
2. Nagoya University, Japan
3. Nagoya University, Department of Materials and Design Innovation Engineering, Japan

Carbon fiber reinforced plastics (CFRPs) have been used for structural materials of automobiles and aircrafts because of its properties, and expected to be used in more applications. Meanwhile, recycling process of waste CFRP hasn't been established. Considering sustainability, it is urgently needed to establish recycling process. Since waste CF is usually collected shredded from waste CFRP, this study focused on using recycled short fiber as filler of carbon fiber/carbon composite (C/C composite). Assuming the use of recycled CF as heat transfer material, pitch CF that has a high crystallinity and thermal conductivity was adopted. CFRPs containing 20wt% CF were prepared with furan resin as matrix and discontinuous CF (length: about 100 μm), imitating recycled CF, as filler. They were carbonized at 1500 °C to obtain C/C composites. The density of matrix in C/C composite increased significantly more than that of furan resin itself. It is considered that this was caused by the stress resulting from the difference of shrinkage rate of resin and filler. And this mechanism is called stress graphitization. Furthermore, thermal conductivity of C/C composite, in which graphitization degree of matrix resin increased due to stress graphitization, was much higher than the theoretical value.

3:20 PM

(ICACC-S11-005-2020) Preparation and Characterization of Natural Rubber with Nanodiamond Nanomatrix Structure (Invited)

S. Kawahara*[1]

1. Nagaoka University of Technology, Japan

Inorganic nanomatrix structure is one of the prominent filler network structures, which meets demands of modern and future applications of rubbery polymers. It is formed by dispersing rubber particles as a major component into matrix of nano-composite with inorganic nanoparticles as a minor component. Mechanical properties of the polymers, which are related to the nanomatrix structure may be tunable with size, surface area, interaction and dispersion of the inorganic nanoparticles in the matrix. Nanodiamond is one of the promising nano-fillers to form the nanomatrix structure, since it possesses unique properties and surface. For instance, nanodiamond exhibits diamond like properties, i.e., high hardness, high Young's modulus, high electrical and thermal resistivity and optical properties in nanometer scale. In addition, functional groups on large accessible surface of nanodiamond are useful for chemical reactions without sacrificing the properties as a diamond. In the present study, an attempt to prepare natural rubber with the nanodiamond nanomatrix structure was made to tune the viscoelastic properties.

3:50 PM

(ICACC-S11-006-2020) Role of TiC in the two-steps self-healing mechanism of $Y_2Ti_2O_7$-Y_2TiO_5 -TiC system

A. Okawa*[1]; T. Nguyen[2]; W. J. Paulo[1]; H. Iwasawa[1]; T. Nakayama[1]; T. Do[1]; H. Suematsu[1]; T. Suzuki[1]; T. Goto[1]; K. Niihara[1]

1. Nagaoka University of Technology, Extreme Energy-Density Research Institute, Japan
2. Kushiro National College of Technology, Department of Creative Engineering, Japan

The purpose of this work is to clarify the two-step self-healing behavior of the $Y_2Ti_2O_7$ - Y_2TiO_5 -TiC system. $Y_2Ti_2O_7$-Y_2TiO_5-TiC composites were fabricated by using solid state reaction and hot-press sintering at 1500°C for 1 h in argon (Ar) gas at 25 MPa.

All composites were fabricated at a 1:1 molar ratio between Y_2TiO_5 and TiC. Self-healing was evaluated via annealing at 1200°C for 1 h in air. X-ray diffraction (XRD) confirmed that Y_2TiO_5 reacted with dispersed TiC to form $Y_2Ti_2O_7$ and hence trigger a self-healing process. Thermogravimetry and differential scanning calorimetry (TG-DSC) confirmed that the oxidation of TiC to TiO_2 occurred between 400°C and 650°C as an initial step to activate the crack-healing. Scanning electron microscope (SEM), Vickers hardness and fracture toughness tests were used to evaluate the TiC spatial distribution and mechanical properties of $Y_2Ti_2O_7$-Y_2TiO_5-TiC composites. Results suggest that the adequate addition of TiC is an effective way to enhance the mechanical properties and promote the self-healing process of $Y_2Ti_2O_7$-Y_2TiO_5-TiC composites to extend the lifetime of environmental barrier coatings under thermal cycling operations.

4:10 PM

(ICACC-S11-007-2020) Evaluation of the Water Vapor Corrosion Resistance of $Y_2Ti_2O_7$ as Thermal/ Environmental Barrier Coating

Y. Nakazawa*[1]; T. Nguyen[2]; W. J. Paulo[1]; A. Okawa[1]; T. Nakayama[1]; T. Do[1]; T. Suzuki[1]; T. Goto[1]; K. Niihara[1]

1. Nagaoka University of Technology, Extreme Energy-Density Research Institute, Japan
2. Kushiro National College of Technology, Department of Creative Engineering, Japan

Corrosion behavior of Y2Ti2O2 in a high temperature steam environment was investigated in this study. Y2Ti2O7 sample was fabricated by using solid state reaction and hot-press sintering at 1500°C for 1 h in Ar at 25 MPa. Water vapor corrosion tests were performed at 1100°C 10 h. X-ray diffraction (XRD) and scanning electron microscopy (SEM) were used to characterize the crystallographic phase transformations and Y2Ti2O7 microstructure evolution after water vapor corrosion tests. Vickers hardness and fracture toughness of samples were evaluated before and after water vapor corrosion test. In addition, the coefficient of thermal expansion (CTE) of Y2Ti2O7 is also measured. The bending strength decreased by about 20%, but no decrease in hardness was observed. As a result of X-ray analysis, did not change the crystallographic phase before and after the water vapor test.

4:30 PM

(ICACC-S11-008-2020) Microstructural Properties and Sintering Behavior of Y_2O_3 by Spark Plasma Sintering (Invited)

B. Jang*[2]; B. Kim[1]

1. National Institute for Materials Science (NIMS), Research Center for Functional Materials, Japan
2. Kyushu University, Interdisciplinary Graduate School of Engineering Sciences, Japan

The yttria was densified by spark plasma sintering (SPS) with various heating rates (5, 10, 20, and 50°C/min) and holding times (2, 5, 20, and 60 mins) at 1000°C. In entire sintered bodies, grain size of center was larger than periphery. In case of the sintering loaded high heating rates (20 and 50°C/min) especially, the difference of grain size was significantly large (around 3.5 times on average). There was also a difference in pore distribution. In the uniformly sintered bodies, inter and intra-granular pore existed, but in the non-uniformly sintered bodies, differed pore distributions were observed such as the center (inter granular pore was dominant) and the periphery (inter and intra-granular pore). The porosity of the center dominated by inter-granular pore was higher than that of the periphery. Because there is not any clear mechanism, it is necessary to set the assumption (the defect diffusion occurred from periphery to center by mass transfer) to explain for this phenomenon. Due to the endemic characteristics of SPS, the grain boundary mobility has increased with the phenomenon that the vacancies are gathered in center due to the mass transfer from center to periphery.

5:00 PM

(ICACC-S11-009-2020) Effect of SiC addition on $Yb_2Si_2O_7$ and its corrosion behavior in high temperature water vapor

K. Arai*[2]; T. Nguyen[1]; W. J. Paulo[2]; A. Okawa[2]; T. Nakayama[2]; T. Do[2]; T. Suzuki[2]; T. Goto[2]; K. Niihara[2]

1. Kushiro National College of Technology, Department of Creative Engineering, Japan
2. Nagaoka University of Technology, Extreme Energy-Density Research Institute, Japan

Effect of SiC addition on $Yb_2Si_2O_2O_7$ and its recession behavior in a high temperature steam environment were evaluated in this study. Several $Yb_2Si_2O_7$ - SiC composites (x = 0, 10, 20 vol% SiC) were fabricated by using solid state reaction and hot-press sintering at 1550°C for 1 h in Ar at 30 MPa. All composites were subjected to a water vapor corrosion test at 800°C for 1 h. X-ray diffraction (XRD) was used to characterize the phase transformations before and after water vapor corrosion test, whereas scanning electron microscope (SEM) was used for observing SiC distribution and micro cracks on the surface. Vickers hardness, flexural strength and fracture toughness were measured to evaluate the effect of SiC addition on the mechanical properties of composite samples. After the water vapor corrosion test, Yb_2SiO_5 was identified and the peak intensity was increased. However, SiC vol.20% composite could keep the $Yb_2Si_2O_7$ phase, and its indicate the better water vapor corrosion resistance in this study. Oxidation of SiC yielded SiO_2, which reacted with the remaining Yb_2SiO_5 to form $Yb_2Si_2O_7$. This process is the main mechanism of keep $Yb_2Si_2O_7$ phase. Results suggesting that the adequate amount of SiC addition could help to keep $Yb_2Si_2O_7$ phase under high temperature steam environment, and it is expected to extend the life time.

5:20 PM

(ICACC-S11-010-2020) Impact of Ceramic Raw Materials Variation on Investment Casting Refractories

E. Jones*[1]

1. University of Birmingham, Chemical Engineering, United Kingdom

A fundamental part of the investment casting of turbine blades is the ceramic shell that has to be thermally and chemically stable at high temperature. By analysing several commercially available zircon powders, the effect of mining location and processing on physical and chemical properties have been characterised. When investigating how sourcing effects material properties, the surface properties of the zircon powders has been found to be a significant difference, which has been studied by analysing the surface energies and the different phases present. To predict how differently sourced zircon powders behave in a colloidal system, electroacoustics and ultrasonic attenuation measurement techniques have been implemented to determine differences in zeta potential and the isoelectric point. The effect of zircon's particle size, shape, composition and surface properties on the sintering behaviour of the shell have also been investigated by changing these properties to produce bespoke samples to observe their effect on the dimensional stability of the shell. These properties will then be related to the development of key shell properties and the consequent effect on casting dimensions. Detailed understanding of the material's microstructural development through shell manufacture and subsequent casting will allow Rolls-Royce plc to drive improvements into the dimensional capability of their cast product.

S12: On the Design of Nano-Laminated Ternary Transition Metal Carbides/Nitrides (MAX Phases) and Borides (MAB Phases), and their 2D Counterparts (MXENES, MBENES)

Mechanical Behavior of MAX Phases

Room: Coquina Salon F
Session Chairs: Michel Barsoum, Drexel University; Thierry Cabioch, University of Poitiers

1:30 PM

(ICACC-S12-001-2020) Nanomechanical testing experiments to investigate elementary plastic deformation mechanisms in MAX phases (Invited)

C. Tromas*[1]; S. Parent[1]; A. Joulain[1]; L. Thilly[1]; P. Villechaise[1]; T. Ouisse[2]

1. Institut PPRIME, Physics and mechanics of materials, France
2. Grenoble INP, France

The complex mechanical behavior of MAX phases arises both from their crystallography, with a nanolayered structure, and from their macroscopic polycrystalline structure, composed of platelets-like grains. Nanomechanical testing (nanoindentation, micropillar compression tests) allows distinguishing from these two contributions by probing the mechanical response of individual grains. Two MAX phases have been studied: a Ti_2AlN polycrystal, and Cr_2AlC single crystals recently synthesized by high temperature solution growth. For this second case, the samples are thin platelets of few mm^2, oriented along the basal plane, which have been embedded in a chosen orientation. The elementary plastic deformation mechanisms involved by nanoindentation tests have been studied by Atomic Force Microscopy (AFM) surface observation, in order to analyze the slip lines, and by transmission electron microscopy (TEM) to characterize the dislocations structure in cross section through the indents. An automated mapping of crystallographic orientations was also performed using the ACOM (Automatic Crystal Orientation and phase Mapping) ASTAR technique. These experiments revealed the presence of low angle tilt boundaries associated with dislocation walls, but also highly disoriented domains below the indents, as well as dislocations structures out of the basal plane.

2:00 PM

(ICACC-S12-002-2020) Tensile Creep of Textured Ti_2AlC in the 1000–1150°C Temperature Range

M. Sokol*[1]; T. El-Melegy[1]; M. Barsoum[1]

1. Drexel University, Materials Science and Engineering, USA

The creep behavior of a dense and textured Ti_2AlC MAX phase was investigated, for the first time. The textured creep samples were prepared by two stages: in the first stage a mixture of TiC, Al and C were reactively pre-sintered at 1250°C resulting in a sample close to ~80 % of theoretical density. In the second stage, the porous preform was hot forged using a hot-press at 1400°C under an uniaxial load corresponding to a stress of 40 MPa. The creep experiments were conducted in the 1050–1100 °C temperature range, under an applied tensile stress of 15–50 MPa. The load was applied parallel to the basal planes. The post-creep samples and the protective Al_2O_3 scale were characterized by SEM and TEM, both equipped with an EDS detector. It was found that the creep rates of the textured MAX specimens were considerably lower when compared to untextured Ti_2AlC. Not only the creep rate values for the textured samples were lower than the untextured ones, but the maximal stress values of the former were considerably lower. Considering that the densities, impurities and oxidation kinetics are identical for both microstructures, the enhancements in maximal stress must be due to the higher damage tolerance of the textured microstructure normal to crack propagation. Said otherwise, the textured sample, as a direct result of their basal orientation, can sustain higher stresses than the untextured ones.

2:20 PM

(ICACC-S12-003-2020) Intrinsic deformation and failure response of single crystal MAX phases

Z. Zhan[1]; H. Rathod[1]; T. Ouisse[2]; M. Radovic[1]; A. Srivastava*[1]

1. Texas A&M University, USA
2. Université Grenoble-Alpes, France

A family of ternary carbides and nitrides, referred to as MAX phases, possess unique set of properties. These are light, stiff, thermodynamically stable and refractory, like ceramics, but damage-tolerant, pseudo-ductile at high temperatures and readily machinable like metals. Prior works have shown that polycrystalline MAX phases exhibit a range of deformation and failure mechanisms, such as crystallographic slip, ripplocation, twist, delamination and kinking. Here, we aim to correlate the single crystal level mechanical response of MAX phases to their crystallographic orientation relative to the applied load. To this end, micropillars are extracted from grains of know orientations using FIB milling and are subsequently deformed under compression using a flat-punch nanoindenter. The mciropillar experiments are complemented with novel in-situ SEM indentation experiments on single crystals of MAX phases. Our results shed new lights on the activation of various competing deformation and failure mechanisms in MAX phases at the single crystal level.

2:40 PM

(ICACC-S12-004-2020) Mechanical exfolation of MAX phase and $Mo_4Ce_4Al_7C_3$ single crystals

A. Gkountaras*[3]; Y. Kim[2]; J. Coraux[3]; S. Lisi[3]; V. Bouchiat[3]; M. Barsoum[1]; T. Ouisse[2]

1. Drexel University, Materials Science and Engineering, USA
2. Grenoble INP, France
3. CNRS, NEEL Institute, France

MXenes are usually obtained by chemically etching the aluminum layers intercalated between the 2D $M_{n+1}C_n$ transition metal-carbon layers of Al-based MAX phases. Although MAX phases are not van der Waals solids, we show that single crystals can also be mechanically exfoliated in order to produce flakes whose thickness can be reduced down to half a unit cell of the starting crystal with few tens of microns lateral dimensions. Mechanical exfoliation can be applied to all phases already known for leading to stable MXenes (such as V_2AlC). Besides, it can be applied to MAX phases that do not resist chemical etching (such as Cr_2AlC), to phases where the A element is not Al (such as Ti_2SnC), or to phases where it is important to keep an intercalating A layer in order to preserve, e.g., magnetic properties (such as $Mo_4Ce_4Al_7C_3$). Using electric force microscopy, we analyse the exfoliated flakes transferred onto SiO_2/Si and we find no appreciably variation with flake thickness. Using an appropriate surface preparation, the measurements reveal a contribution from both contact surface potential and the local capacitance, which can be used to confirm the metallic character of the flakes.

Functional Behavior of MAX Phases

Room: Coquina Salon F

Session Chairs: Miladin Radovic, Texas A&M University; Jie Zhang, Institute of Metal Research, Chinese Academy of Sciences

3:20 PM

(ICACC-S12-005-2020) Revealing the catalytic features of MAX phase (Invited)

M. Florea*[1]

1. National Institute of Materials Physics, Romania

MAX phases are well known for their unique properties, of both metals and ceramics, and thousands of studies on many other properties have been conducted so far. However, their potential use as catalysts or as support for catalytic active species had been totally ignored until recently, although they possess the required properties for such processes. Herein, we present the catalytic features of MAX phase powders in catalytic chemoselective selective hydrogenation of a nitro group in the presence of C=C or C=O groups, as well as, their interesting activity in selective oxidation reactions. They are able to chemoselectively hydrogenate 4-nitrostyrene to 4-aminostyrene and selectively oxidize p-cymene, produced from biomass, to terephthalic acid, without noble metal addition. However, to boost their activity, ultra-low noble metal loadings were deposited by different methods on MAX surface, in this way reaching 100% conversion in both, oxidation and reduction reactions. Thenewly discovery of this highly efficient and chemoselective system, MAX phase based one, has broad implications for the design of a new generationcost-effective, earth-abundant, non-toxic, metal catalysts.

3:50 PM

(ICACC-S12-006-2020) Role of intermediate intermetallics in the formation of MAX phases

J. Lyons*[1]; F. Giuliani[2]

1. Imperial College London, Materials, United Kingdom
2. Imperial College London, United Kingdom

An investigation into the formation of the 312 MAX phase Zr_3AlC_2, has been performed to develop the understanding of the mechanism of formation in order to create phase pure material. Principally, the investigation focused in the region of 600-900 °C, to interrogate the formation of the intermediate Zr-Al intermetallics. The effect of the type and concentration of these intermediates on the phase purity of the MAX phase will be discussed along with its relation to the heating cycle. The controlled formation of these intermediates has allowed for consistent high phase purity (> 70 %) MAX phase formation, without the inclusion of additional elements such as Si and in some cases material that is > 85 % MAX phase. We hope that a full understanding of the mechanism of formation of Zr_3AlC_2 will allow the improvement in phase purity of a range of other MAX phases.

4:10 PM

(ICACC-S12-007-2020) Multifunctional cellular MAX phase architectures

J. Gonzalez-Julian*[1]; M. Belmonte[2]; P. Miranzo[3]; M. I. Osendi[3]; W. Araki[4]; J. Malzbender[1]; R. Vassen[1]

1. Forschungszentrum Juelich, Germany
2. Institute of Ceramics and Glass, CSIC, Spain
3. Institute of Ceramics and Glass, CSIC, Ceramics, Spain
4. Saitama University, Japan

Aluminum-based MAX phases are well known by their unique combination of properties, such as lightweight, high elastic modulus, outstanding oxidation and corrosion resistance, good damage tolerance, easily machinability, excellent creep resistance, and high thermal and electrical conductivities. However, most of these properties have been only measured for bulk MAX phases, and this combination of features is of high interest for their porous counterparts. Unfortunately, processing of MAX phase structures with controlled porosity (from 30 to 90 vol.%) is still rather limited despite of the high potential of these cellular architectures. Furthermore, these porous structures might contribute to find final application of MAX phases such as in volumetric solar receivers, burners, electrodes, and/or catalytic supports. In this work, cellular Cr_2AlC structures have been prepared by different methods to control the content (up to 80 vol.%) and the size (from nm to mm) of the porosity. The processing methods are: space holder technique, replica method, and robocasting - a suitable additive manufacturing technique for MAX phases. Processing will be explained in detail, as well as the oxidation, mechanical, electrical and thermal response of the developed cellular structures.

4:30 PM

(ICACC-S12-008-2020) Sol-gel based synthesis of selected MAX phases

C. Birkel*[1]; J. Siebert[1]

1. Arizona State University, School of Molecular Sciences, USA

MAX phases are typically prepared by high-temperature – oftentimes high-pressure – solid-state techniques. This can limit the doping with additional elements as well as the processability and accessibility of varying morphologies of the target materials. We have developed an alternative wet chemistry-based synthesis path to selected MAX phases. An aqueous gel that contains the metal ions and an excess of carbon is heated under argon initially forming metal oxides that undergo a carbothermal reduction as well as reaction with carbon to form the carbide phase. In the case of Cr2GaC, anisotropic, highly crystalline particles are formed. Additionally, the precursor can be deposited on a polymer hollow microsphere template leading to amorphous carbon spheres that are decorated with the MAX phase. This shows the enhanced processability using this sol-gel based approach that could lead to printability of MAX phases.

4:50 PM

(ICACC-S12-009-2020) Synthesis and Characterization of Novel PEEK-MAX and PEEK-MoAlB Composites

S. Javaid*[1]; S. Gupta[1]

1. University of North Dakota, Mechanical Engineering, USA

In this presentation, we report for the synthesis and characterization of PEEK-MAX and PEEK-MoAlB composites by hot pressing. Novel PEEK-Ti_3SiC_2, PEEK-Cr_2AlC, PEEK-Ti_3AlC_2, and PEEK-MoAlB compsoites were fabricated by hot pressing. The microstructure analysis, mechanical, and tribological behavior will be presented. In addition, the interaction of MAX phases with different microbes will be presented to document the potential antimicrobial behavior of PEEK-Composites. Finally, the potential biomedical behavior of these composites will be compared with other relevant composites.

S13: Development and Applications of Advanced Ceramics and Composites for Nuclear Fission and Fusion Energy Systems

Novel Ceramics and Composites for Nuclear Systems I

Room: Coquina Salon H

Session Chair: Young-Wook Kim, University of Seoul

1:30 PM

(ICACC-S13-001-2020) Composite Moderators for Advanced Nuclear Systems (Invited)

L. Snead*[1]; J. Trelewicz[1]; B. Cheng[1]; C. Ang[2]; N. Brown[2]

1. Stony Brook University, USA
2. University of Tennesse, Knoxville, USA

Following the original Chicago Pile, graphite became most studied nuclear material and de-facto moderator of high-temperature reactors, with historic alternatives being BeO, Be, and ZrH.

While graphite selection was driven by considerations of neutron economy, modern reactors are less neutronically constrained given higher enriched fuels. A question for reactor design is then, given the inherent life-limitation and safety issues posed by historic monolithic solid moderators, does it make performance/economic sense to develop composite moderators as safe lifetime reactor components? This paper presents initial development work of an ARPA-E MEITNER project. In the first phase of this processing work, two-phase moderators MgO-Be(x), MgO-BeO(x), SiC-Be(x), and MgO-ZrHx are pursued. Materials are processed via direct current sintering. For the consolidation of SiC and MgO matrix composites the team has taken advantage of the transient eutectoid processing of SiC (using refractory oxide sintering aids.) To suppress the MgO sintering temperature fugitive LiF flux is included in the MgO + Be or BeO powder mix, allowing fully dense compacts at temperature <1000°C. The moderating power of these materials is determined as a combination of the two-phase mixtures, which is quantified using computed X-ray micro-tomography. Results from the tomography directly informs neutronic analysis determining moderating power of development materials.

2:00 PM

(ICACC-S13-002-2020) Alternative Nuclear Fuel Materials by Rapid-Laser Chemical Vapor Deposition

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

1. Free Form Fibers, USA

Uranium-silicon compounds were produced via the rapid-laser chemical vapor deposition process by Free Form Fibers in collaboration with Materials Characterization Laboratory in Oak Ridge, Tennessee. Utilizing depleted uranium delivered via uranium hexafluoride, a series of uranium-silicon compositions were fabricated into fiber format with the goal of producing uranium disilicide (U_3Si_2). The chemical and crystalline nature of the formed fibers were evaluated by EDX, XRD, and TEM. The formation of uranium tetrafluoride solid crystals as a by-product of the gas-phase reaction was minimized through manipulation of the gas precursor composition and relative ratio mixtures. The demonstration of a R-LCVD approach to making this alternative nuclear fuel material is a significant development in providing alternate manufacturing pathways from powder metallurgical technology.

2:20 PM

(ICACC-S13-003-2020) Embedding Sensors in Additively Manufactured Silicon Carbide

C. Petrie*[1]; D. N. Leonard[1]; Y. Yang[1]; M. Trammel[1]; B. Jolly[1]; K. Terrani[1]

1. Oak Ridge National Laboratory, USA

Silicon carbide (SiC) is an attractive material for many nuclear applications due to its high-temperature strength retention, stability under neutron irradiation, reduced interaction with high-temperature steam, and low neutron absorption. Recent work has demonstrated the ability to fabricate monolithic SiC components with complex geometries using a binder jet additive manufacturing technique followed by densification using chemical vapor infiltration (CVI). This enabling technology could allow sensors to be embedded in critical locations within the component, provided that the sensors themselves can survive the CVI process. This presentation describes efforts to embed materials and sensors in additively manufactured SiC components. Material selection (primarily refractory metals) is guided by computational thermodynamics and is confirmed by embedding coupons and examining material interactions. The presentation covers the results of the analysis, sectioning and microscopy of the embedded materials, and the initial testing of functional sensors including thermocouples and optical fiber-based, spatially distributed temperature/strain sensors.

2:40 PM

(ICACC-S13-004-2020) Fabrication and Characterization of SiC_f/SiC Composites Made by Polymer Infiltration and Pyrolysis (PIP) Process

Q. Zhang*[1]; H. Liu[2]; P. Xiao[1]

1. University of Manchester, Department of Materials, United Kingdom
2. University of Manchester, United Kingdom

SiC_f/SiC composites are promising materials used as fuel cladding materials in light water reactors and other high temperature applications, due to their excellent high-temperature thermal stability and mechanical properties. In this study, we fabricated and characterized SiC_f/SiC composites using Polymer Infiltration and Pyrolysis (PIP) method. Fast fabrication of β-SiC matrix can be achieved after 10 polymer-infiltration-pyrolysis cycles, with open porosity reduced to 16%. Microstructure and composition of polymer-derived SiC_f/SiC composites are investigated using SEM, Raman, and XRD. Hardness and Young's module of SiC_f/SiC composites are analyzed using micro-indentation, and the cracking path during the 4-point bending test is observed as well. In addition, the high-temperature chemical stability of polymer-derived SiC_f/SiC composites has been studied by investigating the changes in microstructure, composition and micro-mechanical properties after serving in high temperature environment.

Radiation Damage, Defect Production, Evolutions, and Interactions

Room: Coquina Salon H
Session Chair: Lance Snead, Stony Brook University

3:20 PM

(ICACC-S13-005-2020) Radiation effects in fluorite dervative oxides within the Sc_2O_3: $(Zr,Hf)O_2$ systems (Invited)

M. K. Patel*[1]

1. University of Liverpool, Mechanical Materials and Aerospace, United Kingdom

The Sc_2O_3:ZrO_2 and Sc_2O_3:HfO_2 system consist of three phases that primarily crystallise in a rhombohedral symmetry with $A_4B_3O_{12}$, $A_2B_5O_{13}$, and $A_2B_7O_{17}$ stoichiometries and are designated as δ-, γ-, and β-phases respectively. These phases undergo an order-disorder (OD) transformation under ion irradiation or at high temperatures. The OD transformation is believed to occure via to formation of cation anti-sites. Computational studies have shown the δ-phase compunds have cation antisite formation energies lower than that of pyrochlores. Oxides derived from a parent fluorite structure have been the centre of interest in the nuclear materials community owing to their excellent radiation tolerance, flexibility to accommodate radioactive fission products and act as surrogates to understand evolution of nuclear fuel and waste. Some of these systems include, stoichiometric and non-stochiometric fluorites UO_2, CeO_2, PrO_2 and TbO_2; aliovalent cation doped mixed oxides like pyrochlores and bixbyites (A_2O_3). In comparison very little is knows about the crystallography and disordering processes in δ-, γ-, and β-phases and how they relate to these above mentioned fluorite related compounds. I will thus present a review of the research on radiation effects in these phases and discuss fundamental questions that stil remain unanswered to enhance our understanding of radation effects in complex oxides.

3:50 PM

(ICACC-S13-006-2020) Recovery of irradiation-induced defects in SiC: A pair distribution function analysis study

D. Sprouster*[1]; L. Snead[1]; T. Koyanagi[2]; Y. Katoh[2]; E. Dooryhee[3]

1. Stony Brook University, Materials Science and Chemical Engineering, USA
2. Oak Ridge National Laboratory, USA
3. Brookhaven National Laboratory, USA

We have recently shown that the Wigner energy of neutron irradiated SiC can be an issue, that is, the stored energy release of neutron irradiated SiC can exceed the specific heat depending on irradiation condition and heating rate. In this work, we present results dedicated to understanding and unraveling the atomic and microstructural origins of this energy release. Samples of highly-faulted poly-crystalline CVD 3C-SiC were irradiated in a mixed spectrum fission reactor near 60 Cin a fluence range from 5*1023 to 2*1026n/m2(E>0.1 MeV), or about 0.05e20 dpaWe have employed a combination of synchrotron-based X-ray diffraction (XRD) and pair distribution function (PDF) analysis to quantify the microstructural and atomic defects present both before and after annealing. Both XRD and PDF results show that prior to amorphization the atomic structure is significantly perturbed due to a large fraction of vacancies and interstitial defects. After anneling, the recovery of carbon vacancies is more dominant than silicon vacancies during annealing. The recovery of the different sub-lattices is highly correlated to the large stored energy release and highlight that the recovery of Carbon defects (both I and V-type) contributes to stored energy more than Silicon.

4:10 PM

(ICACC-S13-007-2020) In Situ Observation of Radiation Enhanced Diffusivity in Nanoparticle Ceramics

N. J. Madden[1]; J. A. Krogstad*[1]

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

Radiation induced grain growth requires reliable measurement of radiation enhanced diffusion (RED) as a function of material chemistry, temperature, and radiation dose, type and rate—yet experimental quantification of RED, especially for nonmetallic materials is both tedious and constrained. Here we present a new route to rapid quantification of RED through in situ observation of irradiated nanoparticles and the consolidation thereof. Specifically, we have measured the consolidation of nanoparticle cerium dioxide and yttria stabilized zirconia (YSZ), with a common fluorite crystal structure, across a range of particle sizes and degree of initial crystallinity. Using a two-particle sintering model we have extracted the effective diffusivity from this radiation induced consolidation process. Values for the ceria nanoparticles agree well with the extrapolated values for RED in ceria previously measured at high temperature in single crystal specimens, while no previous experimental data exists for YSZ. Moreover, we can also extract RED values as a function of temperature and document the transition between the sink-limited regime at lower temperatures, facilitated by diffusion of radiation induced defects to the free surfaces of the particles, to the recombination-limited regimes at intermediate temperatures before recovering the thermally activated diffusion behavior at high temperatures.

4:30 PM

(ICACC-S13-008-2020) Methodology for Irradiation Creep Testing of SiC / SiC Composite Cladding

P. A. Champlin*[1]; C. Petrie[1]; K. R. Smith[1]

1. Oak Ridge National Lab, Reactor and Nuclear Systems Division, USA

Irradiation creep compliance is a critical parameter for evaluating the stress evolution of silicon carbide (SiC) components during operation in various reactor concepts. Recent instrumented irradiation creep testing of SiC, performed at low dose in the Halden reactor, showed a creep compliance that is orders of magnitude higher than previous uninstrumented bend relaxation experiments performed in the High Flux Isotope Reactor (HFIR). This discrepancy illustrates the need for additional irradiation creep data for SiC. To this end, a novel irradiation capsule design has been developed to test miniature SiC tensile specimens under loading during irradiation in HFIR. The experiment uses the coolant pressure of HFIR to compress a bellows, which in turn applies the loading to the specimen. Finite element thermal and structural models have been developed to evaluate the temperature and stress distributions in the experiment, which show uniform values within the gauge region of the specimen. Work has also been performed to produce prototypes for the validation of these models. These results show that irradiation creep testing of SiC in HFIR is feasible and could provide a low-cost, high-throughput vehicle for gathering needed data.

4:50 PM

(ICACC-S13-009-2020) Radiation tolerance of stabilized alumina coatings: An in situ irradiation study

M. Vanazzi*[1]; F. Di Fonzo[1]; M. Li[2]

1. Center for Nano Science and Technology (CNST) - IIT, Italy
2. Argonne National Lab, USA

Innovative reactors require new material strategies. Recently, coatings have earned great interest, since they could tackle major issues like corrosion and erosion, gas permeation and fretting. In this framework, alumina coatings by IIT were characterized as corrosion-resistant and tested under heavy ion irradiation, being stable up to 150 dpa. The radiation tolerance was related to the amorphousness of the material, which crystallizes under irradiation. To preserve this tolerance and improve the range of operation, the amorphous matrix must be conserved. Here, the stabilization of alumina by doping is evaluated. Firstly, thermal stability tests are performed on the pure and doped materials. Then, irradiation studies are conducted with in situ TEM at the IVEM-Tandem facility (ANL). Samples are irradiated with different ions, up to 20 dpa. Experiments are carried out at 600 and 800 °C, to decouple the temperature contribution from the radiation-induced effects. Doped alumina retards the crystallization, even under irradiation. Doping stabilizes the metastable phases of alumina, preserving the previously observed radiation tolerance. Moreover, while pure alumina suffers from voids formation, no swelling appears in the doped counterpart. To conclude, the stabilization of nano-ceramic coatings has been proven by in situ ion irradiation tests.

S14: Crystalline Materials for Electrical, Optical and Medical Applications

New Direction

Room: Halifax A/B

Session Chairs: Tetsuo Tsuchiya, National Institute of Advanced Industrial Science and Technology (AIST); Minoru Osada, Nagoya University

1:30 PM

(ICACC-S14-001-2020) Rare earth free ceramic magnet with high energy for motor applications

P. K. Roy*[1]; D. Shekhawat[1]

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

In recent years, low or non-rare earth permanent magnets are under investigation for motor applications. In the permanent magnets, the strontium hexaferrite can find its place owing to its low price and moderate electro-magnetic properties. Wide range of strontium hexaferrites, have been developed, which are promising materials in this area. However, only a limited variety of strontium hexaferrites

are commercially available for the development of permanent magnets. In the present work, an effort has been made to improve the electro-magnetic properties of $SrAl_4Fe_8O_{19}$ hexaferrite, which can be utilized for motor applications. Cr–Sn substitution in $SrAl_4(Cr_{0.5}Sn_{0.5})_xFe_{8-x}O_{19}$ (where, x = 0.0, 0.2, 0.4, 0.6, and 0.8) hexaferrite shows that grains are in well-defined hexagonal platelet shape with clear boundaries. Bulk density and grain size of the ferrites are increased with increasing in Cr–Sn substitution. The relationship among the allocation of cations over the 5 sub-lattices of Fe^{+3} with substitutions is also analyzed. Electro-magnetic properties are maximized in $SrAl_4(Cr_{0.5}Sn_{0.5})_{0.6}Fe_{7.4}O_{19}$ composition. The maximum B_r and $(BH)_{max}$ was theoretically calculated about 7.62 kG and 7.60 MGOe, respectively for x=0.60 composition. Cr-Sn substituted Sr-hexa-ferrite can be proposed for application as a permanent magnet with a good deal of consistency.

1:50 PM

(ICACC-S14-002-2020) Sintering, Characterization and Evaluation of Ceramics Recycled From Waste Soda-Lime-Silica Glass and White Corn Cob Ash

B. Oji*[1]

1. Federal Polytechnic Ado-Ekiti, Glass and Ceramics Technology, Nigeria

This research examines the influence of soda-lime glass on the porosity, crystallization and phase transformation of ceramics produced from white corn cob ash. The Corn cobs (CCB) and soda-lime-silica glass (SLSG) are both agricultural and industrial wastes. The CCB decays emitting greenhouse gases that cumulate into global warming and the later being non-biodegradable occupies useful land fit for economic purpose. These wastes are however both sources of silica and can be recycled to produce piezoelectric based ceramics materials. The SLSG were crushed and sieved into 45μm grain sizes, while the corn cob was subjected to thermal combustion in open air to yield amorphous silica and then conditioned to 700°C for 6hrs before screening. A mixture of 98, 96, 94, and 92% of CCBA and 2, 4, 6, 8% of SLSG were mixed in a ball mill and then pressed into pellets using polyvinyl alcohol (PVA) as binder and then sintered at 900°C, 1000°C and 1200°C respectively. Microstructure analysis (SEM), XRD, XRF and FTIR were used to analyze the developed ceramic materials. The SEM result shows that, as the sintering temperature increases the porosity of the samples decreased. The XRD analysis also reveals a cristobalite dominant phase and small traces of tridymite phases. Fourier transform infrared spectroscope (FTIR) was used to analyze the chemical bond formed in the material.

2:10 PM

(ICACC-S14-003-2020) Grain Size Dependent Properties in Transparent Dense Nanocrystalline Ceramics (Invited)

J. Wollmershauser*[1]; B. Feigelson[1]; H. Ryou[1]; E. Gorzkowski[1]

1. U.S. Naval Research Laboratory, Materials Science & Technology Division, USA

Reducing the grain size of polycrystalline ceramics can increase the strength and hardness, as well as, enhance light transmission in birefringent ceramics. However, only a limited amount of experimental work exists which demonstrates dense nanocrystalline ceramics with grain sizes under 100 nanometers with porosity levels required for optical applications, i.e. < 0.01%. This talk will focus on some of NRL's work on synthesis and properties of nanocrystalline ceramics with grain sizes much smaller than 100 nanometers and negligible porosity. As the grain size is reduced well below 50 nanometers, traditional descriptions of bulk behaviors in polycrystalline materials appear to break down and new mechanisms are needed to explain the grain size dependent mechanical behavior. Specifically, increasing dissipative energy accommodation is observed below the break down point indicating a change in strain accommodation.

2:40 PM

(ICACC-S14-004-2020) Steady Non-classical Giant Electrostriction in Calcium Doped Cerium Oxide

A. Kabir*[1]; V. Esposito[2]

1. Technical University of Denmark, DTU Energy, Denmark
2. Technical University of Denmark, Denmark

In recent times, rare-earth-doped cerium oxides, e.g. Gd-doped ceria have shown giant electrostriction that are orders of magnitude higher than known inorganic electrostrictors. This non-classical electrostriction is fundamentally independent of Newnham″s scaling law and is functioned with the existence of oxygen vacancies (Vo) in the host lattice, associated with Ce-Vo pairs. Here, electro-chemo-mechanical properties of highly defective calcium doped ceria (CDC) ceramics, with variously doping levels ($Ce_{0.9}Ca_xO_{2-x}$, x = 0.025-0.15) are investigated. As expected, CDC samples display rather different electrochemical properties depending on a vacancy concentration and microstructure. They reveal primary creep at room temperature, emphasizing an unexpected anelasticity in ceria. The electrostrictive effect describes being independent of either the nominal vacancy concentration or the related blocking effects at the grain boundary, exhibiting a high electrostriction coefficient (M_{33}) in order of $\approx 10^{-18}$ m^2 V^{-2} in a wide range of frequency. Remarkably, electrostriction show neither strain saturation nor relaxation with field amplitude and frequencies, respectively. These key findings unveil a new form of electromechanical response in ceria that is independent of composition fluctuations and dominated by strong columbic and elastic energy of Ca^{2+}- Vo pairs.

Optical Material I

Room: Halifax A/B
Session Chairs: Yiquan Wu, Alfred University

3:20 PM

(ICACC-S14-005-2020) Nanocrystal Technology for New Electronic Applications (Invited)

M. Osada*[1]

1. Nagoya University, IMaSS, Japan

Nanocrystals are emerging as key materials due to their novel shape- and size-dependent physical/chemical properties that totally differ from their bulk counterparts. The main challenges in this field remain controlled synthesis and large-scale assembly of nanocrystals for materials and device fabrications. Here, we review our recent progress related to solution-based synthesis and assembly of oxide nanocrystals, including 2D nanosheets. An attractive aspect is that nanocrystalscan be organized into various nano-architecturesby applying solution-based assembly techniques involving electrostatic layer-by-layer assembly, Langmuir-Blodgett deposition and spin coating. We utilized nanocrystalsas building blocks in the solution-basedassembly, and successfully developed various functional nanodevices such as all-nanosheet FETs high-density capacitors, artificial ferroelectrics/multiferroics, spinelectronic devices, Li-ion batteries/solar cells, actuator crystals, etc. The virtually infinite varieties of oxide nanocrystals, which can be used to assemble various nanodevices, suggest that nanocrystaltechnology will contribute to striking progress in intelligent material processes and new electronic applications.

3:50 PM

(ICACC-S14-006-2020) Fast quenching in scintillators observed by transient absorption spectroscopy (Invited)

M. Koshimizu*[1]; Y. Muroya[2]; S. Yamashita[3]; H. Yamamoto[4]; T. Yanagida[5]; Y. Fujimoto[1]; K. Asai[1]

1. Tohoku University, Department of Applied Chemistry, Japan
2. Osaka University, Japan
3. University of Tokyo, Japan
4. National Institute for Quantum and Radiological Science and Technology, Japan
5. Nara Institute of Science and Technology, Japan

The development of novel scintillators with high light yields is of considerable interest. Most scintillators are composed of an insulator host with dopants used as the luminescent centers. In these cases, the energy of the ionizing radiation is initially deposited in the host, and some of the excitation energy in the host is subsequently transferred to the luminescent centers. By contrast, some scintillators exhibit efficient scintillation without such dopants; these are called self-activated scintillators. In self-activated scintillators, the energy of the ionizing radiation produces electronic excitations in the scintillators that sometimes result in scintillation. For both cases, only a fraction of the energy deposited by the ionizing radiation results in scintillation. With respect to the number of excited states or electron-hole pairs, some scintillators experience quenching. Among these processes, we have observed a fast quenching process of within 1 ns, using transient absorption spectroscopy based on pulsed electron beams. Quenching at such speeds has been observed for almost all the scintillators that we investigate. The contribution of fast quenching processes varies according to different scintillators.

4:20 PM

(ICACC-S14-007-2020) Development of crystalline scintillators for radiation detector applications (Invited)

T. Yanagida*[1]; N. Kawaguchi[1]

1. Nara Institute of Science and Technology, Japan

Scintillators are one of the key detector materials for ionizing radiation detectors, and up to now, most of scintillation materials have been a single crystal. Therefore, we have investigated crystalline scintillator materials for radiation detector applications. Eu-doped $LiCaAlF_6$ scintillator was developed for neutron detectors, and especially, it exhibited good spectroscopic and imaging capabilities. Ce-doped GAGG ($Gd_3(Al,Ga)_5O_{12}$) crystal has been applied for gamma-ray detectors, and it has shown remarkable scintillation light yield. Eu-doped SrI2 was also developed for gamma-ray spectroscopy, and we could achieve comparative scintillation properties with commercial SrI_2 by other groups even the Eu concentration was lower than them. Tl and Bi doped CsI was developed for X-ray detectors, and we successfully suppress the afterglow levels. In the conference, we will introduce these results.

4:50 PM

(ICACC-S14-008-2020) Growth and Scintillation Properties of Perovskite Single Crystals (Invited)

J. Pejchal*[1]; V. Babin[1]; M. Buryi[1]; J. Barta[2]; C. Guguschev[3]; E. Mihokova[1]; P. Prusa[2]; K. Rubesova[4]; V. Jakes[4]; P. Zemenova[1]; R. Kral[1]; A. Beitlerova[1]; R. Kucerkova[1]; M. Schulze[5]; M. Nikl[1]

1. Institute of Physics, Czech Academy of Sciences, Czechia
2. Faculty of Nuclear Sciences and Physical Engineering, Czech Technical University, Czechia
3. Leibniz Institute for Crystal Growth, Germany
4. University of Chemistry and Technology, Czechia
5. ScIDre Scientific Instruments Dresden Ltd., Germany

The $SrHfO_3$ (SHO) – based perovskite scintillators have been studied since 1990's. High efficiency and quite fast decay time were reported for Sr-deficient undoped SHO powders. Similar scintillation properties were found for the Sr-deficient lighter analogue, the strontium zirconate $SrZrO_3$ (SZO). Due to very high melting points (2730°C for SHO, 2646°C for SZO), all the research was conducted on powders or ceramics. Therefore, we prepared single crystalline samples of undoped Sr-deficient SHO and SZO using the optical floating zone method to perform a study on their luminescence and scintillation properties. The overall scintillation efficiency of SHO was comparable to the $Bi_4Ge_3O_{12}$ reference scintillating material, while that of SZO was considerably lower. The luminescence mechanism and scintillation properties of the SHO and SZO crystals will be presented and discussed together with the crystal growth and morphology. Rare-earth aluminates with perovskite structure represent another important group of promising scintillation materials, but due to difficulties in crystal growth, not much attention has been paid to them so far. We decided to study the $LaAlO_3$, whose growth is relatively easy and whose potential cannot be ruled out yet. Crystal growth by the micro-pulling-down method will be briefly reported together with luminescence and scintillation properties and interplay of the luminescence phenomena.

S15: 4th International Symposium on Additive Manufacturing and 3-D Printing Technologies

Design and Characterization

Room: Coquina Salon B
Session Chair: Dileep Singh, Argonne National Lab

1:30 PM

(ICACC-S15-001-2020) Developments and Challenges Related to Thermal Process Modeling of Metallic Laser Powder Bed Fusion to Advance Certification of Flight Hardware (Invited)

J. Fody*[1]; C. Lang[1]

1. NASA Langley Research Center, USA

Metallic laser powder bed fusion additive manufacturing (AM) is being considered by the aerospace community to reduce cost and expand design space for structures. Certification standards are being written to address variability in builds resulting from poorly understood cause and effects in the AM process. With a focus on individual parts and witness coupons, large data sets and factors of safety are specified and costly and time consuming post processing and inspections are required. A better understanding of the processes, mechanisms, and effects of defects would help relax such requirements. Accordingly, NASA's is working under the Transformational Tools and Technologies project to develop computational and experimental capabilities for a next generation certification paradigm. The computational capabilities target a better understanding of the mechanisms causing residual stress distribution and part deformation, and the process - microstructure - part performance relationship including the formation and effect of defects. Fundamentally, a physically correct AM process thermal model is needed. This talk will discuss challenges related to development and validation of such a thermal process model, accomplishments, and advances required for these goals.

2:00 PM

(ICACC-S15-002-2020) Design approaches of multifunctional ceramic architectures produced by additive manufacturing

M. Pelanconi*[1]; A. Ortona[1]

1. SUPSI, MEMTi, Switzerland

The strategy of designing a component has always been influenced by its manufacturing. The advent of Additive Manufacturing (AM) has allowed conceiving components by their function and no longer by their manufacture. This great benefit allows improving the components' performances and the fabrication of geometrically complex parts such as cellular structures. Through this approach

the layout of these structures is the result of a design and simulation approach; therefore, the design method is fundamental. In this work, we present the design approaches of regular and irregular strut-based lattices and triply periodic minimal surface-based structures. Moreover, we propose a novel design method of multifunctional cellular architectures for additive manufacturing. The method enables to generate structures with morphological variations that provide a component with different features and functionalities in its own volume, depending on its requirements.

2:20 PM

(ICACC-S15-003-2020) Materials research and measurement needs for additive manufacturing of ceramic materials

A. J. Allen*[1]; I. Levin[1]

1. NIST, Materials Measurement Science Division, USA

The conclusions of a NIST materials research & measurement needs workshop for ceramic additive manufacturing (AM), held in November 2019, will be discussed. Following a NIST-led discussion panel at last year's ICACC2019 that identified several potential areas for development, the workshop brought together leading experts from early industrial adopters of ceramics AM, AM-equipment and feedstock manufacturers, government agencies, and academia. The goals were to discuss the most promising broad-impact applications of ceramics AM and identify the associated materials measurement needs which, if addressed, would accelerate the incorporation of AM methods into commercially viable ceramic manufacturing technologies. The opportunities and challenges for ceramics AM in key market sectors were critically reviewed with discussions focused on formulating materials research directions to bridge critical knowledge gaps presently hindering a broader deployment of ceramics AM technologies. While AM of dense ceramic products may remain a long-term challenge, the situation is different for green-body fabrication or for porous materials, where a significant AM presence already exists. This will become more important with better controlled processes and materials process characterization. Highlights of the workshop and the potential cross-cutting research efforts it identified will be presented.

2:40 PM

(ICACC-S15-004-2020) A comparative study of mechanical behavior of ceramics prepared by different additive manufacturing techniques

P. Tumurugoti*[1]; K. McGarrity[1]; K. Ning[1]; H. Shulman[1]

1. Alfred University, USA

Fabrication of ceramics with complex shapes and geometries by additive manufacturing (AM) has been a subject of great interest. Recent developments in a variety of ceramic AM platforms may allow for improvements in part quality. There is a need for a better understanding of the quality of 3D printed ceramics, and how the properties compare to ceramics fabricated via traditional processing methods. In this work, the mechanical properties and fracture behavior of structural ceramics such as alumina and silicon nitride prepared by different AM platforms – stereolithography, binder jetting, material jetting and robocasting – are investigated. Mechanical strength, determined by four-point bend or biaxial testing, and fracture behavior, will be presented. The effects of printing parameters and build-orientation on the generation of flaws/fracture origins are analyzed. Further, microscopic characterization and challenges with AM fabrication of complex shaped parts are discussed.

Binder Jetting and Powder Bed Fusion

Room: Coquina Salon B

Session Chair: Joshua Fody, NASA Langley Research Center

3:20 PM

(ICACC-S15-005-2020) Hydrothermal-assisted Powder Bed Fusion of Ceramics for Achieving High Green Density

X. Song*[1]; F. Fei[1]

1. University of Iowa, Mechanical and Industrial Engineering, USA

Ceramic additive manufacturing (AM) provides a freeform fabrication method for creating complex ceramic structures that have been extremely difficult to build by traditional manufacturing processes. However, ceramic structures made by AM processes usually exhibit a relatively low density, which is largely due to the use of a large amount of organic binder in shaping green bodies. In this research, we present a new ceramic AM process, named hydrothermal-assisted powder bed fusion (HPBF), which utilizes a water-based hydrothermal mechanism to fuse particles, eliminating the use of binders in forming green bodies. A prototype system for the proposed HPBF process is introduced. The effects of process parameters (such as layer thickness, printing passes, prepressing and final pressing pressure, temperature) on the properties of achieved green parts are investigated. Experimental results indicate that with optimized process parameters, HPBF can achieve 3D ceramic green parts with a high density up to 90%.

3:40 PM

(ICACC-S15-006-2020) Progress on 3D-printing of high-purity and crystalline silicon carbide

K. Terrani*[1]; B. Jolly[1]; M. Trammel[1]; D. Richardson[1]; A. Schumacher[1]

1. Oak Ridge National Laboratory, USA

While additive manufacturing is now routinely used for polymers and metals, its application to ceramics has been limited to primarily oxide systems. A novel method for additive manufacturing of refractory ceramics has been developed at Oak Ridge National Laboratory that allows for realization of full 3D geometric complexity. Furthermore, the process is capable of yielding fully crystalline microstructure free of any additives and second phase impurities. The specific example of the process discussed is 3D printing of silicon carbide resulting in >90% theoretical density parts manufactured at ~1000°C. This process is highly flexible and extendable to other refractory ceramic systems and is expected to be disruptive in our approach in manufacturing and deploying ceramic components for a wide range of applications.

4:00 PM

(ICACC-S15-007-2020) Model-guided Irregular Powder Mixing for Feedstock Preparation in Ceramic Binder Jetting

D. Singh*[1]; M. Du[1]; M. Singh[2]

1. Argonne National Lab, USA
2. Ohio Aerospace Institute, USA

A limitation of binder jetting additive manufacturing is the low density of fabricated parts. Mixing powders with different sizes is promising to increase powder bed packing density and, hence, printed part density. In previous studies, mixed powder feedstock was prepared by trial and error method. In this research, linear packing model was proposed to model the packing density of the binary and ternary powder mixtures. Modified interaction functions were introduced to model irregular powders and compare the performance with the original model. To validate the model, three different-sized silicon carbide powders were mixed and their tap densities were measured. Powder bed packing density was also measured by a commercial binder jetting printer. Results showed that the model could predict the tap density of the powder mixtures with high accuracy. The trend of actual powder bed density agrees

well with the prediction. This model can guide the selection of powder fraction to increase the powder bed density and printed part density in the ceramic binder jetting process. This work was sponsored by Solar Energy Technology Office of the U. S. Department of Energy (DOE) under Contract No. DE-AC02-06CH11357 at the Argonne National Laboratory, managed by UChicago Argonne LLC.

Multi-Material and Hybrid Printing

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

4:20 PM

(ICACC-S15-008-2020) Ceramic Additive Manufacturing Methods Applied to Sintered Glass Components with Novel Properties

J. Schilm*[1]; T. Moritz[2]; E. Schwarzer[2]; K. Waetzig[1]; D. Wagner[1]; S. Weingarten[2]; A. Michaelis[3]

1. Fraunhofer IKTS, Materials and components, Germany
2. Fraunhofer IKTS, Processes/Components, Germany
3. Fraunhofer IKTS, Germany

Additive Manufacturing (AM) processes for polymers, ceramics and metals have become increasingly important in recent years for the manufacture of near-net-shape components. In contrast, the use of glass for this technology has so far only been studied in isolated cases. AM processes for glass components either comprise a direct melt extrusion limiting the shape of the glass components comparable to conventional melt derived glass shaping technologies or stereolithography 3D printing and sintering of fused silica powders. Here the possibilities are demonstrated to adapt powder based AM processes for µm-sized standard glass powders and for the production of complex near-netshaped shaped glass components. Special attention is paid to the sintering behaviour of glass powders driven by the viscous flow of the melt, which affects achievable sintering densities and contour accuracy. The integration of photoluminescent particles into the sintered glass matrices is used as an example to present the principle of functionalisation and hybridisation of glass. A significant afterglow behavior of sintered components has been realized by amounts as low as 15 Vol-% of the particles. Furthermore, the possibilities of co-sintering different adjusted suspensions and feedstock for the realization of multicomponent components will be demonstrated by the Ceram T3DP and der Ceram VPP technologies.

4:40 PM

(ICACC-S15-009-2020) Thermal Properties of 3D Printed Multi Materials

P. A. Warkentien*[1]; M. Singh[2]; M. C. Halbig[3]; H. Leonard[4]; A. Salem[5]

1. OSGC/Lorain County Community College, USA
2. Ohio Aerospace Institute, USA
3. NASA Glenn Research Center, USA
4. Rochester Institute of Technology, Mechanical Engineering, USA
5. Washington University in St. Louis, USA

Additive manufacturing/3D printing is becoming more important to industry, due to its relative ease of use and the ability to make production changes quickly. A variety of filaments are being created to fill specific needs in respective industries. Because of this, it is necessary to understand how printed materials will stand up in their environment/application. NASA is especially interested in thermal management and the evaluation of new materials is a critical part of that. The purpose of this project is to evaluate thermal properties of materials used within FDM-based 3D printing, and the effect of different processing variables on thermal diffusivity, thermal conductivity, and specific heat, along with the effect of thermal testing methods. Samples were printed both of different materials, and materials in bi-layer configurations and their thermal properties were evaluated. In this presentation, different aspects of both pre and post printing variables and their effect on thermal properties will be discussed.

S17: Advanced Ceramic Materials and Processing for Photonics and Energy

Advanced and Nanostructured Materials for Photonics, Electronics and Sensing I

Room: Tomoka C
Session Chair: Oomman Varghese, University of Houston

1:30 PM

(ICACC-S17-001-2020) Photogenerated catalytic processes of reduced graphene oxide decorated with V_2O_5 and WO_3 (Invited)

G. Fanchini*[1]

1. University of Western Ontario, Physics and Astronomy, Canada

Reduced graphene oxide (rGO) has attracted significant attention in photocatalysis. Enhanced photocatalytic degradation of methylene blue (MB) in water has been recently shown in the presence of rGO and vanadium pentoxide nanowires and nanocomposites (NW-V_2O_5) with the splitting of H_2O. The solid-state counterparts of these processes, for example involving polyvinil alcohol (PVA), are very little known, however, although they may have important applications in thin-film photocatalysis as well as the fabrication of low-cost organic photochromic thin films, in which the reversible discolouring of incorporated MB play a critical role. In this work, near-field scanning optical microscopy (NSOM) have been used to monitor the evolution of these processes at the nanoscale. MB nanoparticles are formed on top of rGO/NW-V_2O_5. Our NSOM study shows that both rGO and V_2O_5 are playing an essential role in achieving the reversible discolouring of MB nanopartcles, which is size-dependent, occurring at times ~5-20 min, and shorter for smaller particles size. In tmhe last part of our talk we will show that a similar system, reduced graphene oxide decorated with WO_3 nanowires, the photocatalytic activity of nanowire functionalized rGO can be extended to decompose polyciclic hydrocarbons, with dramatic advantages to eliminate these pollutants frequently associated to the oil and energy industry.

2:00 PM

(ICACC-S17-002-2020) Pulsed laser deposition of metal-insulator transition materials (Invited)

M. Chaker*[1]

1. INRS, Energie materiaux télécommunications, Canada

Current trend towards the development of smart materials with enhanced switching properties may be a relevant approach for realizing switching and/or tunable devices. In this context, transition metal oxides present potential electrical and optical properties making them very promising for many applications in electronic and optical devices. Among these materials, vanadium dioxide (VO_2) is particularly interesting because of its sharp reversible Metal-to-Insulator Transition (MIT) that occurs near room temperature ($T_{MIT} \approx 68°C$). This MIT is associated with a structural transition from a high-temperature tetragonal phase to a low-temperature monoclinic phase, and is characterized by significant changes of electrical and optical properties in response to external stimuli such as temperature, electric field and/or optical control signal. In this presentation, our recent achievements on the synthesis and characterization of undoped and doped vanadium dioxide thin films will be reviewed. We will also report some of their applications to photonic and smart radiator devices.

2:30 PM

(ICACC-S17-003-2020) Fabrication and Application of Polycrystal Silver Nanowire Transparent conductive film by SDGs-oriented Organic Precursor Splay Painting Reduction method (Invited)

Y. Hayashi*[1]

1. Tohoku University, School of Engineering, Japan

Silver nanowire transparent conductive film is one of alternatives to indium tin oxide films in the application of flexible touch panels. Generally, polyol method, which is performed in liquid phase, is used as a synthetic method of silver nanowires. However silver nanowires are some problems on synthetic conditions, processes, and waste emission. To improve these problems, a novel method, painting and subsequent reduction of organic precursor, is proposed in this study. In this method, needle-shaped organic precursor was fabricated by SDGs process, and then silver nanowire is simply obtained by splaying and reducing them to metallic silver with retaining their needle-shaped morphology. By optimizing painting and reducing conditions of the precursor, preparation of highly transparent and conductive polycrystal silver nanowire transparent conductive film could be expected. Transparent conductive film fabricated by using these materials showed about under 10 Ohm/sq. of resistivity and over 80 % of transparency, relative high performance. In addition, it doesn't require high temperature condition when reducing the precursor, that is, it can be applicable to low heat resistance materials, such as PET, PC, and so on.

Advanced and Nanostructured Materials for Photonics, Electronics and Sensing II

Room: Tomoka C

Session Chairs: Giovanni Fanchini, University of Western Ontario; Mohamed Chaker, INRS

3:20 PM

(ICACC-S17-004-2020) Effect of ligand selection on the grain growth of CZTSSe and graphitic carbon layers in thin film PVs (Invited)

C. Luscombe*[1]

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

One approach of fabricating thin-film PVs is through the deposition of solution-processed nanocrystal (NC) inks. These inks are comprised of inorganic NCs suspended and solubilized by organic coordinating ligands to form a printable solution. While these ligands used in the solution-based synthesis are critical for depositing the inks, post-thermal treatments of the NC films reveal that the ligand selection influences NC coarsening and carbon distribution in the final film. In this work, we synthesize metal-chalcogenide Cu_2ZnSnS_4 (CZTS) NCs using ligands of varying bond saturation to tailor the morphology and properties of annealed NC films. During NC synthesis, we find that ligands with unsaturated bonds cross-link with the chalcogenide source (elemental sulfur) to form a polysulfide, which caps and stabilizes the CZTS NCs. When selecting a ligand with two unsaturated bonds (Linoleylamine) a higher molecular weight polysulfide will form when reacting with sulfur, compared to a ligand with only one unsaturated bond (Oleylamine). Our results reveal that higher molecular weight polysulfides assist with more pronounced grain growth of CZTS upon annealing in an inert, selenium-rich environment.

3:50 PM

(ICACC-S17-005-2020) Oxide-chalcogenide heterojunctions for solar energy applications (Invited)

O. K. Varghese*[1]

1. University of Houston, Department of Physics, USA

Oxides and chalcogenides are two classes of materials used in a wide range of commercial applications. While chalcogenide semiconductors are used as light absorbers in solar cells, oxide semiconductors are used there for purposes like charge transport and charge blocking. A variety of binary and multinary oxide semiconductors are known to be promising for fuel generation processes such as solar photoelectrochemical water splitting also. With the advent of low dimensional structures, especially 2D layers, chalcogenides such as MoS_2 have become highly attractive for the technologies for water splitting. Our recent studies showed that heterojunctions of certain oxides and chalcogenides could enhance the performance of solar cells and solar fuel generation devices. This presentation will provide an overview of the oxide-chalcogenide heterostructures used for solar energy conversion. The focus of the discussion will be on the performance of solar cells and photoelectrochemical cells employing oxide-chalcogenide heterojunctions.

4:20 PM

(ICACC-S17-006-2020) Tuning the charge transport properties in semiconducting nanomaterials for solar energy harvesting devices

D. Benetti*[1]; H. Zhao[4]; A. Vomiero[3]; F. Rosei[2]

1. INRS, Energy, Materials and Telecommunication, Canada
2. INRS, Canada
3. Lulea University of Technology, Engineering Sciences & Mathematics, Sweden
4. Qingdao University, College of physics, China

To reduce our dependence on fossil fuels and control the greenhouse emissions, it is necessary to transit to more sustainable sources of energy. This challenge may be addressed by the so-called 3rd generation solar cells such as Dye Sensitized Solar Cells (DSSCs), Quantum Dots Solar Cells (QDSCs) and Perovskite Solar Cells (PSCs). In the last decade, these devices have rapidly improved in terms of photoconversion efficiency and became one of the most cost-effective alternatives to the commercial available silicon cells, due to simple, low-cost materials and fabrication process. In this work, we explore how semiconducting nanomaterials interact with each other and how, by tuning the interfaces of nanomaterials, we can control proprieties, such as charge injection and light absorption, in order to improve the final performances of the devices.

4:40 PM

(ICACC-S17-007-2020) Structure, Optical and Electronic properties of chemically engineered $BiFeO_3$ thin films for PV applications (Invited)

P. Machado[1]; I. Caño[1]; C. Escudero[2]; M. Tallarida[2]; M. Coll*[1]

1. ICMAB-CSIC, Superconducting Materials and Large Scale Nanostructures, Spain
2. ALBA Synchrotron, Spain

The use of ferroelectric perovskite oxides as a stable photoactive layer has opened up a ground-breaking new arena of research. They present an alternative mechanism for solar energy conversion that could surpass the fundamental efficiency limits of conventional semiconductors. One of the biggest challenges so far is to reduce their band gap toward the visible region while simultaneously retaining ferroelectricity. To address these two issues, we perform elemental composition engineering of $BiFeO_3$ as a means to tune the characteristics of the cation-oxygen bonds and gain new insights on its influence on the structure, optical and electronic properties by means of X-ray diffraction, spectroscopic ellipsometry, photoemission and X-ray absorption spectroscopy. We demonstrate by chemical deposition techniques the formation of epitaxial

pure-phase and stable co-substituted $BiFeO_3$ films. Importantly, the band gap can be tuned from 2.7 to 2.3 eV upon transition metal substitution (cobalt) while enhancing ferroelectricity. A-site substitution by lanthanides can further modify the optical absorption while increasing film reproducibility at high Co loads. Finally non-optimized all-oxide vertical devices have been fabricated and the electrical photoresponse in the visible region is improved with respect to the pristine $BiFeO_3$ films.

5:10 PM

(ICACC-S17-008-2020) Surface-Engineered 2D Hexagonal Boron Nitride Nanosheets for Energy Applications (Invited)

H. Zarrin*[1]; J. Kaur[1]

1. Ryerson Univeristy, Chemical Engineering, Canada

In the modern era of smart wearable electronics and compact transportation systems, there is an urge for the development of solid, lightweight, flexible and efficient energy-storage devices. For realizing this, novel solid electrolyte with high energy and power density are demanding. The recent developments in 2D nanomaterials, including graphene, have exhibited promise for a wide range of applications. In particular, 2D hexagonal boron nitride (2D-hBN) or "white graphene" is an isomorph of graphene with similar layered structure in a hexagonal lattice, which is uniquely featured by its opto-electrical properties, mechanical robustness, and thermal stability. Due to these and more importantly the electrical-insulating behavior, in this project we used 2D-hBNs as promising solid electrolytes for next generation energy-storage and -conversion applications including fuel cells, batteries and supercapacitors. Solvent-assisted exfoliation method was employed to simultaneously produce few-layered hBNs and surface-engineer it with ion-conductive groups. The augmentation of hBNs significantly improved their ion-conductivity and electrochemical stability. Significant enhancement in both of in-plane and through-plane ion-conductivity was observed at different operating conditions (e.g., 0.34 S/cm at 25°C and 50% relative humidity) which was 14 times higher than that of commercial Nafion membranes.

5:40 PM

(ICACC-S17-009-2020) Sr/$MgCl_2$-Carbon/Graphene oxide composites with high structural stability as robust ammonia carriers (Invited)

F. Akhtar*[1]; Z. Cao[1]

1. Lulea University of Technology, Department of Engineering Sciences and Mathematics, Sweden

Chemisorbents, such as halide salts, holds potential as ammonia carriers for energy storage and environmental applications. The chemisorbents require structuring and tailoring of adsorptive properties to optimize for economical storage and delivery of ammonia. We will present novel carbon/Graphene oxide foams-Sr/$MgCl_2$ composites as robust ammonia carriers, with graphite (Gt) and graphene nanoplatelets aggregates (GNA) and graphene oxide foams as additives and carrier, respectively, to $SrCl_2$ and $MgCl_2$. The structured composites manifested high structural stability above the melting temperature of Sr/$MgCl_2$ with 95% mass retention. Furthermore, these composites demonstrated rapid ammonia sorption and desorption kinetics, due to the enhanced surface area and creation of additional microporosity. Our results demonstrated that GNA-Sr/$MgCl_2$ composites presented 80% faster kinetics in ammonia sorption and 73% faster in the first-2-minutes of desorption compared to the pure $MgCl_2$. The enhancement of both structural stability and sorption kinetics makes these novel composite robust ammonia carriers. Furthermore, a concept of physorbent-chemisorbent composite will be introduced as a feasible way to achieve a structural-stable ammonia carrier for automobile selective catalytic reduction (SCR) systems.

Tuesday, January 28, 2020

4th Pacific Rim Engineering Ceramics Summit

Challenges and Opportunities for Ceramic Technologies I

Room: Coquina Salon E
Session Chair: Young-Wook Kim, University of Seoul

8:30 AM

(ICACC-PACRIM-001-2020) Challenges and opportunities for various ceramic technologies (Invited)

A. K. Bakshi*[1]

1. Morgan Advanced Materials, USA

Ceramic components are used in many challenging applications, such as semiconductor fabrication, aerospace engines, medical implants and oil well drilling. As operating conditions in these applications become increasingly demanding, they pose new challenges to the ceramics being used. In the semiconductor fabrication chambers, stringent resistivity control, thermal stability and corrosion resistance of the ceramic fixtures are paramount to making faster chips. In aerospace industry, as jet engines run hotter, they put more stress on joints in the ceramic sensors compromising their reliability. In the medical field, as implantable devices such as defibrillators are miniaturized, defects in ceramics are exacerbated leading into premature failure of the implants in the body. Similarly, in deep well drilling for oil and gas exploration, traditional zirconia ceramics may not survive the higher temperature and corrosive atmospheres. Nevertheless, these challenges open tremendous opportunities for new ceramic technologies in the industry. In this topic, we will discuss the challenges and opportunities for ceramics in the above applications.

9:00 AM

(ICACC-PACRIM-002-2020) Silicon Nitride: A summary of new findings for biomedical applications (Invited)

D. J. Bray*[1]; R. M. Bock[1]; B. J. McEntire[1]; G. Pezzotti[2]

1. SINTX Technologies, USA
2. Kyoto Institute of Technology, Japan

Silicon Nitride, while first synthesized in the 1850's, didn't find industrial applications until a hundred years later. Today, silicon nitride materials and applications represent a multi-billion-dollar industry, becoming a key component in electronics, turbo machinery, bearings, and even orbital satellites. One of the most recent applications for this unique material is as a biomaterial for orthopedic implants. Silicon nitride exhibits a strength comparable to titanium alloys, does not induce a foreign body response when implanted, bonds directly to bone, demonstrates antibacterial properties, and can be imaged artifact-free using all standard medical modalities. These promising findings led to the development of a silicon nitride well-suited for implantable medical device applications. When silicon nitride is immersed in the biologic environment, production of silicic acid and ammonia from a slow surface hydrolysis reaction enhances healing of soft and osseous tissue, inhibits bacterial proliferation, and inactivates viruses. These benefits permit it to be used in a wide array of disciplines inside and outside of the human body including orthopedics, dentistry, virology, agronomy, and environmental remediation. This paper will summarize new key findings related to the mechanisms of enhanced osteogenic response and antibacterial characteristics and discuss some of the new applications for silicon nitride.

9:30 AM

(ICACC-PACRIM-003-2020) Easy conversion process of titanium surface covered with passive film into functional surface (Invited)

T. Ishikawa*[1]; K. Tsujikura[1]

1. Tokyo University of Science, Yamaguchi, Applied Chemistry, Japan

Surface-functionalization of titanium metal is of great interest, especially as the conversion of the surface passive film into objective functional film can be widely used in bio- and environmental applications. However, since the surface passive film is very stable, the functionalization (such as conversion into photocatalytic anatase-TiO_2) has been performed using relatively complicated processes e.g. hydrothermal reaction and anodic oxidation. This is because direct oxidation of titanium metal only leads to thermodynamic formation of octahedral-rutile-TiO_2 in the entire temperature region, while chemical synthesis using precursors can easily produce tetrahedral-anatase-TiO_2 at 600ºC or less. Here, we report a simple process for the direct formation of photocatalytic anatase-TiO_2 on titanium by simple oxidation. In the first step in our process, titanium is treated with a reducing agent to create a surface titaniumhydride layer. After that, the treated titanium covered with titaniumhydride is immerged in aqueous silica; subsequent calcination at 900ºC (Specific temperature) in air effectively generates the surface anatase-TiO_2 layer. This is because the covered tetrahedral-silica stabilizes the anatase structure that is formed. The covered silica is easily removed by quenching in water to reveal the photocatalytic surface layer composed of anatase-TiO_2

10:20 AM

(ICACC-PACRIM-004-2020) Current research on coupled electronic and atomic effects in ceramics (Invited)

Y. Zhang*[1]; W. J. Weber[2]

1. Oak Ridge National Lab, USA
2. University of Tennessee, Materials Science & Engineering, USA

Controlling non-equilibrium energy transfer processes among electrons and atoms to synthesize materials with unique properties and novel functionalities is a grand challenge. The response of ceramics to energy deposition from energetic ions is inherently connected with a simultaneous disturbance of the electronic and atomic structures. The effects of extreme ionization can have a first order effect on the kinetics of atomic processes in many ionic-covalent materials, affecting evolution of microstructure, phase changes and thermodynamic properties. The coupled effects of complex synergistic, additive, or competitive (annealing) interactions between inelastic energy deposition and pre-existing or evolving defects can occur, often exhibiting a threshold on magnitude of the electronic energy loss. Understanding such coupled effects will allow prediction of complex non-equilibrium defect processes in ceramics and lead to improved predictive models and new approaches to functionalize ceramic films and structures. This work was support by U.S. DOE, BES, MSED.

10:50 AM

(ICACC-PACRIM-005-2020) Advanced Ceramics Industry in Japan and Introduction of JFCA Activities (Invited)

H. Takemura*[1]

1. Japan Fine Ceramics Association, Japan

Japan Fine Ceramics Association (JFCA) has 101 members including major Japanese manufacturers and users of advanced ceramics and foreign subsidiaries in Japan. According to an industrial trend survey, the total domestic production of advanced ceramics in Japan reached approximately $ 30 billion in 2018. To meet our members' needs, we strive to develop the basic technologies for the future of advanced ceramics and offer a variety of projects which include developing international standards, hosting seminars, technical visits and developing industrial roadmaps. In this presentation, we introduce about our activities on R&D, Advanced Coating Alliance, CMC Consortium, High Reliability Study Working Group, Optical Ceramics Study Working Group, international liaison with USACA and PEC and ISO Fine Ceramics international standards development. Concerning the international standards development, JFCA serves as the international secretariat for ISO/TC206 of Fine Ceramics.

11:20 AM

(ICACC-PACRIM-006-2020) Biomaterial with bioactive and antimicrobial function (Invited)

G. Turri*[1]; H. Muto[2]; T. Noshiro[2]

1. Namiki Precision of Europe SA, Switzerland
2. Adamant Namiki Precision Jewel Co., Ltd., Advanced R&D Dept, Japan

Our company contributes to societal changes by designing only-one products and combining our core and newest technologies. These include high-performance ceramic products and ultra precision machining techniques. In recent years, we have succeeded in developing bioactive zirconia using an original manufacturing method. The strength of our porous-bioactive ceramic is outstanding, comparable to that of conventional zirconia. At the same time, thanks to the controllable porosity level, its density is only a fraction, making it the perfect material for bio-implants. Adamant-Namiki can also produce porous zirconia with high strength and high rigidity combined with porous ceramic. This material has the structural features of the human tissue and can create a more human-compatible material. We report the updated results on the "Zirconia Disc for Dental," which is one of our products, with very distinctive characteristics.

Applications of Engineering and Functional Ceramics

Room: Coquina Salon E

Session Chairs: Jingyang Wang, Shenyang National Laboratory for Materials Science, Institute of Metal Research, Chinese Academy of Sciences; Miki Inada, Kyushu University

1:30 PM

(ICACC-PACRIM-007-2020) Ceramics Marking and Drilling by Lasers (Invited)

S. Jiang*[1]

1. AdValue Photonics Inc, USA

The advanced ceramic marking and drilling process by using lasers will be presented.

2:00 PM

(ICACC-PACRIM-008-2020) Defected nano-cerium oxide: Biomedical properties (Invited)

S. Seal*[1]

1. University of Central Florida, Advanced Materials Processing and Analysis Center (AMPAC), Materials Science & Engineering, Nanoscience Technology Center (NSTC), Biionix cluster, College of Medicine, USA

Cerium is a rare earth metal with two different oxidation states, 3+ and 4+, which differ by the single occupation of a 4f orbital. Creation of an oxygen vacancy by removing a neutral oxygen atom leads to localization of two electrons over 4f states and thereby Ce^{4+} reduction to Ce^{3+} (e.g. $CeO_{2-x} + O_2$à $2xCe^{3+} + xO_{Vac.}$), with a thermodynamically stable structure. Oxygen vacancy formation occurs more readily for nanoceria (relative to bulk), with equilibrium vacancy concentrations being sensitive to particle size, morphology, surface facet distribution (e.g. [100] show lowest vacancy site formation energies), surface termination/coordinate species (e.g. hydroxyl, hydrate), and synthesis conditions. Enzyme-mimetic reactions have been observed for nanoceria, especially towards radical oxygen species, due to redox cycling at these surface

sites, leading to wide-spread application in biomedical sciences. We have evaluated the anti-oxidant behavior and biocompatibility of nanoceria for application in spinal cord repair, protection of retina, anti-cancer, neurodegeneration, diabetic wound healing and radiation protection. Beyond simple redox chemistry, the influence of environmental oxygen partial pressure on ceria oxygen uptake-release has been demonstrated in (pro-) angiogenesis studies as well as the influence of local pH in cancer environments.

2:30 PM

(ICACC-PACRIM-009-2020) Effects of Texture and Silica Content on Crack Growth of Boron Nitrides for Electric Propulsion (Invited)

J. Salem*[1]; J. Mackey[1]; H. Kamahwi[1]

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

High-power solar electric propulsion is a key technology for Human exploration of space. One control system being considered involves Hall thrusters. Historically, Hall thruster discharge channels have used various grades of hot pressed hexagonal boron nitride (BN). This study investigates the fracture toughness and slow crack growth parameters of several commercially available BN grades. Prior work revealed that some grades of BN absorb water and change strength, thus presenting a philosophical conundrum on crack growth test methods. The grades selected for this study are of interest because their available billet size is sufficient for Hall thruster discharge channels, or because they have heritage in Hall thruster applications. Ultimately, the data will be used to perform material selection, system design and reliability analyses. Boron nitrides exhibit limited slow crack growth in humidity, with an orientation dependence due to crystallographic texture: orientations with van der Waal bonds are insensitive to water whereas those with covalent bonds are sensitive. Fracture toughness is low, around that of glass, and is also a function of orientation (bond type), with fracture occurring more readily along van der Waal bonds between hexagonal BN platelets than at covalent bonds across platelets. Silica additions toughen BN, but increase slow crack growth susceptibility.

3:20 PM

(ICACC-PACRIM-010-2020) Additively Manufactured Motors for Electrified Aircraft (Invited)

M. C. Halbig*[1]

1. NASA Glenn Research Center, USA

Electric motors are becoming a greater focus within the aeronautics missions and programs of the National Aeronautics and Space Administration (NASA). There is a shift occurring from solely turbine engine propulsion to hybrid-electric and all electric propulsion for commercial air transportation. The NASA activity entitled Compact Additively Manufactured Innovative Electric Motor (CAMIEM) had the goal of utilizing additive manufacturing methods to achieve new motor designs that have significantly higher power densities as well as other performance and manufacturing benefits to provide reduced weight, emissions, costs, fabrication time, and energy/fuel consumption. New electric motor components to include rotors, housings, stator-cooling rings, a wire embedded stator, and direct printed stators were designed and fabricated using additive manufacturing processes. The results of component fabrication and of testing motor configurations against a baseline electric motor will be presented. Also, systems studies were conducted to evaluate the benefits to be gained from higher performance motors to include reduced motor and aircraft weights, reduced energy demands, and increased range for urban air mobility and single aisle aircraft.

3:50 PM

(ICACC-PACRIM-011-2020) Multifunctional Ceramics La_2ZnMnO_6 for magnetic and electric applications (Invited)

D. K. Mahato*[1]; D. N. Singh[1]

1. National Institute of Technology Patna (NITP), Physics, India

During the past few decades there have been significant advances in fabricating functional ceramics and their significant developments in innovational research, microwave telecommunications, the emergence of low temperature co-fired ceramic technology and commercialization. Functional ceramics play essential roles in an ever-increasing extent of the functioning of manufactured products. Different classes of multifunctional double perovskite ceramics have gained much scientific interest owing to their rich physics, industrial and technological applications. This functional property can be explored for intelligent sensor, spintronic and functional device applications. In this work, we report the dielectric, electrical and magnetic properties of rare earth based double perovskite ceramics host Zn and Mn. The study of cyclic voltammetry charging discharging plot exhibit its use in supercapacitor and solar cells. It is observed that ac conductivity obeys power law at high frequency region as explained by various models. The present structural, dielectric and magnetic data make this system for its multiferroic character a promising candidate to various modern functional device applications. The impedance spectroscopy studies disclose different conduction processes at the grain and grain boundary. M-H and FC-ZFC vs temperature behaviour and sensing properties of the present material will also be discussed.

4:20 PM

(ICACC-PACRIM-012-2020) Influence of calcium phosphate ceramic substrate surface properties on biological cell invasion (Invited)

A. Leriche*[1]; M. Lasgorceix[1]; S. Chamary[1]; N. Somers[1]; J. Hornez[1]; L. Boilet[2]; S. Hocquet[2]; F. J. Cambier[2]; A. Daskalova[3]

1. Universite Polytechnique Hauts de France, LMCPA, France
2. Belgian Ceramic Research Centre, Belgium
3. Bulgarian Academy of Sciences, Institute of Electronics, Bulgaria

This study highlights the effects of surface quality of bone substitutes based on β-tricalcium phosphate on cellular invasion properties. Previous studies comparing the cell proliferation and invasion inside macroporous scaffolds prepared from different routes: PMMA beads replica, freeze casting and microstereolithography have shown the importance of macroporosity morphology and size and the evidence of some alignment of the cells along the layer border generated by the stereolithography. In order to study more precisely the influence of the surface topography on the biological response, the β-tricalcium phosphate dense surface was modified by femtosecond laser irradiation to produce grooves 3 μm deep, 15 μm wide and spaced by 100 μm with different designs. The increase of the wettability with the micro-patterning, compared to smooth surfaces, was highlighted. An improvement of the osteoblastic proliferation was also demonstrated. Finally, the tendency of cell elongation along the grooves direction has shown the ability of osteoblastic cells to adapt their morphology to the support topography on which they grow and the positive effect on invasion rate.

9th Global Young Investigator Forum

Novel Ceramic Processing Methods and Synthesis Routes

Room: Coquina Salon G
Session Chair: Giorgia Franchin, University of Padova

8:30 AM

(ICACC-GYIF-009-2020) Role of Graphene on the Mechanical, Structural, and Electrical Changes in Silicon Oxycarbide Ceramics

E. A. Barrios*[1]; R. Kuliiev[2]; N. Orlovskaya[2]; L. Zhai[3]

1. University of Central Florida, Materials Science and Engineering, USA
2. University of Central Florida, Mechanical and Aerospace Engineering, USA
3. University of Central Florida, NanoScience Technology Center and Department of Chemistry, USA

Polymer derived ceramics (PDCs) are ceramics synthesized from polymeric precursors and have been recently under investigation for the development of next generation energy materials. Silicon oxycarbide (SiOC) is one type of PDC that has harbored many of these research efforts due to its ease of accessibility and processability, high thermal and chemical stability, and tailorable semiconducting properties. Typically, SiOC ceramics consist of a disordered matrix of SiOC with randomly dispersed graphitic carbon domains. While these domains are said to be highly conductive, their lack of percolation often leads to less than desirable electrical properties. In this work, highly connected graphene networks have been added to the SiOC matrix in order to create a lightweight, conductive ceramic composite. The effects of graphene on the mechanical, structural, and electrical properties of this system will be explored and discussed.

8:50 AM

(ICACC-GYIF-010-2020) First-principles thermodynamics calculations of thermal properties for environmental barrier coatings and their oxidation byproducts

C. Bodenschatz*[1]; G. Costa[2]; N. S. Jacobson[1]; C. W. Bauschlicher[3]; D. L. Myers[4]

1. NASA Glenn Research Center, USA
2. Vantage Partners, LLC, USA
3. NASA Ames Research Center, USA
4. East Central University, Chemistry, USA

A large effort is underway to replace metal-based components in the hot section of turbine engines with ceramic matrix composites (CMCs). CMCs have higher temperature capabilities and lower density, which can lead to improved fuel efficiency in aircraft engines. However, silicon-based CMCs are prone to oxidation, so protective environmental barrier coatings (EBCs) are deposited on the CMC. However, EBC design is challenging due to the large number of candidate materials and a lack of understanding of the interactions between the EBC and the engine environment. To aid in EBC design, we are developing a database of thermodynamic properties for candidate EBCs and their corrosion byproducts via experimental and computational methods. First-principles thermodynamics is a useful computational tool which can potentially reduce the need for experiments and decrease the time and cost for coating development. We present thermodynamics properties calculated from density functional theory (DFT) for EBC candidate materials and compare these with experimental results. Additionally, we calculate thermodynamic properties possible gas-phase corrosion products from EBC reactions with calcium-magnesium-aluminosilicates (CMAS). These methods will aid in the screening of EBC candidate materials and enhance the design of EBCs for high-temperature turbine engine use.

9:10 AM

(ICACC-GYIF-011-2020) Novel Processing of Directionally Porous Sintered Barium Titanate Ceramics

J. John*[1]; R. Parai[1]; S. Akurati[1]; D. Ghosh[1]

1. Old Dominion University, Mechanical and Aerospace Engineering, USA

Ice-templating is an emerging technique which enables the synthesis of directionally porous functional ceramics and multilayered ceramic-polymer composites. Properties of ceramic-polymer dielectric composites can be enhanced by improving the connectivity of ceramic phase in the composites. Due to the low pore tortuosity, ice-templated ceramics can be easily infiltrated and thus provides a novel method for improving three-dimensional connectivity of ceramic phase in dielectric composites. Also, through the variations of material and process variables, composition and morphology of ice-templated composites can be adjusted. Toward this end, this work will present results on the processing of directionally porous sintered barium titanate ceramics employing novel ice-templating technique. To develop barium titanate based composites, it is important to understand the tailorability of porosity, grain size and structure that can be achieved in ice-templating technique with respect to the particle size of as-received barium titanate powder. This work will discuss the effects of starting ceramic powder particle size, composition of aqueous ceramic suspension, unidirectional freezing condition, and sintering temperature on the development of ice-templated macroporous barium titanate ceramics. The results will provide valuable insights of the processing-microstructure relationships.

9:30 AM

(ICACC-GYIF-012-2020) Engineering of eco-friendly spinel-periclase refractories for RH degasser steelmaking (Invited)

S. Mandal*[1]; C. Kumar[3]; D. Kumar[2]

1. University of California, Irvine, Materials Science and Engineering, USA
2. Indian Institute of Technology, Department of Ceramic Engineering, India
3. TRL Krosaki Refractories Limited, India

Despite health hazards, magnesia-chrome refractory bricks are widely used in Ruhrstahl Heraeus (RH) degasser which is an equipment for making vacuum-treated steel for railway track, lightweight automobile and transformer core. As an eco-friendly alternative, spinel-periclase refractory was engineered to enhance density, high temperature strength and corrosion resistance. Optimized formulation using 14 wt% in situ spinel, Mg-rich spinel, fused and sintered magnesia along with meticulously selected mixing, pressing and sintering strategies reduced porosity below 13% and gave hot modulus of rupture (MOR) comparable to magnesia-chrome bricks. Zirconia and titania were added to engineer the microstructure, and ensuing phase evolution and the impact on compressive strength, hot MOR, thermal expansion, thermal shock resistance and RH slag corrosion resistance was evaluated. SEM-EDS of thermal shocked and corroded refractories gave insights into failure mechanism. Though the additives resist corrosion locally, larger pore size offset the benefit, even when total porosity was constant. Results lead to a direction towards development of eco-friendly alternatives for RH degasser.

Careers in Science, Technology, Engineering and Mathematics (STEM)

Room: Coquina Salon G
Session Chair: Giorgia Franchin, University of Padova

10:20 AM

(ICACC-GYIF-013-2020) Opportunities and Challenges for Advancing the Next Generation of Armor Ceramics (Invited)

L. Vargas*[1]

1. CCDC Army Research Laboratory, Physics of Soldier Protection to Defeat Evolving Theats, USA

High performance boride- and carbide-based ceramics have revolutionized dismounted Soldier protection systems due to their unique blend of properties (compressive strength, hardness) and their extremely low densities. Over the last few decades, the advent of innovative processing techniques and the sourcing and synthesis of high quality materials have yielded significant performance improvements to the ceramics for these body armor systems. However, with increasing need for lighter Soldier burden and improved performance, there is a need to continue to drive toward enabling disruptive capability in both materials and materials design. For the past three years, the researchers at CCDC ARL have been pushing the envelope for 1) development of novel ceramic materials and composites, 2) advancement of experimental characterization techniques, and 3) innovative methods for processing dense, mesostructured ceramics. The purpose of this presentation is to discuss areas where community participation is highly valued and ultimately necessary to achieve shared materials research goals. In addition, a portion of the presentation will be devoted to discussion on the advantages and merits (and of course challenges) of a career as a scientist/engineer in the public service.

10:50 AM

(ICACC-GYIF-014-2020) Careers in STEM: Bridging the Gender Divide (Invited)

P. Singh*[1]

1. Centre for Policy Research, Department of Science and Technology, India

This article attempts to identify the drivers and barriers for inclusion of women in STEM careers and suggest policy measures. This study adopts in-person, telephonic interviews and review of literature as its methods. It is well established that scientific advancements and technological innovation enhances economic competitiveness and growth of a nation that depends upon technically advanced human resources based on STEM. There is a huge demand of lucrative careers in STEM but whether these demands are equally filled by all the sections of the society? It is found that there is huge gender divide in terms of representation of women in STEM careers. Less then 30 percent of world's women are researchers in STEM. This study suggests drivers for inclusion of women in STEM being enormous career opportunities, high growth prospects, professional stability, feeling of pride and high wages. The barriers being lack of education and training amongst women in STEM, community and gender roles, family background, work pressure and inappropriate learning environment. The study concludes that with appropriate policy interventions by improving the dropout ratios of women in STEM at schools and universities; enhancements in fellowships for women; free training programs; awareness programs for STEM careers; opening more day care facilities for working mothers, may remove such hindrances and bring equity in STEM careers for women.

Advanced and Nanostructured Materials

Room: Coquina Salon G
Session Chairs: Daniele Benetti, Institut National de la Recherche Scientifique; Rebekah Webster, University of Virginia

1:30 PM

(ICACC-GYIF-015-2020) Cerium oxide nanoparticles abrogate therapy-induced tumor relapse via non-redox mechanisms (Invited)

F. Corsi*[1]; S. Briganti[2]; F. Capradossi[3]; S. Licoccia[1]; E. Traversa[4]; L. Ghibelli[3]

1. University of Rome "Tor Vergata", Chemical Science and Technologies, Italy
2. San Gallicano Dermatological Institute IRCCS, Italy
3. University of Rome Tor Vergata, Biology, Italy
4. King Abdullah University of Science and Technology, China

Chemotherapy is the standard care for many advanced carcinomas, effectively reducing tumor mass; however, treated patients eventually undergo fatal relapse due to cancer repopulation and acquired chemoresistance (CRAC). CRAC occurs because cells forced to die by the treatment release bioactive signals allowing surviving cancer cells to i) repopulate the injured tissue via the prostaglandinE2 (PGE_2)-mediated "phoenix rising" pathway, and ii) acquire resistance to future therapy cycles via still unknown mediators. Cerium oxide nanoparticles (CNPs) are receiving much attention in biomedicine for their antioxidant properties, promising to act as potent anticancer drugs, modulating the redox status of tumor microenvironment. Here, we show that CNPs efficiently counteract therapy-induced repopulation inhibiting PGE_2 synthesis in prostate cancer; moreover, CNPs prevent acquired chemoresistance by inhibiting leukotriene production, helping unveiling the mechanism of acquired resistance. Surprisingly, Sm-doped CNPs, devoid of redox activity, exert similar anti-CRAC effects, showing these occur via novel non-redox mechanisms, which we are now working to clarify. Our results imply that CNPs may represent an invaluable tool to help i) clarifying the mechanism leading to therapy resistance and ii) achieving a CRAC-free chemotherapy, i.e., combining efficient tumor reduction with durable remission.

2:00 PM

(ICACC-GYIF-016-2020) Physico-chemical and biological properties of Ce, Mg, Sr and Zn (0.5 – 10 at.%) substituted hydroxyapatite nanopowders

M. Chirica*[1]; T. Tite[1]; I. Pasuk[1]; A. Kuncser[1]; S. Iconaru[1]; D. Predoi[1]; A. Popa[1]; G. Stan[1]; L. Albulescu[2]; G. Manda[2]; C. Tanase[2]; S. Nita[3]

1. National Institute of Materials Physics, Romania
2. "Victor Babes" National Institute of Pathology, Romania
3. National Institute for Chemical Pharmaceutical Research and Development, Romania

The biomedical use of hydroxyapatite (HA), a bioceramic capable to promote new bone formation in certain conditions, is often limited by its slow osteointegration rate and absent antibacterial activity. The most recent findings suggested that ion-substitution in HA could pave the road towards superior bioceramics with improved biological performances, able to generate a strong biointerface with the host tissue. In congruence with this view, series of HA materials, single substituted with Ce, Mg, Sr and Zn ($0.005<\{x_{Ce}, x_{Mg}, x_{Sr}, x_{Zn}\}<0.1$), were synthesized at room-temperature. The work tackled in a systematic manner the multi-parametric physico-chemical characterization and biological performance of such doped HAs. Thereby, the comparative influence of the doping cation on the physical (e.g. crystallinity, grain size) and biological (e.g. cation release rate, biocompatibility, angiogenic/osteogenic capability, ROS reduction) of the nano-powders were investigated by TEM, EDXS, XRD, FTIR, ICP-MS and a sequence of dedicated in vitro assays. While the inter-dependence of the type and concentration of the dopants – crystallinity – ion-release profile – biofunctionality is unveiled, the opportunity to advance a new generation of bioceramics with controlled degradation and designed therapeutic effects for temporary bone regeneration scaffolds becomes feasible.

2:20 PM

(ICACC-GYIF-017-2020) $La_{0.6}Sr_{0.4}Fe_{0.8}Mn_{0.2}O_{3-\delta}$-based anode for SOFC applications: New insights on redox-stability and catalytic activity (Invited)

L. Duranti*[1]; I. Natali Sora[2]; F. Zurlo[1]; I. Luisetto[3]; S. Licoccia[1]; E. Di Bartolomeo[1]

1. University di Roma Tor Vergata, Department of Chemical Sciences and Technologies, Italy
2. University of Bergamo, Dept. of Engineering and Applied Science, Italy
3. ENEA C.R. Casaccia DTE-PCU-IPSE, Department of Energy Technologies, Italy

Perovskite oxides such as ferrites have been widely investigated for SOFC applications for their remarkable electrochemical activity as electrodes. However, their phase instability in reducing conditions still remains an issue for anode application. The role of Mn substitution into B-site of $La_{0.6}Sr_{0.4}FeO_3$ (LSF) perovskite oxide was investigated. New insights on the structural evolution of $La_{0.6}Sr_{0.4}Fe_{0.8}Mn_{0.2}O_{3-\delta}$ (LSFMn) perovskite oxide in different reducing conditions were revealed. Specifically, Mn-doping seems to promote structural stability, reducing the oxygen vacancy concentration and driving the formation of Fe nanoparticles as highlighted by XRD diffraction measurements. Redox-cycles were investigated and properties of the re-oxidized compound were highlighted. Finally, different strategies to provide the anodic compartment with Ni, to promote the oxidation of methane and ethanol-based fuels avoiding the formation of carbonaceous deposits, were evaluated.

2:50 PM

(ICACC-GYIF-018-2020) La_2O_3-doped alumina as active catalytic support in CH_4 combustion

M. M. Trandafir*[1]; S. Neatu[1]; F. Neatu[1]; A. Stanoiu[1]; O. Florea[1]; C. Simion[1]; C. Cobianu[2]; M. Gheorghe[2]; L. Leonat[1]; M. Florea[1]

1. National Institute of Materials Physics, Romania
2. Nanom-Mems, Romania

Complete oxidation of carbon-based fossil fuel is one of the highest scientific challenges aiming the reduction of polluting gases released in the atmosphere from the automotive and industrial chemistry based on internal combustion engines. The catalysts used for the oxidation of the fossil fuel are expensive and consists of Pd supported on high specific area metal oxide with oxygen storage capabilities able to provide oxygen during the fuel rich regime.[1] In the last two decades, research was focused on developing novel generations of noble metal-free catalysts. The catalytic support plays an important role in this process, for instance high oxygen mobility oxides as CeO_2 was used as a host for different oxides of transition metals dopants as MnO_x, composites which shown excellent catalytic behavior in the low temperature combustion of CO and soot.[2] This study presents the synthesis and characterization of La_2O_3-doped alumina supported ceria–manganese mixed oxides, prepared by coprecipitation, impregnation or citrate method. The materials were characterized by XRD, BET surface area, UV-Vis, XPS, SEM, and H_2-TPR. CH_4 catalytic combustion was evaluated on the self-heated catalytic micro-converter and the best results were obtained for CeO_2-MnO_x/La_2O_3-Al_2O_3 catalyst, prepared by impregnation.

3:30 PM

(ICACC-GYIF-019-2020) Solution-Processed P-type Copper Thiocyanate (CuSCN) Enhanced Sensitivity of PbS-QDs Based Photodiode (Invited)

I. Ka*[1]

1. INRS, emt, Canada

Lead sulphide (PbS) quantum dots (QDs) have become one of the most attractive material for optoelectronic applications. They have been demonstrated to be suitable candidates for broadband light sensing thanks to their tunable band gap from UV to near infrared (NIR). Recent reports have shown that reducing the dark current in PbS-QDs based photodetector could lead to higher performance as it minimises the noise sources in the photodetector. Here, to reach this goal we appropriately implement a solution-processed low-cost P-type copper thiocyanate (CuSCN) as hole transporter in our devices. We demonstrate that depositing the CuSCN layer prior to the evaporation of the metal electrode (silver or gold) results in the reduction of the dark current, regardless of the electrode type. Consequently, the on–off ratio together with the detectivity in the NIR of the photodiodes have been improved from 6 to 200 and 10^9 to 10^{11} $cmHz^{1/2}W^{-1}$, respectively. Interestingly, the response time of the photodiode which is in the range of tens of microsecond is not affected by the addition of the CuSCN layer. Our ability to engineer the interface between the PbS-QDs layer and the metal electrode by introducing CuSCN thin film, which is compatible with most of the current technologies, establishes an alternative way for enhancing the performance of the PbS-QDs based photodiodes.

4:00 PM

(ICACC-GYIF-020-2020) 0D / 2D Hetero-structured nanomaterials for high efficient optoelectronic devices

F. Li*[1]; M. Zhang[1]; L. Shi[4]; D. Benetti[1]; Q. Wei[2]; F. Rosei[3]

1. Institut National de la Recherche Scientifique, Énergie Matériaux Télécommunications, Canada
2. University of Jinan, China
3. INRS, Canada
4. INRS, EMT, Canada

Photoelectrochemical (PEC) hydrogen (H_2) production appears to be a prominent and suitable route to address in part worldwide energy and climate challenges. The use of a 0D/2D instead of a 3D material, can increase the efficiency of the PEC system due to the formation of an intimate interface between the two semiconductors. In this way, a preferential electrical transport is created that can suppress charge carrier recombination and overall improve the photoelectrocatalytic properties. Herein, heterojunctions of "green" ZnCuInS quantum dots (QDs)/Zn doped molybdenum disulfide (Zn/MoS_2) nanosheets (NSs) have been synthesized and used as photoanodes for water splitting application. This material combination can leverage the strong light harvesting capability of QDs and catalytic performance of MoS_2 simultaneously. The synergistic effect between 0D/2D heterojunctions, broad absorption of QDs, zinc coordinating sites of MoS_2 NSs, as well as the strong coupling and band alignment between them, lead to superior visible-light-driven PEC and photocatalytic performance. This work can provide a new platform to construct multifunctional 0D/2D nanocomposites for a large variety of opto-electronic applications, not limited only to photoelectrochemical devices.

4:20 PM

(ICACC-GYIF-021-2020) Undergraduate Research: Design of Novel Materials from Ag-based Precursors

A. Miles*[1]; C. Matzke[1]; D. Gerard[1]; R. Riihinen[1]; N. Johnson[1]; S. Gupta[1]

1. University of North Dakota, Mechanical Engineering, USA

Recently, National Academy of Engineering (NAE) has come up with 14 Grand Challenges. Different schools are developing Global Challenge Scholar's Program (GCSP) to incorporate some of the key elements of grand challenge in the educational program. Some of the important elements of the GCSP program are: "(a) A creative learning experience connected to the Grand Challenges such as research or design projects, (b) Authentic experiential learning with clients and mentors that includes interdisciplinary experience in fields such as public policy, business, law, medicine, ethics, and communications, (c) Entrepreneurship and innovation, (d) Global and cross-cultural perspectives, and (e) Development of social consciousness through service-learning "[1]. In this paper, we will present the recent progress in undergraduate (UG) research for designing novel Agriculture based materials from waste precursors

like Corn, Wheat Straw, and Sugar Beet etc. As a part of this study, we will also review different types of Ag-based materials like hydrogels, composites etc. during this research study. Reference: http://www.engineeringchallenges.org/File.aspx?id=15680&v=c29105cb

4:40 PM

(ICACC-GYIF-022-2020) "Green"Cu doped Zn-In-Se quantum dots for sustainable liquid luminescent solar concentrators

X. Liu*[1]; B. Luo[2]; J. Liu[1]; D. J. Luo[2]; D. Benetti[1]; F. Rosei[1]

1. University du Quebec, Institut National de la Recherche Scientifique, Centre - Energie Materiaux Telecommunications, Canada
2. Xi'an Jiaotong University, International Research Center for Renewable Energy & State Key Laboratory of Multiphase Flow in Power Engineering, China

Luminescent solar concentrators (LSCs) have gathered much attention as an effective technology to reduce the cost of integrated photovoltaic cells. Colloidal quantum dots (QDs) have shown to be promising candidates as a new type of absorber/emitter in LSCs. However, the majority of QDs-based LSC use QDs containing heavy metals (Pb, Cd), which limits the potential large scale use of LSCs. Here, we present the synthesis of green Cu-doped ZnInSe QDs and their use as luminophores to realize eco-friendly liquid and polymer LSCs. By tailoring the amount of Cu doping, absorption spectra and PL emission can be tuned over the complete visible spectral window, an enlarged stokes shift can be also achieved. By further surface passivation by ZnS shell, the QDs exhibited a quantum yield of more than 60%, making them feasible for LSC application. Under optimal conditions of doping and QDs concentration, an eco-friendly QDs-based liquid LSC shows high optical efficiency over 3.5%. This result is over two folds higher than that of polymer LSC (η_{opt}=1.75%, G=10). These results represent a promising direction to use liquid LSCs as a low-cost and sustainable solar energy harvester system, due to their recyclable waveguides, and ease to interchange and tune the concentrations of the various luminophores. Furthermore, it can provide a platform that will allow future researchers to optimize LSC applications.

S1: Mechanical Behavior and Performance of Ceramics & Composites

Processing-Microstructure-Mechanical Properties Correlation I

Room: Coquina Salon D
Session Chairs: Walter Krenkel, University of Bayreuth; Michael Jenkins, Bothell Engineering and Science Technologies

8:30 AM

(ICACC-S1-012-2020) Development of SiC/SiC ceramic matrix composites using a combination of PIP and LSI processes

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1. DLR - German Aerospace Center, Institute of Structures and Design, Germany
2. University of Augsburg, Institute of Materials Resource Management, Germany

Silicon carbide fiber-reinforced silicon carbide matrix composites (SiC/SiC) are promising candidates for components in the hot gas section of jet engines, as they exhibit high temperature resistance and low density compared to their metal alloy counterparts. SiC/SiC ceramic matrix composites were manufactured by polymer infiltration and pyrolysis (PIP) process using a polycarbosilane precursor and Hi-Nicalon Type S fibers. Generally, the processing of SiC/SiC, produced solely by PIP route, is rather time-consuming and the composites show a certain residual porosity. In order to obtain a dense matrix and to reduce the processing time, an additional liquid silicon infiltration (LSI) step is carried out after a reduced number of polymer infiltration and pyrolysis cycles. After PIP a phenolic resin is infiltrated as the first step, followed by pyrolysis and reactive melt infiltration with liquid silicon or silicon alloys. To protect the fibers during the LSI process, a CVD BN/SiC/PyC fiber coating was applied. Microstructural analysis and fiber degradation were examined using SEM and CT analysis. Mechanical tests were carried out to determine the strength and to assess the damage tolerance of the material. The overall process time has been successfully reduced by up to 50 %.

8:50 AM

(ICACC-S1-013-2020) Development of non-oxide ceramic matrix composites for application in advanced gas turbine

H. Klemm*[1]; C. Steinborn[1]; K. Schönfeld[1]; A. Michaelis[2]

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2. Fraunhofer IKTS, Germany

Ceramic matrix composites (CMC) offer a high potential for applications as structural parts in advanced gas turbines. During recent years, significant progress in material development of oxide and non-oxide CMC has been achieved, however, there are still considerable deficits especially in the long-term behavior of the materials at elevated temperatures. Besides the development of CMC with superior corrosion resistance in hot gas atmospheres, the mechanical behavior of CMC with high damage tolerance crack was found to be one of the most important challenges for these applications in aero engines. In the present study SiC/SiCN composites were fabricated by a precursor infiltration and pyrolysis process (PIP). Several matrix systems with and without fiber coating were used in order to achieve a flaw tolerant behavior. The composites were analyzed regarding their mechanical properties. Special emphasis was placed on the crack formation and propagation behavior in correlation to the microstructural features of the composites. Finally some idea about the design of flaw tolerant CMC will be provided.

9:10 AM

(ICACC-S1-014-2020) Processing and properties evaluation of long and nano carbon fibers reinforced SiC-based hybrid composites

M. Shaik*[1]; S. P[2]; M. Kolan[3]; A. Khanra[4]; B. Saha[1]

1. ARCI, Centre for Non Oxide Ceramics, India
2. ARCI, Centre for Engineered Coatings, India
3. Shanghai Jiao Tong University, School of Materials Science and Engineering, China
4. National Institute of Technology (NIT), Metallurgical and Materials Engineering, India

Carbon fiber (C_f) is considered to be one of the best reinforcing materials for composites due to its superior thermal, mechanical and electrical properties. In this study, a novel approach was adopted for the fabrication of Boron nitride coated continuous carbon fibers (BN-C_f) reinforced C_f/SiC-CNFs hybrid composite tubes. Long carbon fibers with specific weaving pattern were incorporated in the spray granulated premixed carbon nanofibers (CNFs) containing SiC powder. Green tubes were shaped by using cold isostatic pressing (CIP) which was further pressureless sintered to obtain highly dense composite tubes of theoretical density more than 95%. High speed nanoindentation was performed to measure the local mechanical properties of the composites. In addition, the physical and mechanical properties of the hybrid composite tubes were evaluated and compared using advanced high-speed nanoindentation mapping. The mechanical properties of hybrid composite tubes were found to improve with the carbon fibers reinforcement. Microstructure analysis of the composites, interface bonding of matrix between BN coated long C_f and other secondary phases were also studied using XRD, FTIR, SEM, and TEM to analyze the structure-property correlation.

9:30 AM

(ICACC-S1-015-2020) High Thermal Conductivity and High Mechanical Strength of Pressureless Sintered Silicon Nitride Ceramics with Rare-Earth Oxide Additives

J. Kong*[1]; W. Jung[1]; H. Ma[1]; D. Kim[1]

1. Korea Advanced Institute of Science and Engineering (KAIST), Material Science and Engineering, Republic of Korea

In recent years, the electronic devices require higher voltage, higher current, and greater power density. The high power consumption results in large thermal stresses, which can be overcome by the ceramic substrates with high thermal conductivity and high mechanical strength. However, most of researches and industrial companies used gas pressure sintering method which contributes to expensive production costs to fabricate silicon nitride substrates. In this study, the Si_3N_4 ceramics with high thermal conductivity and high mechanical strength were pressureless sintered with MgO, Y_2O_3, Yb_2O_3, La_2O_3, and SiO_2 sintering additives. The sintering schedule was optimized to achieve high density and the microstructure was examined to compare the thermal and mechanical properties of sintered samples using various sintering additive systems. In order to investigate the factors affecting the thermal conductivity, the distribution of the secondary phase in grain boundary was evaluated.

10:10 AM

(ICACC-S1-016-2020) Novel Ceramic/Metal Brake Disks

W. Krenkel*[1]

1. University of Bayreuth, Germany

Full-ceramic LSI-derived C/C-SiC brake disks have proven their outstanding tribological properties in high performance brake systems for automotive applications since many years. With the increasing replacement of combustion engines by electric engines, the requirements on the brake systems moves from a service to an emergency brake system, i.e. the material of the brake disk and pads have to withstand extreme conditions only few times. These emergency brake disks should be as light and small as possible as they belong to the unsprung mass. Additionally, the costs for an emergency system should be much lower compared to the current status of C/C-SiC brake disks. A new concept of a lightweight and cost-efficient ceramic/metal brake disk, comprising an aluminum load-bearing disk, lined with short-fiber reinforced C/C-SiC composite segments, is presented. A prototype was designed on the basis of a thermal finite element analysis and different joining methods to integrate the ceramic composite into the metallic substructure were examined. The hybrid ceramic/metal component was manufactured and tested on a dynamometer to evaluate the tribological properties and the structural integrity of the system. The relationships between the frictional behavior (CoF, wear), the temperature profile in the aluminum load bearing structure and the ceramic linings as well as the bonding strengths of the ceramic/metal joints are discussed.

10:30 AM

(ICACC-S1-017-2020) Tribological and mechanical behavior of 45S5 Bioglass®-based compositions containing alumina and strontium

M. S. Araujo*[1]; J. F. Bartolomé[2]; A. C. Da Silva[1]; S. R. Mello-Castanho[1]

1. Energy and Nuclear Research Institute, Center of Materials Science and Technology, Brazil
2. Consejo Superior de Investigaciones Científicas (CSIC), Spain

Although bioactive glasses have been widely used for surfaces of orthopedic and dental implants its limited mechanical strength, low toughness and wear resistance has prevented their use as load bearing devices. Considering that in suchlike material even a small variation in the composition can deeply modify its features leading to very different physico-chemical or mechanical properties, the present research was conducted by modifying the glass network of 45S5 Bioglass® by adding Al_2O_3 and SrO in order to obtain a glass high bioactive and with better mechanical and tribological performance for biomedical applications. The addition of 2 mol% of Al_2O_3 and 2 mol% of SrO produced a dense material with elastic modulus around 50 GPa same as 45S5. Moreover, the bending strength increased 60% and toughness doubled. On the other hand, the wear rate obtained against steel was found to be three times lower than 45S5. Additionally, biocide test performed against gram-negative bacteria E. coli indicates that antimicrobial efficacy was enhanced. Also, biological evaluation from SBF test showed higher bioactivity. From the results it can be assumed that both alumina and strontium in a synergetic way play a crucial role in mechanical and tribological properties.

10:50 AM

(ICACC-S1-018-2020) Experimental study on carbon fiber reinforced SiC subjected to the unpenetrated impact and post-impact mechanical behavior

W. Hu*[1]

1. Northwestern Polytechnical University, School of aeronautics, China

In this paper, the impact behavior of 2D-C/SiC at different velocities was investigated by experimental method. The specimens were impacted at the speed of 50m/s, 70m/s, 90m/s and 110m/s respectively by a steel sphere with the diameter of 4mm. At the lower velocities of 50m/s, there was no visible damage on the surface of the back side. When it comes to the speed of 70m/s, the corner fracture could be found on the edge of the specimen. The reason is that the compressive wave propagates to two related free surfaces, then the two unloading tensile waves interacted with each other. The spallation occurred at the velocity of 90m/s and 110m/s, coupled by one-fracture mode and three-fracture mode respectively. When the specimen was impacted at 110m/s, the back surface initially fractured at the center of the impact zone, followed by the two fractures near the center. The difference between the two modes can be attributed to the different interfacial strength and fiber strength. Moreover, the DIC method was adopted to measure the strain of the impact area to understand the post-impact tensile behavior of 2D-C/SiC. As the impact velocity increases, the residual strength of the material decreases slowly initially, and then it drops sharply.

11:10 AM

(ICACC-S1-019-2020) Toughening Additively Manufactured Ceramics by Laser Direct Deposition

X. Dong*[1]; J. Pappas[1]

1. Missouri University of Science & Technology, Mechanical and Aerospace Engineering, USA

Additive manufacturing of ceramics via laser direct deposition is difficult to achieve due to high thermal gradients, resultant transient and residual stresses as well as typically low efficiency. In particular, prevalent cracking issues pose a major challenge and limit its application in ceramics. In this study, toughening via dopants is investigated for laser direct deposited ceramics. Microstructural characteristics of doped ceramics are studied in terms of part density, grain size and distribution, crack prevalence and characteristics. Nearly fully dense ceramics are successfully fabricated by laser direct deposition. A multi-scale analysis is developed to study microstructure evolution and its relations with crack formation and mechanical strength. This is achieved by bridging numerical techniques at different length scales ranging from first principles to continuum modeling. It is shown that the composition and interfacial phase play a major role in cracking. Dopants help toughen the deposited ceramic samples through crack inhibition and suppression. Increased dopants also result in grain refinement, which is correlated with an increased fracture strength of the fabricated parts. These findings, combined with high cooling rates inherent in laser deposition processes, make it possible to control grain size, density, and crack prevalence and tailor corresponding mechanical strength.

Processing-Microstructure-Mechanical Properties Correlation II

Room: Coquina Salon D
Session Chairs: Raul Bermejo, Montanuniversitaet Leoben; Marina Ruggles-Wrenn, Air Force Institute of Technology

1:30 PM

(ICACC-S1-020-2020) Processing and Properties of Engineered Metal Matrix Composites Produced Via Co-Extrusion for High-Temperature Friction Stir Welding

P. Brune*[1]; G. Hilmas[1]; J. Watts[1]

1. Missouri University of Science & Technology, Materials Science and Engineering, USA

Metal matrix composites are widely used in the tooling and mining industries due to their high hardness, fracture toughness, and wear resistance. Research at Missouri S&T is focused on improving the performance of tools used in the friction stir welding (FSW) of steels, by using a process called co-extrusion. Co-extrusion offers a way to fabricate hierarchical architectures in composites, by co-extruding a core of one material (e.g., a ceramic) and a shell of a second material (e.g., a metal). Compositions being investigated consist of the following core - shell combinations: WC/Co - Co, WC/Co – cBN/Co, and TiB_2/SiC/B_4C (TSB) - W/Mo. The mechanical properties of each composition were evaluated for viability in FSW, including measurements of flexural strength, hardness, wear resistance, and fracture toughness. Flexural strength and fracture toughness were also evaluated between 900°C and 1200°C in an Ar atmosphere. Cylindrical pin FSW tools were machined and tested using both spot and linear welding methods.

1:50 PM

(ICACC-S1-021-2020) Direct observation of failure in ice-templated ceramics under dynamic and quasistatic compressive loading conditions

S. Akurati*[1]; D. Ghosh[1]; M. Banda[1]; D. Terrones[1]

1. Old Dominion University, Mechanical and Aerospace Engineering, USA

Hierarchical materials design provides opportunities for developing engineering materials with novel architectures that are yet to achieve in traditional approach of materials processing. Ice-templating technique enables the synthesis of directionally porous ceramics, which are promising for structural, biomedical and energy related applications. This work investigated the effects of porosity, microstructure, and strain rate regime on the damage evolution and failure behavior in ice-templated alumina under dynamic and quasistatic compressive loading conditions. Dynamic behavior was characterized employing a split-Hopkinson pressure bar (SHPB) setup and a high-speed camera was employed to capture the deformation and failure characteristics. In the processed ice-templated ceramic materials, porosity, lamellar bridge density, microstructural morphology and other length-scale features were modified through the systematic variations of process and material variables. It was observed that compressive response, damage evolution and failure characteristics were strongly dependent on porosity, pore morphology, and strain rate. The results also strongly suggest greater structural stability in the ice-templated porous ceramics at high-strain rates. This work can be useful in the design and development of mechanically robust lightweight materials for extreme environments.

2:10 PM

(ICACC-S1-022-2020) Investigation on optical properties, mechanical properties and low temperature degradation(LTD) of yttria-stabilized zirconia

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2. Korea Advanced Institute of Science and Engineering (KAIST), Dept. of Mater Sci & Eng, Republic of Korea

Yttria-stabilized zirconia(YSZ) has been widely used as a dental restorative material due to its excellent mechanical properties. However, YSZ, which is mainly used as a dental material, is optically opaque and has low temperature degradation(LTD). Recently, as interest in aesthetics increases, translucency similar to real teeth is being emphasized. As a result, there is an increasing demand for full-contour zirconia which improves translucency by introducing a higher content of cubic phase despite the deterioration of mechanical properties. In this study, we investigated optically transparent 8YSZ which is stabilized with cubic phase. Since it is necessary to complete densification for the transparency, Li_2O is doped to improve the sinterability of 8YSZ and two step sintering was applied in order to compensate mechanical properties by inhibiting grain growth. And the consequent phase change, optical properties, mechanical properties and LTD were studied.

2:30 PM

(ICACC-S1-023-2020) Investigation on temperature dependent deformation mechanisms of flash sintered 3YSZ via in-situ microcompression test

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Flash sintering has an enormous potential over conventional sintering, hot pressing, and spark plasma sintering in that it can significantly reduce furnace temperature as well as sintering time. However, the mechanical behavior of flash-sintered ceramics remains less understood despite their intriguing microstructures and formation of numerous defects. Here, the deformation mechanisms of flash-sintered 3 mol % yttria stabilized zirconia (3YSZ) were studied via in-situ microcompression test at temperatures ranging 25 to 650°C. The flash sintered 3YSZ exhibits high fracture strain due to transformation induced toughening below the test temperatures of 400°C. At higher temperatures, crack nucleation and propagation are significantly retarded, and no more catastrophic failures are observed. Strain rate jump tests were also performed at elevated temperature (450 ~ 650°C) to investigate the temperature dependent deformation mechanisms. The activation energy for deformation and its implication are discussed.

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(ICACC-S1-024-2020) Mechanical performance and fractography of stereolithography-printed fully stabilized zirconia

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Additive manufacturing (AM) of ceramics, particularly of zirconia, is becoming of increasing interest due to the substantial freedom available in the design and fabrication process. However, due to the novelty of the field and the challenges associated with printing dense bulk ceramics suitable for structural applications, thorough investigations into the effect of printing on the mechanical performance are limited. Previous work has identified anisotropy in the mechanical properties, and attributed it to the layer-by-layer deposition method used in most AM techniques. Fractographic investigations

detailing the origins and effects of layer lines are limited. This study investigates the mechanical properties of a dense (>98 %TD), fully stabilized zirconia fabricated by a stereolithographic printing method following ASTM standards. Hardness and 3 pt. flexure testing were conducted followed by Weibull analysis and fractography. The analysis entailed five unique build directions and a conventionally manufactured reference material used as a control. Although the strengths were comparable to the reference material for some orientations, fracture frequently initiated at layer lines and related defects in all orientations. The findings indicate that if the layer lines can be prevented or engineered, the strength of stereolithography-produced ceramics can be improved.

3:30 PM

(ICACC-S1-025-2020) Phase Relation in Zirconia: High pressure and Temperature synthesized of Orthorhombic Phases and its Mechanical Properties

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Zirconia was well-known in 3 basis polymorphs: Monoclinic, Tetragonal, and Cubic. Besides, other high-pressure phases of Zirconia were Orthorhombic I and II. A Monoclinic has 7 coordination of cations, whenever it transforms to Orthorhombic II, the number will increase to 9. The reported theoretical bulk modulus of Orthorhombic II was 296 GPa which can be a hard material. On another hand, some previous studies were shown that it was not a super-hard material. So, the purpose of this study is to confirm whether an Orthorhombic II is a hard material or not. In our experiments, a substrate is Monoclinic Zirconia which had 99.99% of purity and synthesized by multi-anvil presser under high pressure and temperature conditions. Pressure and temperature were performed at 15.6 GPa and 600 to 1,600 °C respectively. After soaking 30 minutes, rapidly quenched the sample to room temperature then relieve the pressures. For the results, the XRD patterns were shown that a single phase of Orthorhombic II could be synthesized at 800 – 1400 °C and 15.6 GPa. We found that the density of Orthorhombic II was higher than the Monoclinic phase ~20%, met to the theoretical values. Elastic Moduli were measured by the sound velocity measurement method. The bulk modulus of Orthorhombic II was significantly higher than Monoclinic but shear modulus were the same values.

3:50 PM

(ICACC-S1-026-2020) CALPHAD-guided Alloy Design and Processing for Improved Strength and Toughness in Titanium Boride (TiB) Ceramic Containing a Ductile Phase

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A CALPHAD-guided alloy design strategy is used here to design and process fully dense titanium boride (TiB) ceramics containing a ductile phase for toughening. To design the compositions, ternary phase diagrams of Ti-B-Fe, Ti-B-Mo, and Fe-Mo-B systems were constructed using the thermodynamic databases which were verified to predict well the binary phase diagrams of components. It enabled to identify relatively lower reaction sintering temperatures for various alloy compositions. Two alloy compositions of the TiB ceramic with the ductile beta-phase (both having ~85% TiB + ~15% β-Ti phase) were considered for the demonstration of the present approach. The processing temperature was as low as 1423 K for a Fe/Mo-rich ceramic alloy composition using spark plasma sintering, but the formation of brittle intermetallic phase FeTi here was found to be detrimental to mechanical properties. The second TiB ceramic alloy composition, which was Fe/Mo-lean, could be processed at 1623 K. This composition is shown here to possess a very good combination of high flexural strength (~850 MPa) and high fracture toughness (~7.7 MPa√m), making it attractive for potential applications. The microstructural factors that control the strength and toughness have been identified and the ways to increase these properties further, in this class of materials, are suggested.

4:10 PM

(ICACC-S1-027-2020) Hall-Petch Behavior in Nanocrystalline $ZnAl_{2.01}O_4$ Sintered by Different Methods

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

Stoichiometric $ZnAl_{2.01}O_4$ powder was synthesized via reverse-strike co-precipitation and sintered into solid pellets in an SPS. The pellets were sintered using two different punches: a diamond/silicon carbide composite (HP) and tungsten carbide (DP). DP-sintered pellets followed the Hall-Petch relationship, exhibiting an increase in hardness as material grain size decreased down to 13 nm; however, HP-sintered pellets followed an inverse Hall-Petch behavior at low grain sizes where hardness decreased with grain size. This shift in performance between sintering methods may be attributed to the nature of DP-SPS in that tungsten carbide punches deform during late stages of sintering, allowing for higher densities to be achieved at similar grain sizes.

4:30 PM

(ICACC-S1-028-2020) Lot-to-lot variability of BN grades for space electric propulsion applications

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Historically, several grades of hot pressed hexagonal boron nitride have been used for space electric propulsion applications. This study investigates the material properties of a selection of commercially available boron nitride grades including HP, M26, M, BNXX, and Shapal Hi-M. This work complements the data presented at JANNAF 2018. The grades selected for this study are of interest because their available billet size is sufficient for space electric propulsion, or because they have thruster heritage. This research investigates a range of material properties, tailored and focused on enhancing performance, reliability, and economics of electric propulsion thrusters. This work builds upon previous efforts by studying lot-to-lot variability of some of the properties of interest. Coefficient of friction, moisture absorption, moisture sensitivity, hot press directionality, flexural strength, compression strength, elastic modulus, thermal conductivity, thermal emissivity, thermal expansion, density, X-ray diffraction phase, microstructure, and chemical composition were all investigated across two lots of materials to help guide the selection of advanced ceramics. Variability between lots is the critical concern of the study.

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

Environmental and Thermal Barrier Coatings I

Room: Ponce de Leon
Session Chairs: Gustavo Costa, NASA Glenn Research Center; Bryan Harder, NASA Glenn Research Center

8:30 AM

(ICACC-S2-012-2020) T-EBC system concepts for SiC-based ceramics

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Chemical reaction between reactive species in the gas stream and the SiC components results in the formation of SiO_2(s) and CO(g), and subsequent steam volatilization of the SiO_2 by formation of $Si(OH)_4$(g). Protection of SiC-based components using EBC systems, such as the

silicon-$Yb_2Si_2O_7$ (YbDS) system, has shown to survive thermal and steam cycling up to 2,000 hours at 1316°C when deposited densely upon a monolithic SiC substrate. At this time a ~3μm thick SiO_2 thermally grown oxide (TGO) grows in between the silicon and YbDS layers, which endures significant tensile elastic strain during thermal cycling, resulting in cracking and eventual failure of the coating due to ingress of oxidizing species along the edges of the sample. Furthermore, volatility of SiO_2 in the form of $Si(OH)_4$ from the YbDS layer due to interaction with steam at 1316°C reduces it to Yb_2SiO_5 (YbMS). This leaves behind a porous layer with a mismatching coefficient of thermal expansion (CTE), resulting in cracking and flaking of the top of the EBC system. Efforts are made to reduce the propensity for failure of silicon-YbDS coating systems by EB-CPD deposition of a reactive HfO_2 layer in between the silicon and YbDS layers to transform the CTE mismatched SiO_2 TGO in a CTE matching $HfSiO_4$ layer. Furthermore, a porous, steam impervious, HfO_2-based thermal barrier coating layer was deposited using EB-DVD on the YbDS layer to reduce the loss of SiO_2.

8:50 AM

(ICACC-S2-013-2020) Effects of Topcoat Modifications on Bond Coat Oxidation and Internal Stresses in Multilayer Si/$Yb_2Si_2O_7$ Environmental Barrier Coatings

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2. Argonne National Lab, USA
3. NASA Glenn Research Center, USA

With exposure to high-temperature steam environments, progressive thermal oxidation of the bond coat in environmental barrier coating (EBC) systems leads to a poor match in material properties and, with sufficient oxide thickness, spallation of the EBC topcoat – a common failure mode of EBCs. Thus, limiting oxide growth of the bond coat is expected to increase the reliability and lifespan of EBCs. This study examines the effects of topcoat thickness and compositional modification on oxide growth and internal stresses in a current state-of-the-art EBC (Si/$Yb_2Si_2O_7$). Cyclic steam oxidation tests of these EBC systems were implemented to simulate the humid, high-temperature engine environment. Post-exposure analyses were used to investigate the effects of topcoat thickness and compositional properties on the thermally grown oxide (TGO) thickness and microstructure. High-energy synchrotron X-ray scattering and imaging techniques at Advanced Photon Source, Argonne National Laboratory were used to assess residual strain and damage in the multilayer systems as steam oxidation progressed. A comparison of strains, associated internal stresses, and microstructure between modified and baseline EBCs demonstrated the effects of topcoat modification as they may apply to EBC longevity.

9:10 AM

(ICACC-S2-014-2020) Effect of stabilization annealing on Ytterbium silicate phase transformation mechanism

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2. IHI ASIA Pacific(Thailand) Co., Ltd., Thailand

EBC (Environmental Barrier Coating) is indispensable for CMC(Ceramic matrix composites) which is considered to apply aircraft engine because of water vapor thinning. Rare earth silicate is gathering attention because of its low CTE(Coefficient of thermal expansion) and high water vapor resistance. In recent years, many studies of the development of phase transformation mechanism of Ytterbium silicates were reported. On the other hand, few studies have been reported on phase transformation of Ytterbium silicates sprayed by LPPS(Low pressure plasma spraying). In this study, we investigated the phase transformation mechanism of Ytterbium monosilicate sprayed by LPPS. And we suggested stabilization annealing of its coating designed from transformation mechanism, furthermore, we investigated the effect of stabilization annealing against water vapor resistance.

9:30 AM

(ICACC-S2-015-2020) Developing Environmental Barrier Coatings on Turbine Blades with Very-Fast Self-Crack Healing Ability by a Novel Thermal Process

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

The ceramic-matrix-composites reinforced with silicon carbide, such as $Yb_2Si_2O_7$-Yb_2SiO_5-SiC, with self-crack-healing ability are promising top-coat materials for the multilayered environmental barrier coatings (EBCs) on the next-gen jet engine's turbine blades. Stress-induced micro-cracks have been confirmed that can be fully healed by a two-steps process: the oxidation of the SiC filler to SiO_2, then the reaction of this glass with the matrix minor phase Yb_2SiO_5 to form fresh $Yb_2Si_2O_7$. In the present research, we describe a novel thermal process, using a handy gas burner to heat a pre-cracked composite of $Yb_2Si_2O_7$-Yb_2SiO_5-SiC up to around 1000 °C in air ambient, to activate its self-healing ability. The composite demonstrated a very-fast healing rate which is confirmed by the disappearance of the pre-cracks after only five minutes of burning. The fact that bending strength of the composite was completely recovered and further improved indicates the effect of this healing method. The mechanism of this very-fast healing rate is associated with the aforementioned double reactions which both are involved in the volume expansion at the composite surface. The characterizations such as SEM and XRD were carried out to elucidate this mechanism.

9:50 AM

(ICACC-S2-016-2020) Improvement in oxygen shielding properties of $Yb_2Si_2O_7$ at high temperatures using discontinuous changes of chemical potentials

M. Wada*[1]; T. Matsudaira[1]; T. Yokoi[1]; N. Yamaguchi[1]; N. Kawashima[1]; T. Ogawa[1]; D. Yokoe[1]; T. Kato[1]; S. Kitaoka[1]; M. Takata[1]

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The mass transfer phenomena in polycrystalline $Yb_2Si_2O_7$ (YDS) wafer under oxygen potential gradients ($d\mu_O$) have been evaluated using an oxygen permeation technique. The oxygen permeation of YDS under dry $d\mu_O$ was determined by interdiffusion of Yb and oxide ions along grain boundaries (GBs). Under wet $d\mu_O$, the oxygen permeation was below the detection limit. However, SIMS measurements showed the acceleration of diffusion of oxide ions in the vicinity of the YDS surface. In addition, the Yb_2SiO_5 (YMS) layer completely covered the high P_{O2} surface and YMS particles were segregated along the GBs inside the wafer. The oxygen shielding properties of YDS are improved by the YMS covering on the high P_{O2} surface, although the oxygen permeability of YMS is larger than that of YDS. The Yb chemical potential increases discontinuously at the YDS/YMS interface in the direction from the YDS wafer to the YMS layer, which results in the suppression of outward GB diffusion of Yb ions in the YDS. Simultaneously, YMS particles are newly formed along the GBs via the reaction of accumulated Yb ions with diffused oxide ions. The YMS coated YDS wafer have improved oxygen shielding property even under dry $d\mu_O$.

10:30 AM

(ICACC-S2-017-2020) Effect of plasma spray deposition process history on polymorphism and cracking response of yttrium silicate environmental barrier coatings (Invited)

R. Sarrafi-Nour*[1]; C. Johnson[1]; L. Rosenzweig[1]; Y. Gao[1]; J. Wan[1]; K. Luthra[1]

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Owing to their attractive properties, rare-earth silicate material system is now well-established as the material system of choice for environmental barrier coating (EBC) on Si-based ceramics and ceramic matrix composites (CMC) components of advanced gas turbines and propulsion engines. Thermal spray methods, atmospheric plasma spray (APS) in particular, have been a common platform for application of both thermal and environmental barrier coatings at industrial scale. Complexities of EBC material systems combined with some of the stringent performance requirementscompelscloserattentiontocomposition-process-property-performance relationship to ensure long-life and durability of EBC systems. Yttrium silicates, both the disilicate and monosilicate, have been evaluated for EBC applications as an economically favorable material system in comparison to the Yb- and Lu-based counterparts. All rare-earth silicate materials, except for Lu-disilicate, are subject to polymorphism. This presentation will discuss the effect of polymorphism in yttrium disilicate APS EBC systems, its interactions with the processing history of the coatings, and its impact on the performance of the EBC system during laboratory cyclic steam oxidation tests.

11:00 AM

(ICACC-S2-018-2020) Solid Solutions in the $Yb_2Si_2O_7$-$Gd_2Si_2O_7$ and $Y_2Si_2O_7$-$Gd_2Si_2O_7$ Systems: Phase Transformations and Structure-Property Relationships

J. L. Stokes*[1]; B. J. Harder[1]; V. L. Wiesner[2]; D. E. Wolfe[3]

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2. NASA Glenn Research Center, Materials and Structures Division, USA
3. Pennsylvania State University, USA

Rare earth (RE) disilicates ($RE_2Si_2O_7$) have attracted much scientific interest due to their suitability as environmental barrier coatings (EBCs) and applicability in many other technological fields. In order to study the phase transformation and property-structure relationships, select $RE_2Si_2O_7$ were studied. In particular, solid solutions within the $Yb_2Si_2O_7$-$Gd_2Si_2O_7$ and $Y_2Si_2O_7$-$Gd_2Si_2O_7$ systems were synthesized by solid state reaction route and characterized for their structural and thermal properties pertinent to application as EBCs. $Y_2Si_2O_7$-$Gd_2Si_2O_7$ formed a complete solid solution range exhibiting an orthorhombic δ-$RE_2Si_2O_7$ structure. Incorporation of $Gd_2Si_2O_7$ into $Yb_2Si_2O_7$ resulted in multiple phase transformations, with the solid solutions exhibiting monoclinic β-$RE_2Si_2O_7$, monoclinic γ-$RE_2Si_2O_7$, and δ-$RE_2Si_2O_7$ structures. The implications of phase stability and thermal properties of these solid solutions on the application in EBCs are discussed.

11:20 AM

(ICACC-S2-019-2020) Function Graded Material (FGM) $Yb_2Si_2O_7$/ Yb_2SiO_5 Volatilization Barrier for Durable TBC/EBC Architectures

E. Garcia Granados*[1]; E. J. Gildersleeve[1]; F. R. Caliari[1]; S. Sampath[1]

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

The volatilization barrier in multilayered TBC/EBC systems envisioned to protect CMC components in forthcoming power generation and aircraft/spacecraft turbine engines will consist on a top layer with low SiO_2 recession rates in water vapor atmospheres and a bottom layer with a CTE close to Si based CMCs. The most promising candidate materials for this application are atmospheric plasma sprayed (APS) Yb_2SiO_5 and $Yb_2Si_2O_7$ to act as top and bottom layers respectively. The main concern of this aproach is the CTE mismatch between these two materials which recommends the presence of extra layers between them with graded CTE to accommodate the thermal stresses. In previous works has been shown that plasma characteristics, like secondary gas and plasma power, modify the SiO_2 content of the final coating driving to different $Yb_2Si_2O_7$/ Yb_2SiO_5 ratio and CTE. In the present work are described the in-situ processing of function graded material (FGM) EBC and the limits of these approach using different plasma guns and plasma characteristics. Different techniques are employed to monitoring the in-flight characteristics of the particles (DPV2000) and the crystalline phases (XRD, DTA, CTE), microstructure (SEM) and chemical composition (EDX) of the as sprayed and heat-treated coatings, which are intimately related with their performance as volatilization barrier in TBC/EBC architectures.

11:40 AM

(ICACC-S2-020-2020) Equiatomic quaternary rare earth silicate solid solutions for multifunctional thermal and environmental barrier coating materials

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Environmental barrier coating (EBC) plays an indispensable role on the protection of engine components against extreme highly corrosive combustion environment. RE-silicates are the cutting-edge new EBC materials, but they are facing crucial challenge to solve the trade-off among critical mechanical, thermal and chemical properties. Recently, we developed a new kind of equiatomic quaternary solid solution materials, such as $(Y_{1/4}Ho_{1/4}Er_{1/4}Yb_{1/4})_2SiO_5$ and $(Ho_{1/4}Er_{1/4}Yb_{1/4}Lu_{1/4})_2SiO_5$. Dense and pure materials were prepared by hot-pressing method, and the compositional homogeneity at macroscale and atomic scale were confirmed by electron-dispersive spectrometer elemental mapping and high-angle annular dark-field images. Optimizations, especially moderate cocktail effects, are disclosed on elastic stiffness, thermal conductivity, thermal expansion CMAS corrosion and water vapor corrosion. The present work provides a new perspective on the design of EBC materials by complex multicomponent RE-doping strategy. It also highlights equiatomic quaternary solid solution is a competitive multifunctional thermal and environmental barrier coating (TEBC) material with balanced overall properties.

Environmental and Thermal Barrier Coatings II

Room: Ponce de Leon

Session Chairs: Douglas Wolfe, Pennsylvania State University; Kang Lee, NASA Glenn Research Center

1:30 PM

(ICACC-S2-021-2020) Calorimetric Measurements of the Thermodynamic Properties of RE-Silicate Coating Materials (Invited)

G. Costa*[1]; B. J. Harder[2]; N. P. Bansal[1]; B. Kowalski[1]; J. L. Stokes[3]

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2. NASA Glenn Research Center, Environmental Effects and Coatings, USA
3. Pennsylvania State University, Materials Science and Engineering, USA

Thermodynamic quantities of coatings materials and siliceous debris-induced corrosion products are crucial to understand in order to develop mitigation strategies necessary to improve the durability of gas-turbine engines. Siliceous induced corrosion can occur when debris consisting mainly of CaO-MgO-Al_2O_3-SiO_2 (CMAS) is ingested by aircraft engines during and after take-off, which sticks to hot surfaces and forms calcium rare-earth silicate oxyapatites. In this work, high-temperature oxide melt drop solution calorimetry (HT drop solution calorimetry) was used to obtain the enthalpies of formation for RE silicate ($RE_2Si_2O_7$, RE_2SiO_5 where RE = Yb, Er, Y, Dy, Nd, Lu and Gd) environmental barrier coatings (EBCs) and

the calcium RE silicate oxyapatite $Ca_2RE_8(SiO_4)_6O_2$ (RE = Yb, Er, Y, Dy, Nd, Gd and Sm) corrosion products. Trends in the enthalpy of formation as a function of the ionic potential of the rare-earth cations in their related crystallographic sites are discussed.

2:00 PM

(ICACC-S2-022-2020) Low-K, Durable Thermal/Environmental Barrier Coatings for SiC/SiC Ceramic Matrix Composites

A. Ghoshal*[1]; M. J. Walock[1]; C. Mock[1]; M. Murugan[1]; L. Bravo[1]; M. S. Pepi[1]; A. Nieto[2]; L. Fehrenbacher[5]; D. Hass[6]; A. Wright[3]; J. Luo[4]

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4. University of California, San Diego, USA
5. Tech Assess and Transfer Inc, USA
6. Directed Vapor Technologies, USA

US Army CCDC Army Research Laboratory along with their industry and academia partners have been studying performance durability of different compositions of low-k (thermal conductivity) thermal/environmental coating materials for SiC-SiC CMCs. The study includes studying different low-k thermal/environmental barrier coatings including layered Yb monosilicates and disilicates, $(Yb, Gd)_2Si_2O_7$ with and without YSZ layers and higher order Hafnium Oxide based top coats. This research additionally studies of the effects of mullite based and HfO2-Si and direct deposition of Yb disilicates on SiC/SiC CMC. The CMC T/EBCs are subjected to ARL's SMART rig and Hot-Particulate Ingestion Rig durability evaluations including thermal cycling (50 hot and cold cycles of 5 minutes at 1300 deg C) and prolonged combustion exposure of 30 minutes at 1300 deg C, with and without combusted sand impingement. As sprayed and tested coatings are characterized using optical microscopy, scanning acoustic microscopy, and scanning electron microscopy with energy dispersive X-ray spectroscopy and electron backscatter diffraction.

2:20 PM

(ICACC-S2-023-2020) Novel Environmental Barrier Coatings for the protection of SiC components

B. Kowalski*[1]; J. L. Stokes[2]

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2. NASA Glenn Research Center, Environmental Effects and Coatings Branch, USA

Greater gas turbine engine efficiency is a major goal in aeronautics research often pursued through increased engine operating temperatures. However, it is necessary to replace the current hot-stage alloy components with more thermally robust parts, such as Silicon-based ceramics and composites. Unfortunately, these materials are still susceptible to the effects of oxidation, water vapor, and (Calcium-Magnesium-Alumino-Silicate) CMAS interaction, among other issues at high temperature. To mitigate these effects, environmental barrier coating (EBC) materials are employed to help control the rate of degradation to the underlying composite, but must also survive the corrosive environment. The current study explores new design space in Mg-based EBC materials with respect to microstructure, high temperature phase stability, volatilization in water vapor, as well as structure-property relationships at high temperature.

2:40 PM

(ICACC-S2-024-2020) EB-PVD T/EBC systems for oxide fiber ceramic composites

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2. Siemens, Germany

Porous matrix, oxide fiber ceramic composites (OFCCs) are candidate materials for use in highly oxidative environments in gas turbine engines due to their intrinsic oxidative stability. However, limitations with available fibers necessitate using thermal barrier coatings (TBCs) for extended use at high temperatures. Additionally, environmental barrier coatings (EBCs) will improve component lifetime in high-temperature, high-pressure, high-velocity water vapor-containing environments. Mullite, a common OFCC constituent, is susceptible to degradation by volatilization of silica in the moisture-laden combustion environment. Electron beam-physical vapor deposition (EB-PVD) creates adherent coatings with favorable microstructures for low thermal conductivity and good compliance. This research applies this coating technique to OFCC systems, addressing challenges with depositing coatings that are thermomechanically and thermochemically stable with the components of the OFCC. 7YSZ, $Y_4Zr_3O_{12}$ (YZO), and yttria have been investigated as possible T/EBC materials, usually in bi-layer configurations. Selective matrix strengthening has been explored to increase durability of EB-PVD coatings on the porous matrices of OFCCs. The efficacy of EB-PVD T/EBCs has been assessed via thermal cycling in high temperature water vapor. Thermal expansion mismatch, coating material stability, morphologies, and deposition behavior will be discussed.

3:20 PM

(ICACC-S2-025-2020) High Temperature Oxidation Performance of Plasma Spray-Physical Vapor Deposition (PS-PVD) Environmental Barrier Coated SiC (Invited)

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

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2. NASA Glenn Research Center, USA

Turbine engines are becoming increasingly efficient as they incorporate new materials such as silicon-based ceramic matrix composites (CMCs) that allow for higher combustion temperatures and reduced cooling requirements. However, exposure of CMCs to turbine conditions causes significant recession and requires protection in the form of environmental barrier coatings (EBCs). Current EBC systems are limited by the silicon bond coat (T_m~1414°C), so new coating architectures and materials are required for advanced CMC/EBC systems. One method to achieve these advanced coatings is Plasma Spray-Physical Vapor Deposition (PS-PVD), which is a hybrid technique that can tailor microstructures and compositions to optimize performance. This work will discuss the design criteria needed for long life with a focus on high temperature corrosion in the form of a thermally grown oxide (TGO) layer on SiC. This TGO serves as a weak between the EBC system and underlying CMC, thus growth must be slowed to provide the long life needed for turbine engine applications. Coatings deposited on SiC via PS-PVD were evaluated by measuring the TGO growth rate at 1426°C in a 90% H_2O/O_2 environment to determine the expected durability and practicality of these prospective EBC systems.

3:50 PM

(ICACC-S2-026-2020) Microstructure evolution of EBCs in high-temperature high-velocity water vapor

M. J. Ridley*[1]; E. J. Opila[1]

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Environmental Barrier Coatings (EBCs) are required materials to protect SiC-based ceramic matrix composites from high temperature water vapor corrosion in combustion environments. While EBC recession from silica volatility is already known to occur, more research needs to be done to understand microstructural changes in order to determine optimum environments for various coatings. Research will include the following candidate EBCs: $HfSiO_4$, $Yb_2Si_2O_7$, and BSAS ($Ba/SrAl_2Si_2O_8$). Microstructure evolution of bulk EBCs and their product oxides is studied through a simulated steamjet environment, where high velocity water vapor of up to 250 m/s impinges on the material surface within a controlled tube furnace to rapidly progress EBC corrosion. Silica depletion measurements and weight changes are used to help develop lifetime

prediction models for each material. A quantitative understanding of the rate limiting factors of silica depletion is being determined through analysis microstructural changes with varied testing times, temperatures, and water vapor velocities.

4:10 PM

(ICACC-S2-027-2020) Steam oxidation kinetics of SiC coated with multilayer and single layer ytterbium disilicate environmental barrier coatings

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Multilayer environmental barrier coatings (EBCs) comprised of a silicon bond coating and ytterbium disilicate (YbDS) based top coating have become an industry standard for protection of SiC based ceramic matrix composites (CMCs) in water vapor containing combustion environments. SiC/SiC CMCs have primarily been utilized for post-combustion stationary components in aerospace jet turbines. The next generation of EBCs without a bond coating are now being evaluated for industrial gas turbine (IGT) applications where longer lifetimes are required. The present study examined the effect of YbDS-based EBCs on slowing oxidation and volatilization of SiC in water vapor at 1250°-1350°C using 1-h furnace cycle testing in air-90%H_2O. Multilayer (Si/YbDS) and single layer (YbDS) EBCs were deposited onto CVD SiC coupons by atmospheric plasma spray to determine oxidation rate constants using mass change and scale thickness measurements. Research sponsored by the U. S. Department of Energy, Office of Fossil Energy, Advanced Turbine Program.

4:30 PM

(ICACC-S2-028-2020) Deformation mechanisms of YSZ thermal barrier coating processed by air plasma spray and detonation gun thermal spray via in-situ microcompression tests

J. Cho*[1]; J. Li[1]; Z. Shang[1]; J. Lopez[2]; W. Jarosinski[2]; M. Gentleman[2]; V. Viswanathan[2]; S. Xue[1]; H. Wang[1]; X. Zhang[1]

1. Purdue University, Materials Engineering, USA
2. Praxair Surface Technologies, USA

Thermal barrier coatings (TBCs) are widely used in gas turbine engines for thermal insulation of superalloy structural components. Among many candidates for TBCs, yttria stabilized zirconia (YSZ) has received widespread attention in TBCs due to its superb thermal and mechanical properties. In this study, YSZ TBCs with dense vertically cracked (DVC) microstructure and highly porous structure were prepared by detonation gun thermal spray and air plasma spray (APS), respectively. In-situ microcompression tests reveal that the APS coatings showed high variability in fracture strength and poor deformability resulting from cracks and pores in the coating at room temperature. DVC coatings, conversely, exhibited fracture strengths ranging from 3.9 to 6.6 GPa and less variability in fracture strength attributed to the relatively dense and less defective microstructure. At 500°C, both coatings showed degraded fracture strength and significantly improved deformability attributed to ferroelastic domain switching and dislocation activities.

4:50 PM

(ICACC-S2-029-2020) Segmentation Crack Formation Dynamics During Air Plasma Spraying of Ceramic Coatings

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

The durability and performance of Air Plasma Sprayed (APS) ceramic coatings has been of significant interest ever since their introduction as Thermal Barrier Coatings (TBCs) for turbine engine applications. The coating properties that are recognized to be strongly correlated with durability include the fracture toughness and strain tolerance. Through the incorporation of segmentation cracks in the coating microstructure, these two properties are concurrently improved. As efforts are underway to find alternatives to Yttria Stabilized Zirconia (YSZ) coatings for high performance thermal barrier applications, the understanding of segmentation crack formation mechanisms in APS coatings is of prime importance. Concurrently, as the turbine engine industry shifts toward hermetically sealed silicate-based ceramic coatings, the primary focus is to avoid such vertical cracks. This study seeks to systematically study the formation dynamics of vertical cracks in industrially popular ceramics like Alumina, Yttria and Gadolinium Zirconate in comparison to Yttria Stabilized Zirconia through established beam curvature measurement techniques. The goal of this work is to answer the question(s) what makes a vertical crack form and where and when does this formation occur? Additionally, an attempt to categorize these coatings by the conditions facilitating formation of segmentation cracks is proposed.

5:10 PM

(ICACC-S2-030-2020) A micromechanical image-based model for the sintering of an air-plasma sprayed (APS) thermal barrier coating (TBC)

X. Zhang*[1]; Y. Okajima[2]; K. Takeno[2]; T. Torigoe[2]; A. Cocks[1]

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2. Mitsubishi Heavy Industries, Ltd., Japan

Sintering-induced microstructural changes in an APS TBC include the healing of inter-splat cracks and the coarsening of the columnar grains within each splat. Based on these observations, a micromechanical model is developed for the inter-splat cracks that sinter together at contacting asperities. The splats are assumed to undergo Coble creep determined by the diameter of the columnar grains, the splat thickness and the grain-boundary diffusivity. The growth of the columnar grains within the splats is considered. The proposed sintering model is implemented within a finite element (FE) framework. The starting microstructure is meshed from a typical as-deposited TBC. The numerical results predict an increase of the in-plane Young's modulus that is consistent with published experimental values. More importantly, the model enables a direct correlation between the increase in Young's modulus and the evolving microstructure. The modulus is determined by the detailed interaction of nearly horizontal wavy cracks and shorter vertical cracks. The horizontal cracks heal by sintering at asperities close to the crack tips, leading to a gradual shortening and closing of the cracks. The constraint on the sintering of these cracks can be relaxed by other nearby cracks (both horizontal and vertical) opening and stabilising. Later in the process these cracks can also sinter.

S3: 17th International Symposium on Solid Oxide Cells (SOC): Materials, Science and Technology

Novel Processing

Room: Crystal

Session Chair: Federico Smeacetto, Politecnico di Torino

8:30 AM

(ICACC-S3-009-2020) Possibility of SOFC forming by 3D printing (Invited)

A. Lipilin*[1]; V. A. Lipilina[2]

1. IEP UB RAS / SOFC-Technologies, LLC, Russian Federation
2. SOFK-Technologies, LLC, Russian Federation

The solid state of all SOFC components allows us to offer an infinite number of designs of electrochemical elements. To select the most successful designs in 1981, we proposed an experimental method for determining the design efficiency of cells and batteries. In Russia, in

addition to traditional tubular and planar structures, a block structure was developed for the space project, which has a higher packing density than the simplest structures used around the world. The technologies also had a certain development from slip casting into gypsum molds to Tape Casting and plasma spraying. The appearance and development of 3D printing leads to the replacement of ceramic technologies, confirmed the ability to increase the reproducibility of the basic electrochemical characteristics of SOFC, reduce the production time of the hot zone and the cost of products in general. SOFC designs have appeared more energy-intensive energy-efficient structures that cannot be implemented by known ceramic technologies. The specific volumetric capacities of 20 kW / liter no longer look limiting. Having sufficient competencies, it can be assumed that energy-efficient, energy-saving ELECTROCHEMICAL energy within the framework of the new technological structure is inevitably approaching and will finally take its rightful place in our society.

9:00 AM

(ICACC-S3-010-2020) 3D Printing Solid Oxide Fuel Cells

Y. Du*[1]

1. Kent State University, USA

Fuel cells are appealing energy generation gadgets due to their high performance, advanced electricity density, low emissions and quiet operation. Among exceptional types of fuel cells, solid oxide fuel cells (SOFCs) provide greater flexibility because they can run on a diffusion of fuels and are tolerant to impurities within the realistic fuels. 3D printing (also additive manufacturing, AM) processes enable the fabrication of complicated parts with the desired composition, microstructure and properties directly from the computer-aided design (CAD) models. This paper examines the applicability of currently available additive manufacturing processes that could be used to fabricate SOFCs. Successful fuels will be tested and analyzed in comparison with SOFCs made through conventional processes.

9:20 AM

(ICACC-S3-011-2020) Novel Sealing Approach for Improving SOFC Durability

C. Lockhart*[1]; N. J. Kidner[1]; D. Kopechek[1]; G. Arkenberg[1]; M. Seabaugh[1]; S. Swartz[1]

1. Nexceris LLC, USA

Seal durability is a major challenge facing solid oxide fuel cell developers as they strive to achieve the service lifetimes necessary for greater commercial acceptance. In addition to providing a hermetic, gas-tight bond under both reducing and oxidizing conditions, seals are required to provide mechanical compliance, integrity against thermal cycling, and avoid the formation of deleterious phases at component contacting surfaces. Nexceris has developed a new sealing approach, designed to reduce SOFC seal degradation. The approach represents the combination of several tailored materials sets chosen to improve the long-term seal stability. This talk will highlight the improved sealing behavior achieved with the new sealing approach, through offline leak-rate and aging studies, and relate sealing behavior to cell performance through a quantitative in-situ electrochemical model. Finally, the applicability of the sealing approach will be demonstrated through stack-level demonstrations.

9:40 AM

(ICACC-S3-012-2020) Freeze casting and freeze drying of tubular solid oxide fuel cell supports

Y. Du[1]; T. Woodson*[1]

1. Kent State University, USA

This work exhibits the proliferation of freeze casting technology as applied to manufacturing aligned and hierarchically porous tubular solid oxide fuel cell (T-SOFC) membrane supports. NiO-YSZ anode substrates of various compositions were freeze cast and dried to demonstrate enhanced performance/characteristics in T-SOFCs manufactured over traditional methods of manufacture such as extrusion. Freeze casting is a process whereby a solidifying solvent material templates the solute, forming a unique and complex microstructure within the casting. The technique enables control of the resultant structure through manipulation of the freezing environment, paprameters and materials used. Systematic development and subsequent results progressed through experimental adaptation and innovation to resolve challenges, mitigate and understand the root cause of observed defects, and to demonstrate the process on several baseline levels. As a result, T-SOFC anode substrates were cast in a reproducible manner for further coating with functional layers and performance testing. Goals for further work include demonstration of multiple cell casting methods for scalability, mechanical strength and electrochemical testing, permeability of membranes, and characterization by SEM and other methods.

Electrolytes and Sealants

Room: Crystal

Session Chair: Sebastian Molin, Gdansk University of Technology

10:30 AM

(ICACC-S3-013-2020) Tuning the electro-chemo-mechanical properties in defective cerium oxides

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1. Technical University of Denmark, DTU Energy, Denmark
2. Technical University of Denmark, Denmark

Cerium oxide is rendered as a key-material for electrochemical devices e.g. electrolytes, anodes, and other solid-state electrochemical cells. The ionic conductivity of these oxides is controlled by the dopant type/charge, more specifically with the microstructure and dopant configuration at the blocking barrier. These are mainly associated with solid-state mass diffusion phenomena that control the material assembling at the atomic scale. In this study, we explore the mechanisms constructing the ionic conductivity blocking barrier effects as a function of dopant type/concentration. These are the result of mass diffusion effects involved in the microstructural consolidation mechanisms from nanoscaled particles to continuum ceramic dense layers. Our experimental results show that ionic conductivity blocking effects are the net result of a combination of defect-cations configurations controlled by a multifold overlapping of dopant concentration, cation sizes, electrostatic interactions and density of cation diffusion migration paths. We observe that conductivity can be maximized by tailoring the ceria solid-solutions with strong electrostatic-elastic interaction, allowing local enrichment of solute content. Such effect also leads to steady electromechanical properties at low temperatures, highlighting the impact of designing of microstructural features in tuning characteristic properties in electroceramics

10:50 AM

(ICACC-S3-014-2020) Electrochemical Studies on Mixed Na^{+} - O^{2-} ions Conducting Sodium Zirconium Gallate + YSZ Composite

P. Elahi*[1]; A. V. Virkar[1]

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

Two contiguous phase pellets were fabricated containing 69 wt% Ga2O3 and 31 wt% YSZ. Sintered pellets were placed in alumina crucibles packed loosely in Na-B″-alumina powder and heat-treated at 1250C for 10h, 20h, 50h, and 70h. The two-phase Ga2O3+YSZ composite converted into a sodium zirconium gallate of composition Na0.7Ga4.7Zr0.3O8 and balanced YSZ. The formed component is polycrystalline with isotropic microstructure. At the first stage of conversion, Na2O from the vapor phase contributed in the reaction to form sodium zirconium gallate by diffusion of Na+ ions at the gas/solid interface: 2.35Ga2O3+0.35 Na2O+0.3ZrO2 → Na0.7Ga4.7Zr0.3O8. Subsequently, conversion continues by coupled diffusion of Na+ through the formed Na0.7Ga4.7Zr0.3O8

(initial thin surface layer) and $O2^-$ through YSZ at the three-phase boundary in bulk. A Na0.7Ga4.7Zr0.3O8 compound also was fabricated by a solid-state synthesis of a powder mixture containing Na2CO3, Ga2O3 and ZrO2 with the corresponding stoichiometry, dry milled at 200rpm for 3h and calcined in air at 1100 C for 3h. XRD, SEM, EDS had been carried out to ensure full conversion of the pellets concerning the thickness. EIS had been carried out over a temperature range of 30C to 850C. The obtained conductivity data could be fitted to two straight lines on an Arrhenius plot revealing two regimes of ionic conductivity at low and high temperatures.

11:10 AM

(ICACC-S3-015-2020) Particle Atomic Layer Deposition of Alumina for Flash Sintering Yttria-Stabilized Zirconia

R. J. O'Toole*[1]; B. Yoon[3]; C. J. Gump[2]; R. Raj[3]; A. W. Weimer[1]

1. University of Colorado, Chemical and Biological Engineering, USA
2. ALD NanoSolutions, Inc., USA
3. University of Colorado, Mechanical Engineering, USA

Yttria-stabilized zirconia (YSZ) is a common electrolyte material for solid oxide fuel cells due to its reasonable oxygen-ion conductivity and chemical stability. Flash sintering, a type of field assisted sintering, reduces the furnace temperature required to achieve near-theoretical density YSZ ceramics by ~700°C relative to conventional sintering. The addition of a small amount of aluminum oxide (Al_2O_3) has been shown to hinder grain growth, enhance densification, and increase YSZ ionic conductivity during conventional sintering. Typically, Al_2O_3 particles are added to YSZ particles by mechanical mixing which can lead microstructural inhomogeneities. In this work, particle atomic layer deposition (ALD) was used to conformally coat each YSZ particle with a thin film of amorphous Al_2O_3, homogeneously dispersing the Al_2O_3 throughout the green body. The powders were then flash sintered, and scanning electron microscopy was used to determine the effect of the Al_2O_3 films on grain growth and microstructural homogeneity. Density measurements showed that amorphous Al_2O_3 added by particle ALD increased the final density of YSZ after flash sintering, but increased the onset flash temperature. Particle ALD homogeneously adds amorphous Al_2O_3 to YSZ particles, resulting in higher final densities after flash sintering and a route to alter microstructural evolution and properties of interest.

11:30 AM

(ICACC-S3-016-2020) Effects of the paste compositions on the microstructure of reactive air brazed ceramic to metal components

K. Waetzig[1]; J. Schilm*[2]; W. Tillmann[3]; A. Eilers[3]; M. Manka[3]; L. Wojarski[3]

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2. Fraunhofer IKTS, Materials and components, Germany
3. Technical University Dortmund, Institute of Materials Engineering, Germany

Reactive air brazing of ceramics based on Ag-CuO type filler metals is a cost-effective way to produce ceramic-metal composites at air, without a protective gas atmosphere or a vacuum. In addition to furnace technology, brazing by means of induction heating can also be used effectively. Disadvantages of reactive air brazing are given by limited wetting of the ceramic surfaces, formation of pores or cavities in the brazing seams. Effects from incomplete debinding of the brazing pastes, insufficient wetting on the ceramic and side reactions with the copper are discussed as origins of these defects. In this study the sintering and melting behavior of different silver powders have revealed reboil effects of some qualities at high temperatures caused by evolving gases. Therefore, systematic variations of the filler metal composition regarding the copper content and the type of silver powder have been formulated. The sintering and wetting behavior of these pastes were investigated on various ceramics (Al_2O_3, ZTA and ZrO_2). Joining tests were carried out on the combinations of ceramics with steels using muffle furnaces and inductive heating. Investigations of the brazed seams and on the interfaces were investigated by using non-destructive ultrasonic microscopy as well as scanning electron microscopy. Also the tensile strength of the selected combination has been measured.

Interconnects and Cr Getters

Room: Crystal

Session Chair: Norbert Menzler, Forschungszentrum Jülich GmbH

1:30 PM

(ICACC-S3-017-2020) Degradation Characteristic of Ferritic Stainless Steel in SOFC for Automotive Use (Invited)

M. Yaginuma*[1]; T. Shiomi[1]; M. Abdul Jabbar[2]; N. Dale[2]

1. Nissan Motor Co., Ltd., EV System Laboratory, Japan
2. Nissan Motor Co., Ltd., Nissan Technical Center North America, USA

For installing SOFC (Solid Oxide Fuel Cell) -powered system to vehicle use as range extender electric vehicle (REX-EV), it is important to realize the compactness of SOFC stack size. One of its solution to reduce stack size is to apply ferritic stainless steel to SOFC stack parts (ex. Interconnector, Cell Frame and Stack Enclosure etc.) and this contributes to not only compactness and weight reduction but also low thermal expansion, which is one of key stack design parameters. On the other hand, it is known that operation temperature in SOFC stack is high (~700deg.C) and operation humidity is over 3% as an absolute humidity, therefore physical and chemical degradation of stainless steel are concerned. In this research, issues of applying stainless steel to SOFC stack parts for vehicle use are summarized at first. Then, high temperature corrosion and chromium evaporation, which are a kind of chemical degradation, are particularly focused. These issues are considered to cause not only degradation of stack performance but also significant system failure, so typical degradation mode of stainless steel under SOFC operating conditions, its influence and mechanism are also studied. Finally, the countermeasures by metallurgical and/or physical approach are investigated based on these results.

2:00 PM

(ICACC-S3-018-2020) High-temperature corrosion evaluation of Fe22Cr porous steels

S. Molin*[1]; M. Makowska[2]; J. Karczewski[1]; P. Z. Jasinski[1]

1. Gdansk University of Technology, Laboratory of Functional Materials, Faculty of Electronics, Telecommunications and Informatics, Poland
2. Paul Scherrer Institut, Photon Science Division, Switzerland

Porous steels are interesting engineering materials that can be possibly applied as filters, in gas membranes and fuel cells. One of the limitations for their use at high temperature is the formation of the oxide scale, which lowers the porosity, affecting its gas flow properties. In this work we have characterized porous Fe22Cr steels (~30% porosity) oxidized in the temperature range 700 °C-900 °C in air. Weight gain of samples is used to assess the oxidation kinetics followed by microscopy and X-ray diffractometry. In addition to the typical analysis methods, we have also employed synchrotron tomography analysis of the samples at different stages of oxidation, which gave us an insight into their 3D microstructure.

2:20 PM

(ICACC-S3-019-2020) Mass manufacturing of coated steel coils for Solid Oxide Cells: A journey of collaborative research and development

C. Bernuy-Lopez*[1]; L. Rioja-Monllor[1]; U. Bexell[1]; M. Stenström[1]; R. Berger[1]; J. Westlinder[1]

1. Sandvik Materials Technology, Sweden

Sandvik Surface Technology offers a portfolio of cost-effective coated metal strip for applications such as batteries, fuel cells and electrolyzers. The roll-to-roll coating lines for mass production

provides an outstanding opportunity for the cost-effective industrial mass production of coated metal components. The pre-coated solution reduces some of the handling that comes with batch coating of individual components. Sandvik Sanergy® HT 441 is a coated strip product designed to be used as interconnect in both SOFC and SOEC applications. Already today, the product is produced at industrial scale in the above-mentioned roll-to-roll PVD coating line. This product is based on an ASTM 441 steel grade and is double coated with thin layers of Cerium and Cobalt (< 1 µm). The material has shown to form a protective spinel layer with a $(Co,Mn)_3O_4$ composition by diffusion of Mn from the steel over time. This spinel phase is well known to improve the properties of the interconnect material due to the suitable electronic conductivity as well as the qualities to reduce the Cr evaporation more than an order of magnitude. A summary of the achievements realized through several EU-funded projects such as METSAPP, NELLHI, qSOFC and LOWCOST over the last decade will be presented in this work.

2:40 PM

(ICACC-S3-020-2020) Thermal and Electrical Properties of LSM/LSCF Composite as Cr Gettering Material

Y. Chou*[1]; J. F. Bonnett[1]; N. L. Canfield[1]; J. Choi[1]; J. A. Silverstein[1]; J. W. Stevenson[2]

1. Pacific Northwest National Lab, Materials, USA
2. Pacific Northwest National Lab, USA

Chromium poisoning has been known as the leading cause for cell degradation when metallic parts are used in SOFC. Mitigation by various coating and Cr-gettering materials have recently been investigated. In previous work, we have shown LSCF can absorb Cr volatile species when placed upstream of the stack; however, they may suffer poor bonding if used on cell directly due to CTE mismatch. In this work we propose LSM/LSCF composite as a candidate for on-cell Cr-gettering materials. LSCF will be mixed with base material of LSM at 10, 20, 30, and 40 v%. Mixed powders will be pressed and sintered as pellets and rods. Sintered rods will be tested for CTE in as-sintered and 500-1000h 800ºC aged form. Measured CTE will be compared with predictions from rule of mixtures. XRD will be used to characterize crystalline phases to assess chemical compatibility. The sintered pellets will also be measured electrical conductivity at 650-900ºC. Selected sample microstructures will be characterized by SEM/EDS and EPMA. Finally, candidate LSM/LSCF materials will be evaluated in a generic stack test fixture.

Stack / Cell Performance and Durability

Room: Crystal

Session Chair: John Hardy, Pacific Northwest National Laboratory

3:30 PM

(ICACC-S3-021-2020) Summarizing Jülich's results on long-term SOC stack tests and their post-test analysis (Invited)

N. H. Menzler*[1]; L. Blum[2]

1. Forschungszentrum Jülich GmbH, IEK-1, Germany
2. Forschungszentrum Jülich, IEK-3, Germany

Jülich Research Center is working since more than 25 years on the development of Solid Oxide Cell stacks for fuel cell, electrolysis and reverse operation. Meanwhile more than 500 stacks of various designs and size (2 to 40 planes) have been operated under numerous operational regimes and with varying materials, material combinations and different cells. Within these stack tests approx. 20 ran for more than 10,000h. Envisaged operation times are above 50,000h. During operation the overall degradation (loss of performance) should be in the range of ≤ 0.3%/1000h (voltage loss). The verification of these goals must be provided by operating end-product-like stacks under realistic conditions (time, temperature, size, fuel, air, thermal cycling etc.). Intensive post-test analysis of many of the above mentioned stacks gave inside into degradation issues and causes, interactions, degradation reasons, the necessity of protection layers and sometimes incompatibilities. Additionally, in some cases reasons for degradation could not be traced back to physical reasons. This presentation gives an overview on the past developments in Jülich, the post-test analysis of especially long-term operated stacks in fuel cell and electrolysis mode and tries to identify some degradation influencing factors. Finally, fields of in future necessary R&D will be highlighted.

4:00 PM

(ICACC-S3-022-2020) Two Dimensional Degradation Modeling of Planar Solid Oxide Fuel Cell under Practical Application Scenarios

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Lifetime evaluation and prediction is one of the key issues which should be conquered before Solid Oxide Fuel Cells (SOFCs) commercialization. During the past decades, the SOFC material degradation mechanism has become more and more clear, and the related degradation models like nickel coarsening have been built to evaluate SOFCs lifetime. However, the multi-physics and multi-scale effect in real SOFC stack on its degradation rate is another critical and practical issue but scarcely discussed in literature. Based on the real SOFC stack structure and parameters, using Finite Volume Method (FVM), this work builds a 10cm×10cm two-dimensional planar numerical model combined with SOFC performance degradation. A semi-empirical degradation model is added, and the degradation rate as well as multi-physics like current, temperature and components distribution evolution at each nodes are revealed. The impact of operation scenarios including constant current, constant voltage and constant power on the overall degradation rate evolution is studied. This work emphasizes the correlation between localized degradation and overall degradation, and analyzes the real stack stability with varies of application scenarios, which will help guiding the SOFC stack design and lifetime prediction from the perspective of stack stability.

4:20 PM

(ICACC-S3-023-2020) Electrochemical properties of the Ruddlesden-Popper series, $La_{1.7}Ca_{0.3}Cu_{1-x}M_xO_{4+\delta}$ (M: Co, Fe), as solid oxide fuel cells cathode

K. Hwang*[1]; T. Shin[1]

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

Layered Ruddlesden-Popper structured oxides have been extensively studied as candidates for IT-SOFC cathodes. This crystal structure shows high electronic conductivity and good oxygen mobility. The fast oxygen transport properties are related to their ability to accommodate hyper stoichiometric oxygen at the interstitial positions in the rock-salt layers. In particular, little attention, however, has been paid to cuprate-based materials, only a few studies have been carried out for the practical cathode performance in the case of SOFCs, which were concerned with single crystals or doped superconducting materials such as $La(Sr)_2CuO_{4+\delta}$. Therefore, we presented the development of the Sr free cathode materials, $La_{1.7}Ca_{0.3}Cu_{1-x}M_xO_{4+\delta}$ (M: Co, Fe) to avoid the chemical defections. In this study, Influence of Fe and Co contents on the lattice structure and electrochemical properties were investigated using structural analysis and catalytic activity. Furthermore, the performance was examined from 650 °C to 850 °C using LSGM electrolyte-supported cells. $La_{1.7}Ca_{0.3}Cu_{0.75}Fe_{0.25}O_{4-\delta}$ cathode composition showed the reasonable maximum power density of 589 $mWcm^{-2}$ at 850 °C so it is possible to use Sr free cathode.

4:40 PM

(ICACC-S3-024-2020) Residual Stress Evaluation of YSZ in Metal-Supported Solid Oxide Fuel Cell

T. Komaya*[1]; Z. Ruhma[1]; S. Watanabe[1]; K. Kumada[2]; K. Sato[2]; K. Yashiro[1]; T. Kawada[1]

1. Tohoku University, Graduate School of Environmental Studies, Japan
2. Tohoku University, Fracture and Reliability Research Institute, Japan

Metal-Supported design of Solid Oxide Fuel Cell (MS-SOFC) achieves high thermal shock resistance at a reduced material cost. However, it has been reported that when MS-SOFC is operated 1500 h at 1073K, Ni in Ni-YSZ diffuses into the support material, ferritic stainless steel becomes austenitic stainless steel, and thermal expansion coefficient mismatch occurs. In this work, we succeeded measuring the position distribution of residual stress of YSZ at high temperature using cosα method. The residual stress of YSZ was measured quarter of cell radius from the cell center. Measurement was performed at a temperature of 293 K-973 K and a dry 100% H_2 atmosphere. The residual stress of YSZ after co-sintering was most compressive at the cell center. When the temperature was raised, the residual stress of YSZ turned to be tensile showing the maximum value 120MPa at 573K-673K. On heating above 773 K up to 973K, the tensile stress of YSZ did not increase further and kept a nearly constant value. This is considered to be due to plastic deformation of Ni and the support material. The measured residual stress of YSZ at each temperature was largely different from among the thermal expansion coefficients of ferritic stainless steel, Ni-YSZ and YSZ. It could be because the ferritic stainless steel became austenite when co-sintered and thermal expansion coefficient mismatch occurred.

5:00 PM

(ICACC-S3-025-2020) Extreme thermal cycling of micro-tubular solid oxide fuel cells using a miniature ceramic heater

D. Panthi*[1]; H. Feng[2]; Y. Du[2]

1. Kent State University Tuscarawas, Department of Engineering Technology, USA
2. Kent State University, College of Aeronautics and Engineering, USA

Solid oxide fuel cells (SOFCs) must possess rapid start-up characteristics for their application in mobile and portable devices. In addition, they are required to withstand thermal shocks caused by sudden temperature variations. The start-up time and thermal shock resistance of SOFCs strongly depend on the cell and stack designs. Compared to planar designs, tubular designs have superior thermo-mechanical properties. Micro-tubular design, in particular, has low thermal mass and exceptionally high thermal shock resistance, thus making it ideal for rapid start-ups. Although there are several reports that demonstrate the short start-up time and thermal cycling ability of micro-tubular SOFCs, it has been challenging to conduct rapid thermal cycling tests at well-controlled temperatures. Since conventional laboratory furnaces are not suitable for high ramp rates, most of the rapid thermal cycling tests have been reported using gas burners. In the current research, we used a miniature ceramic heater to overcome the issues pertaining to rapid thermal cycling tests. The heater was capable of reaching SOFC operating temperatures in only a few minutes. We were able to conduct extreme thermal cycling of anode-supported micro-tubular SOFCs at heating and cooling rates >100 °C/min. The tested cells exhibited excellent thermal shock resistance without any degradation in OCV for over 100 cycles.

S4: Armor Ceramics - Challenges and New Developments

Quasi-Static and Dynamic Behavior II

Room: St. Johns
Session Chairs: Ghatu Subhash, University of Florida; Sikhanda Satapathy, Army Research Laboratory

8:30 AM

(ICACC-S4-011-2020) Behavior of Advanced Ceramics in Extreme Dynamic Environments (Invited)

K. Ramesh*[1]

1. Johns Hopkins University, Mechanical Engineering, USA

We present the integrated results of a multi-year basic research program on the behavior of advanced ceramics in extreme dynamic environments, bringing the story together through an integrative computational model that captures the major deformation and failure mechanisms active within the material during a major impact event. The approach is described primarily in terms of its application to boron carbide, viewed as a model material system. However, the model is generally applicable to advanced structural ceramics, and we also present results on silicon carbide and aluminum nitride. This integrated approach is based on experimental and computational investigations of ceramic behavior over multiple scales in both length and time, and in this sense is an integrated multiscale model. Our approach captures the fundamental mechanisms of amorphization and dislocation plasticity under pressure, dynamic fracture and fragmentation, and granular flow of the fragmented material. Each mechanism is described in terms of a physics-based submodel, and the coupling of mechanisms is explicitly considered. This physics-based approach thus also provides guidance to experts in synthesis and processing in terms of avenues for increased performance. We also describe some of these avenues through applications of the model to some example problems.

9:00 AM

(ICACC-S4-012-2020) Impact models for ceramics incorporating fragmentation and subsequent breakage

L. Graham-Brady*[1]; A. Bhattacharjee[1]; M. Cil[1]

1. Johns Hopkins University/APL, Civil Engineering, USA

The challenges involved in modeling dynamic behavior of ceramics with large flaw densities call for the use of continuum-based damage models. In such models, the onset of crack coalescence leading to rapid fragmentation and subsequent transition of intact material to granulated material is not well understood, given the lack of real time experimental observations about evolving flaw and fragment properties. Our current work attempts to bridge this gap between the onset of fragmentation and granular behavior, as well as provide a continuum breakage mechanics model for granular flow and evolving fragment size distribution. Crack statistics determined from wing crack growth-based damage model for different initial defect populations has been used to model discrete three-dimensional elliptical cracks. Subsequent coalescence between cracks has been addressed via two approaches: a coalescence surface approach and a coalescence zone approach. A parametric study provides insight on the input fragment statistics for granular flow and an appropriate granular phase transition criterion. The predicted fragment size distribution data is used as input for the breakage mechanics model. An integrative model composed of a micromechanics-based damage model and the breakage mechanics model is employed to simulate the response of ceramic to impact loading.

9:20 AM

(ICACC-S4-013-2020) Quantifying Kinematics During High-Strain-Rate Loading of Granular Materials

A. Gupta*[1]; K. Ramesh[1]; R. Hurley[1]

1. Johns Hopkins University, Mechanical Engineering, USA

The nature of granular materials undergoing high-strain rate compression plays a vital role in determining ballistic performance of armor ceramics. Previous work has not only identified granular flow as an important aspect of fragmentation in ceramics, but also qualitatively linked particle morphology and packing structure to the constitutive behavior of granular flows. To understand the quantitative role of particle morphology and packing structure on material response during high-strain-rate granular flow, it is necessary to develop a new characterization tool to track their internal deformation when subjected to rapid compression and shear in three dimensions. In this talk, we propose a "quasi digital volume correlation" approach for quantifying 3D displacement and strain fields. A three-dimensional image of the sample is first obtained using micro computed tomography (micro-CT). Two-dimensional X-ray phase contrast imaging (XPCI) with high temporal resolution is then obtained during impact. Combining the XPCI with the micro-CT scan and an optimization algorithm makes it possible to estimate the evolution of particle displacement, strain, and fabric throughout impact. This characterization technique will prove useful in probing the relationship between initial fabric and material response during rapid granular flow, and will aid in the development of new constitutive laws for armor ceramics.

9:40 AM

(ICACC-S4-014-2020) Particle Size Effect on Dynamic Granular Flow of Boron Carbide

X. Sun*[1]; K. Ramesh[1]

1. Johns Hopkins University, Mechanical Engineering, USA

Granular flow is one of the major deformation mechanisms of boron carbide under high strain rates (10^5 ~10^6 s^{-1}) and high pressures (~10 GPa). The rheology of granular materials can be influenced by porosity, particle shape, particle size and their evolutional characteristics. However, the effects of particle size on dynamic granular flow under such extreme loading conditions has not been well characterized. To understand the particle size effects, we designed and performed multi-axial loading experiments of pressure shear plate impact as well as normal plate impact on three commercially available boron carbide powders with average grain size: 0.7 μm, 10 μm and 80 μm respectively. The achieved strain rate can be as high as 1×10^5 s^{-1} and the pressure level goes up to 4 GPa. The effective friction coefficient (which is the ratio of shear stress to normal stress) is 0.16 for the 0.7 μm powder and decreases with increasing particle size. For the two larger size powders, many particles are damaged, indicating particle fracture as a key deformation mechanism. Our experiments will provide models of granular flow with important data for validation and parameter calibration.

Quasi-Static and Dynamic Behavior III

Room: St. Johns

Session Chair: Jerry LaSalvia, U.S. Army Research Laboratory

10:20 AM

(ICACC-S4-015-2020) Results from a Round Robin Exercise on Dynamic Compression Strength of Alumina: What Was Learned?

J. Swab*[1]; G. D. Quinn[2]

1. Army Research Laboratory, USA
2. American Dental Association Foundation, Paffenbarger Research Center, USA

Knowing the compression strength of advanced ceramics at high strain rates and whether the ceramic exhibits a strain-rate dependent strength is of interest for many applications. The most common method of obtaining a dynamic compression strength value is the split-Hopkinson pressure bar (SHPB) method. While the method has been used to evaluate ceramics there is no standard methodology or a standard specimen geometry for ceramic materials. In fact the SHPB parameters and specimen geometry tend to be unique to the organization conducting the tests. This leads to questions about data consistency and validity. A five laboratory round robin exercise was conducted to assess the strength of a commercial alumina using the SHPB method and a dumbbell-shaped specimen to address the concerns about data consistency and validity.

10:40 AM

(ICACC-S4-016-2020) On the Dynamic Stress Equilibrium in a Split-Hopkinson Pressure Bar Experiment

K. Upadhyay*[1]; G. Subhash[1]; D. Spearot[1]

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

Dynamic stress equilibrium is a fundamental criterion for a valid stress-strain test in a split-Hopkinson pressure bar (SHPB) experiment. Current literature suggests three popular approaches for achieving fast equilibration: (i) the use of short specimens, (ii) high impedance mismatched systems, and (iii) long rise time of incident stress pulse. Using fundamental wave propagation principles, this study quantifies the time to achieve stress equilibration as a function of transit time, impedance mismatch, and the incident pulse shape. Linear ramp pulse, Heaviside step pulse, and trapezoidal pulses of different rise times are analyzed for a range of impedance mismatch values. This work concludes that recommendations (ii) and (iii) do not necessarily lead to a reduced equilibration time, and their validity depends on the incident pulse shape and the impedance mismatch. Specifically, a linear ramp pulse results in a stress equilibration time that reduces with increasing impedance mismatch, while for a step pulse, an increased stress equilibration time is obtained when impedance mismatch is raised. For a trapezoidal pulse, the trend of stress equilibration time with rise time depends critically on impedance mismatch. As an important finding, in high impedance mismatch cases, trapezoidal pulses with rise times that are early even multiples of the transit time result in very small equilibration times.

11:00 AM

(ICACC-S4-017-2020) Investigating Compression Strength Anisotropy of Hot-Pressed Armor Ceramics

J. J. Pittari*[1]; J. Swab[2]; C. S. Meredith[2]

1. CCDC Army Research Laboratory, Material Response and Design Branch, USA
2. CCDC Army Research Laboratory, USA

The impact modeling community is perpetually seeking accurate material property measurements obtained from experimental efforts to help improve and validate the outcomes of such complex simulations. It is well documented that the hot-pressing procedures used for many strategic armor ceramics introduces anisotropic properties due to insufficient lateral confinement normal to the pressing direction. Therefore, knowing the orientation of a specimen extracted from a hot-pressed plate of material is vital towards appropriately applying the measured properties in a simulation. Prior research has demonstrated that the compressive strength of hot-pressed boron carbide is approximately 15-25% lower in specimens oriented with the long-axis parallel to the pressing direction compared to specimens extracted from the axis normal to the pressing direction. The use of miniature dumbbell-shaped specimens has faciliatated machining of the various specimen orientations from a thin plate of ceramic. In this study, hot-pressed boron carbide and silicon carbide specimens were produced with the major axis aligned at 0°, 45°, and 90° to the surface of the plate to investigate the intrinsic anisotropy of the hot-pressed part. Specimens were tested across a range of strain rates in order to discern the presence of rate effects on compressive strength and fracture behavior of these strategic materials.

11:20 AM

(ICACC-S4-018-2020) Static and Dynamic Compression Strength of Armor Ceramics

J. Swab*[1]; J. J. Pittari[2]; C. S. Meredith[1]

1. Army Research Laboratory, USA
2. CCDC Army Research Laboratory, Material Response and Design Branch, USA

The intrinsic compression strength of ceramics can be very difficult to determine. The specimen geometry and test fixture, if not properly designed, can result in the generation of undesirable tensile stresses that can lead to misleadingly low strength values. The compression strength of ceramics is often inferred from hardness data but this is not appropriate. Since compression strength is a parameter in numerous modeling and simulation packages used to predict performance it is imperative that the compressive strength of the ceramics be properly measured. Materials 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 high-strain rate experiments were performed using a split-Hopkinson pressure bar setup. High speed imaging was used in both cases to record the fracture process and to identify fracture initiation. This presentation will summarize the results to date on boron carbide, silicon carbide, alumina, glasses and transparent materials such as spinel and AlON.

11:40 AM

(ICACC-S4-019-2020) Dynamic Mechanical Characterization of BAM-B_4C Composites

R. A. Riera*[1]; S. Bavdekar[1]; M. DeVries[1]; G. Subhash[1]

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

Aluminum magnesium boride (BAM) and boron carbide (B_4C) are boron-rich icosahedral solids that show great promise for structural and armor applications due to their high strength, wear resistance, impact resistance and low mass density. Presently, the inability to economically mass produce BAM limits the number of experimental studies on its mechanical properties. In this investigation, Quasi-static and dynamic testing procedures were conducted on BAM-B_4C composite specimens, produced by spark plasma sintering, having 0% B_4C, 10% B_4C, 20% B_4C, and 50% B_4C. The compressive strength, hardness, and fracture toughness are experimentally determined in quasi-static and dynamic conditions and compared to values found in literature for BAM and B_4C. Their microstructure and failure modes are characterized using optical and electron microscopy as well as Raman spectroscopy and energy dispersive spectroscopy. Amorphization behavior in the composites was examined under a range of loading conditions (i.e., compression and indentation at multiple strain rates) using Raman spectroscopy.

Quasi-Static and Dynamic Behavior IV

Room: St. Johns

Session Chairs: John Pittari, CCDC Army Research Laboratory; Jeffrey Swab, Army Research Laboratory

1:30 PM

(ICACC-S4-020-2020) Thermodynamics of Pressure-Induced and Shock-Induced Amorphization in Boron Carbide-Unraveling the Mystery Through MD Simulations and Experimental Data (Invited)

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

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

In 1994 Grady conducted plate impact experiments on boron carbide and noted its anomalous behavior which he described as "dramatic loss in strength," "near fluid-like response," "heterogeneous deformation," "anomalous volume compression," and "phase-change-like volume collapse." Despite more than two decades of research, no convincing explanation is available to unravel this mystery. Through two seperate molecular dynamics (MD) simulations of high-pressure compression and shock loading of boron carbide (at velocities up to 6 km/s), develop Hugoniot curves for comparison with experimental data and draw a link between nanoscale thermodynamics to amorphization. For the first time, we generate Rayleigh lines via MD shock simulations and constructed Hugoniot. Shock-induced temperature in the ceramic exceeds the melting point at impact velocities as low as 2.5 km/s causing dramatic loss of strength and near fluid like response leading to amorphization. The simulated temperature data was also compared to estimates by other researchers. The anomalous volume compression is reflected in Hugoniot being below the hydrostat. By confirming amorphization to be a thermodynamically driven phenomenon, we resolve the scientific issues pertaining to anomalous behavior of boron carbide.

2:00 PM

(ICACC-S4-021-2020) Influence of Crystal Orientation on Shock Response of Boron Carbide

A. Cheenady*[1]; M. DeVries[1]; A. Awasthi[1]; G. Subhash[1]

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

Understanding the influence of crystal orientation on mechanical properties of boron carbide allows for optimal design of structures. In this work, we investigate the influence of chain orientation on the shock response of single-crystal boron carbide through molecular dynamics simulations of planar, normal impact experiments in LAMMPS. Three crystal orientations are studied, wherein the three-atom chain lies along, perpendicular to, and at an angle of approximately 53° to the shock direction. Eulerian and Lagrangian binning methods are utilized for calculating the quantities of interest. While the former method enables determination of temperature, density, pressure and particle velocity through time in the direction of shock, the latter method allows for precise tracking of volumetric deformation, construction of Raleigh lines over a wide range of impact velocities and the P-V shock Hugoniot. Further, the level of post-shock crystalline disorder, indicative of amorphization, can be captured using radial distribution function. In addition to revealing the influence of crystal orientation on the HEL and shock Hugoniot of boron carbide, our study also captures the influence of temperature rise on loss of shear strength, level of melting, and the degree of post-shock amorphization at various orientations.

2:20 PM

(ICACC-S4-022-2020) Quasi-plastic zone characterization of regular and Si-doped boron carbide

S. Xiang[1]; Q. Yang[2]; C. Hwang[2]; J. LaSalvia[3]; R. A. Haber[2]; K. Y. Xie*[1]

1. Texas A&M University, Materials Science and Engineering, USA
2. Rutgers University, Dept. of Materials Science and Engineering, USA
3. U.S. Army Research Laboratory, USA

Boron carbide (B_4C) is a hard and lightweight material, which has many engineering applications. However, B_4C loses its strength and toughness when subjected to high shear stresses. To improve its mechanical properties, the pervious computation work has suggested micro-alloying B_4C with Si. Very limited understanding of the failure mechanism of boron carbide, both Si-free and Si-doped, under high shear stress conditions is due to the lack of direct experimental observation at the relevant length scale for damage zone. Here we investigate the local deformation microstructure of regular and Si-doped boron carbide under indents, using a novel precession electron diffraction technique and high-resolution transmission electron microscopy. We observed that Si-doped boron carbide displays dispensed micro-cracks, while Si-free boron carbide exhibits major local cracks and low interfaces.

2:40 PM

(ICACC-S4-023-2020) Suppressing of Amorphization in Boron Carbide: Silicon vs Boron Doping

Q. Yang*[2]; C. Marvel[1]; C. Hwang[2]; K. Christian[2]; M. C. Schaefer[2]; J. LaSalvia[3]; M. Harmer[1]; R. A. Haber[2]

1. Lehigh University, Dept. of Materials Science and Engineering, USA
2. Rutgers University, Dept. of Materials Science and Engineering, USA
3. U.S. Army Research Laboratory, USA

While boron carbide outperforms other armor ceramics at defeating small caliber projectiles, it suffers from catastrophic failure during high pressure and high-velocity events. The cause of such anomaly has been attributed to the formation of the nano-sized amorphous band, leading to fragmentation and loss of shear strength. Experimentally, microalloying boron carbide with silicon or boron can substantiate the suppression of amorphization. However, phase equilibria of B-C-Si suggest that Si-doping of boron carbide inevitably comes with B-doping which complicates isolating the effect of Si-doping. This work is set out to discern if incorporation of Si atoms into the lattice of B-rich boron carbide further suppresses amorphization. A boron-boron carbide and a silicon hexaboride-boron carbide diffusion couples were prepared to form a boron and a silicon-boron diffusion zone, respectively. Vickers indents were placed in the gradient of the diffusion zone to evaluate the hardness and the amorphization mitigation due to different dopant contents. Sections of the diffusion zones with identical B/C ratio but varying in Si content were compared to determine the effect of true Si-doping in boron carbide.

3:20 PM

(ICACC-S4-024-2020) Influence of Porosity on the Destruction of Rhombohedral Boron Carbide Under Shock Loads

V. Kartuzov*[1]; I. Kartuzov[1]; V. L. Bekenev[1]; O. V. Bystrenko[1]

1. IPMS NASU, Ukraine

Boron carbide is a promising material for applications in modern industry, in particular, in the manufacture of armored materials. Therefore, the properties of boron carbide under shock loads are one of the key points that attract the attention of researchers. In this paper, we study the processes of destruction in the rhombohedral boron carbide $B_{12}C_3$ under the action of impact loads in the presence of the pores. The study is based on computer modeling by the molecular dynamics method using ReaxFF interatomic interaction that was previously successfully employed by other authors to describe the amorphization processes in $B_{12}C_3$ under shear loads [1]. The aim of this work was to study of processes of destruction of the crystalline structure in the vicinity of pores as dependent on the intensity of shock loads. We determined the threshold intensity of the loads at which the destruction occurs, examined the effects of pore size, and the mechanism of the destruction. The simulations were performed for impact loads applied along the carbon chains. Intensity of loading was within the range of 50 – 400 GPA. The simulation results are used to explain the microscopic mechanisms of fracture of boron carbide during dynamic loads.

3:40 PM

(ICACC-S4-025-2020) Small amount TiB_2 addition to improve mechanical properties of B_4C

C. Hwang*[1]; S. DiPietro[2]; K. Xie[3]; Q. Yang[1]; A. M. Celik[1]; A. U. Khan[1]; V. Domnich[1]; S. D. Walck[4]; K. J. Hemker[5]; R. A. Haber[1]

1. Rutgers University, Dept. of Materials Science and Engineering, USA
2. Exothermics, Inc, USA
3. Texas A&M University, USA
4. U.S. Army Research Laboratory, Survice Engineering Co., USA
5. Johns Hopkins University, Dept. of Mechanical Engineering, USA

Small amount of TiB_2 (< 5 wt%) was added into B_4C through a novel method that combines the use of sputter deposition and hot pressing. Sputter deposition provided more uniform dispersion of TiB_2 grains with smaller grain sizes as compared to the conventional particulate mixing. Small amount TiB_2 addition demonstrated to be an effective way for improving the fracture behavior and toughness of B_4C while not sacrificing its outstanding lightweight property to a large extent: 2.3 wt% TiB_2 addition brought 15% improvement in indentation fracture toughness while resulting in less than 2% increase in density. The improvement can be attributed to the combination of crack impeding by TiB_2 grains and crack deflection at the B_4C-TiB_2 interfaces. TiB_2 also played as grain growth inhibitor resulting in a slight increase (2%) in Vickers hardness. This study was sponsored by the Defense Advanced Research Projects Agency under Grant No. W31P4Q-13-1-0001 and the Army Research Laboratory under Cooperative Agreement No. W911NF-12-2-0022 with additional support from the National Science Foundation I/UCRC Award No.1540027.

4:00 PM

(ICACC-S4-026-2020) Static and Dynamic Properties of B_6O-B_4C Composite

K. Ghaffari*[1]; R. A. Riera[2]; S. Bavdekar[2]; G. Subhash[2]

1. University of Florida, Material Science and Engineering, USA
2. University of Florida, Mechanical and Aerospace Engineering, USA

Icosahedral ceramics like boron suboxide (B_6O) and boron carbide (B_4C), have been shown to be capable materials for structural and armor applications due to their high strength, high hardness, and low density. Both of these materials have similar atomic structures, and have a propensity for localized amorphization under high loads. While B_4C has been extensively studied, minimal literature exists regarding B_6O as its processing is not yet optimized. However, even in its non-optimized state, the properties of B_6O have been demonstrated to be comparable to those of B_4C. Studies have shown that a composite of B_6O and B_4C could have an improved ductility and fracture toughness while maintaining its exceptional hardness. Our current study investigates the microstructure as well as the mechanical properties of B_6O and a 70 wt.% B_6O-30 wt.% B_4C composite, in order to draw a preliminary conclusion on the effect of forming a composite with B_6O and B_4C. Quasi-static and dynamic uniaxial compression and indentation experiments are performed to investigate the mechanical properties, while scanning electron microscopy is used to characterize microstructure and fracture modes. Finally, Raman spectroscopy is utilized to probe for presence of amorphization, determine compositional information, and investigate the presence of thermally induced residual stresses.

4:20 PM

(ICACC-S4-027-2020) Preparation of TEM cross-sections of Knoop indented hot-pressed boron suboxide

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

1. U.S. Army Research Lab, FCDD-RLW-ME, USA
2. Lehigh University, USA

Understanding of the deformation mechanisms in ceramics is crucial for improved armor materials. Previous studies have investigated amorphization, microcracking and macrocracking in boron carbide. The focus of this study is on the sample preparation and sub-surface effects of Knoop indentation in hot-pressed boron suboxide. Similar to boron carbide experiments, indents will be placed near the edge of samples and cross sections will be exposed using an ion miling technique. The indents will be infiltrated to aid in retention of areas heavily damaged during indentation. The goal of this study is to gain an insight into the responses in the inelasitcally deformed regions in boron sboxide. While it has been shown that an increase in Knoop hardness occurs in the boron suboxide with the addition of just 1 vol.% SiO_2. Further exploration is needed to gain insight into the mechanical response of hot-pressed boron suboxide through the use of sub-surface characterization. Transmission Electron Microscopy (TEM) and aberration corrected scanning transmission electron microscopy (AC-STEM) are used to

examine the ion milled prepared cross sections beneath the Knoop indented regions. Results from hot-pressed boron suboxide will be compared to those using silica based additives as well as indented boron carbide. Experimental procedures, preparation techniques and results will be presented.

4:40 PM

(ICACC-S4-028-2020) Understanding inverse Hall Petch relation in nanocrystalline zirconia

A. Bokov[1]; R. Castro*[1]

1. University of California, Davis, Material Science & Engineering, USA

The increase in hardness with decrease in grain sizes is observed in ceramics with grain sizes ranging from microns to ~100nm. However, the inverse behavior, i.e. a decrease in hardness below a certain grain size, also exists although the mechanisms behaind this phenomenon in ceramics are still under debate. In this talk we discuss the relationship between grain boundary energy increase and the on-set of the inverse Hall-Petch relation. Utilizing nanoceramics of yttria-stabilized zirconia (10YSZ) manufactured by high pressure Spark Plasma Sintering equipped with SiC/diamond plungers, thermodynamic measurements showed the grain boudnary energiers increase with reducting grain size, and the onset of this increase corresponds to the critical grain size of softernin as measured by Vickers indentation hardness. Since grain boundary energy is an excess energy related to boundary strength/stability, the results suggest that softening is driven by the activation of grain boundary mediated processes facilitated by the relatively weakened boundaries at the ultra-fine nanoscale which ultimately induce the formation of an energy dissipating subsurface crack network during indentation.

S5: Next Generation Bioceramics and Biocomposites

Next Generation Bioceramics II

Room: Coquina Salon C

Session Chairs: Igor Zhitomirsky, McMaster University; Anthony Wren, Alfred University; Hendrik Heinz, University of Colorado Boulder

8:30 AM

(ICACC-S5-009-2020) Design of Bioceramics from the Molecular Scale: Molecular Recognition, Assembly, and Applications (Invited)

H. Heinz*[1]

1. University of Colorado Boulder, USA

The development of bioceramics and biocomposites typically involves extensive trial-and-error studies while rational understanding and design using modeling and simulation has become feasible due to more accurate models and affordable computing resources. We will share examples of predictions of biomaterials properties at the 1 to 1000 nm scale, including recognition and assembly of metal, oxide, and biomineral nanostructures mediated by biomolecules and polymers. We will discuss specific adsorption and assembly of peptides and macromolecules on metallic and oxide/hydroxide nanostructures, rules of molecular recognition derived from simulations and comparisons with experiments. Using the Interface force field (IFF), we have been able to obtain predictions in chemical accuracy. Applications to nucleation and growth of bone, low dimensional materials, modified tooth enamel, and therapeutics will be discussed. New opportunities using reactive simulations (IFF-R) and data science tools for reinforcement learning of large computational and experimental data sets will be described.

8:50 AM

(ICACC-S5-010-2020) Synthesis of substituted β-tricalcium phosphate powders to improve their thermal stability and biological properties for bone regeneration

N. Somers*[1]; F. Jean[1]; M. Lasgorceix[1]; A. Thuault[1]; F. Petit[2]; A. Leriche[1]

1. University de Valenciennes, LMCPA, France
2. Belgian Ceramic Research Centre, Belgium

This study aims to synthesize substituted β-tricalcium phosphate powders to increase their thermal stability and biological properties in order to optimize the manufacture of 3D printed porous scaffolds for bone regeneration. For this purpose, the β-TCP is synthetized by coprecipitation of $Ca(NO_3)_2$ and $(NH_4)_2HPO_4$ solutions under controlled temperature and pH. The dopants are added into the reagent solutions to optimize the substitution. Complete characterizations are conducted to evaluate the influence of these dopants on the thermal stability and biological properties of β-TCP powders. The cationic dopants Mg^{2+}, Sr^{2+}, Cu^{2+} and Ag^{+} have been studied in various proportions (4.5 and 9 at%). In addition, conventional and microwave sintering of the obtained powders are compared in terms of microstructural and mechanical properties. The chosen sintering temperature is 1200°C to investigate the presence of α-TCP. The incorporation of dopants allows to avoid the phase transformation in these conditions and increases the relative densities of the ceramic parts.

9:10 AM

(ICACC-S5-011-2020) Future of dental biomaterials: Gazing into Bob's crystal ball (Invited)

J. Kelly*[1]

1. University of Connecticut, School of Dental Medicine, USA

In February 2019 the American Prosthodontics Society asked me to "ponder" the future of dental biomaterials. This talk was titled, "Gazing Into Bob's Crystal Ball". My basic approach was guided by the words of Confucius, "Study the past if you would define the future". Since I have often studied and lectured on how many of our important materials got into dentistry – it was a natural assignment. In this paper then, I review how key materials came to be used in dental practice and organize these "sources" into an extended classification, assuming future materials will likely derive from one of these. Next, I identify important examples in each category and then expound on likely future developments, some already in progress. These include: (1) increasing use of and benefiting from automated systems; (2) better engineering with existing materials via failure analysis (from clinically-retrieved specimens); (3) iterative improvements in materials and practices, again based on observations from clinical behavior; (4) the use of scaffolds for both hard and soft tissue repair; and (5) the development of "smart" materials. It is hoped that this contribution survives the test of time. Please note that this list importantly includes not just materials, but often-overlooked steps in their processing that will affect properties and clinical durability.

9:30 AM

(ICACC-S5-012-2020) Additive Manufacturing of Bioceramic Scaffolds by Combination of FDM and Slip Casting (Invited)

S. Esslinger*[1]; R. Gadow[2]

1. University of Stuttgart, GSaME, Germany
2. Institute for Manufacturing Technologies of Ceramic Components and Composites, University of Stuttgart, Germany

The use of bioceramics like calcium phosphates or bioactive glasses for the regeneration of critical bone defects, as they can occur for example after serious injuries or diseases, is under intensive researched worldwide. The advantages of additive manufacturing technologies make it possible to process these ceramics into customized patient-specific implant devices, so called scaffolds. In this study

the possibilities to produce bioceramic scaffolds by fused deposition modelling (FDM) technique in combination with ceramic slip casting is introduced and described. In the first step the polymer models, which represent the negative geometry of the ceramic component, are printed by a commercial FDM printer. In the second step a bioceramic slurry is casted into the polymer molds according state of the art ceramic processing technologies. After debinding and sintering the mechanical and structural properties of the scaffold sa,ples are characterized and the biocompatibility is tested via simulated body fluid. Results show that filigree structures can be produced with compressive strength up to 15 MPa and with high porosity. First tests indicate that the biocompatibility of these scaffolds is similar to those produced by further additive manufacturing technologies, for example binder jetting or foam casting.

10:10 AM

(ICACC-S5-013-2020) Biomimetic bone scaffolds based on co-substituted calcium phosphates and chitosan (Invited)

A. Ressler*[1]; M. Antunović[1]; M. Ivanković[1]; H. Ivanković[1]

1. Faculty of Chemical Engineering and Technology, University of Zagreb, Croatia

Biological apatite contains various amounts of anionic (i.e. CO_3^{2-}, SiO_4^{4-}, F^-, Cl^-) and cationic (i.e. Na^+, Mg^{2+}, K^+, Sr^{2+}, Zn^{2+}, Ba^{2+}, Al^{3+}) substitutions, which have crucial biochemical functions. Multi-substitution is one of the most widely used approach to mimic the chemical composition of the bone mineral and to improve the biological performance of calcium phosphate (CaP) materials. The multi-substituted CaPs, with varying ion content (Sr^{2+}, Mg^{2+}, Zn^{2+}, SeO_3^{2-}), have been prepared by using cuttlefish bone as precursor of Ca^{2+} ions. The effect of the doping on phase composition, crystal structure, morphology, thermal stability, element content and toxicity of synthesized materials was studied. The precipitated powders were triphasic CaP systems composed of HAp, OCP and amorphous CaP. Phase transformation and ion release were analysed during 7 days of incubation in simulated body fluid at 37 °C. Highly porous multi-substituted CaP/chitosan composite structures have been prepared by freeze-gelation technique, while morphology of scaffolds was imaged by scanning electron microscopy (SEM). The MTT assay of prepared scaffolds have shown no toxicity and the live/dead assay has confirmed good viability and proliferation of seeded cells by the culture time. The differentiation of MSC cells have been determined by RT-qPCR analysis.

10:30 AM

(ICACC-S5-014-2020) Electrochemical fabrication of composites for biomedical applications (Invited)

I. Zhitomirsky*[1]

1. McMaster University, Canada

Electrochemical methods have been developed for the deposition of biopolymer films containing hydroxyapatite, titania, alumina, zirconia, silica and bioglass. Cationic polymers, such as chitosan, linear polyethylenimine, poly-l-lysine, polyl-ornithine, and composites were deposited by cathodic electrodeposition. Anodic electrodeposition strategies have been developed for deposition of composites, based on anionic polymers, such as alginate, hyaluronate, carboxymethyl cellulose and polyacrylic acid. Electrochemical techniques have been designed for the deposition of composites, based on electrically neutral polymers, such as polymethylmethacrylate, polyetheretherketone and polytetrafluoroethylene. New techniques have been developed for the chemical modification of biopolymers. Electrochemical impedance spectroscopy and potentiodynamic polarization studies showed that the composite coatings provided corrosion protection of metallic substrates in simulated body fluid solutions. The use of biopolymers enabled room temperature processing of the films, eliminating the problems related to the sintering of bioceramics. Aqueous electrodeposition techniques have been developed for incorporation of proteins, water-insoluble drugs, and other functional materials in the composite films. New electrochemical methods were used for the fabrication of nanocomposite films for biomedical implants and sensors.

10:50 AM

(ICACC-S5-015-2020) Nanostructured calcium phosphates: From biomineralization to biomaterials (Invited)

R. Wang*[1]

1. University of British Columbia, Materials Engineering, Canada

About seventy percent of healthy bone's mass is carbonated apatite, one type of calcium phosphate minerals. Bone minerals are also a reservoir of calcium needed for body's basic functions. Naturally, calcium phosphates are biocompatible and have thus become one of the top choices in the development of biomedical devices for bone repair and replacement. This presentation provides an overview of the applications of calcium phosphates as orthopaedic biomaterials, with a focus on our progress in developing calcium phosphate coatings on orthopaedic implants and as a testing platform for cell evaluation and drug delivery. This presentation will introduce the ideas of making calcium phosphate coatings on the surfaces of implants to enhance bone/implant integration. Various processing techniques such as biomimetic deposition, electrolytic deposition, and evaporation-induced surface crystallization will be critically reviewed. This will be followed by our latest progress on the processing of a nano-structured hydroxyapatite for the assessment of osteoclast activity. After confirming the efficacy of alendronate on inhibiting osteoclast resorption, the results will demonstrate that prostate cancer cells could stimulate osteoclast activity. The study thus established an in vitro platform for drug screening and potentially studying bone metastasis.

11:10 AM

(ICACC-S5-016-2020) Germanium Based Glass Polyalkenoate Cements for Orthopaedic Applications: Glass Characterization, Physical and Bioactive Properties (Invited)

S. Mokhtari[2]; A. Coughlan[3]; N. P. Mellott[4]; A. W. Wren*[1]

1. Alfred University, Kazuo Inamori School of Engineering, USA
2. Alfred University, Materials Science and Engineering, USA
3. University of Toledo, Dept of Bioengineering, USA
4. Michigan State University, Materials Science and Engineering, USA

Glass polyalkenoate cements (GPCs) were originally formulated for restorative dental applications, however, their beneficial attributes in close proximity to bone mineral (hydroxyapatite, HAp) has stimulated interest in developing these materials as skeletal cements. One of the key benefits of employing GPCs to form skeletal adhesives is related to their versatility, which includes modifying the glass phase to tailor the GPC properties. This study investigates the addition of Germanium (Ge) into a SiO_2-ZnO-CaO-SrO-P_2O_5-based glass system. Glass characterization studies using X-ray photoelectron spectroscopy (XPS) and magic angle spinning nuclear magnetic resonance (MAS-NMR) determined that the addition of Ge increased the fraction of lower Q-speciation and subsequently the concentration of non-bridging oxygens (NBO). Ge-GPC formulated from these glasses presented superior physical properties (setting characteristics and compressive strength) compared to the control GPC and also presented extensive CaP deposition in Simulated Body Fluid (SBF) which crystallized to HAp after 1000 hours in SBF.

Next Generation Bioceramics III

Room: Coquina Salon C
Session Chairs: David Kisailus, University of California, Riverside; Pavel Evdokimov, Lomonosov Moscow State University

1:30 PM

(ICACC-S5-017-2020) Binary Nitric Oxide-Isoprene Breath-Gas Sensing System for Monitoring Human Performance in High-Altitudes and Critical Care (Invited)

P. Gouma*[1]

1. The Ohio State University, MSE, USA

Flying at high altitudes is known to cause distinct physiological changes which are manifested as hypoxia-related episodes. Variants of the endothelial nitric oxide synthase gene may determine how an individual adapts to altitude. Thus, monitoring changes in the exhaled NO in individuals may provide a biomarker for their relative high-altitude tolerance. Another biomarker that increases when acute hypoxic episodes occur to pilots in high altitude flights is isoprene. There has been no direct relationship between the lack of oxygenation and isoprene production. However, isoprene variations are often related to changes in the metabolism (e.g. hypoglycemia). Furthermore, hypoxia tolerance is achieved by reducing oxygen demand, as in hibernation, manifested by a reduced metabolic rate, assumed to protect against cellular hypoxia. Therefore, monitoring and studying the changes in two exhaled breath biomarkers, namely nitric oxide and isoprene in real time, may help elucidate the biophysical, biochemical, and physiological mechanisms that lead to episodes that manifest hypoxia in fighter pilots (and even regular critical care patients). This work focuses on our efforts to develop a binary (two-sensor array) breath gas sensing system for the continuous monitoring of NO and isoprene in exhaled breath of individuals.

1:50 PM

(ICACC-S5-018-2020) Hybrid nanomanufacturing of hierarchical wearable devices for self-powered human-integrated sensors and interfaces (Invited)

W. Wu*[1]

1. Purdue University, School of Industrial Engineering; Birck Nanotechnology Center; Regenstrief Center for Healthcare Engineering, USA

The seamless and adaptive interactions between functional devices and environment (e.g., human body) are critical for advancing emerging technologies, e.g., wearable devices, consumer electronics, and human-machine interface. The state-of-the-art technologies require a complex integration of heterogeneous components to interface the mechanical stimulus ubiquitous and abundant in the above applications. These limitations have severely hampered the advancement and broader utilization of related technologies. I will discuss our recent progress in developing self-powered human-integrated nanodevices through the nanomanufacturing of heterostructured nanodevices with hierarchical architectures based on the hybridization of biopolymer and inorganic nanomaterials. This new class of wearable devices are conformable to human skins and can sustainably perform self-powered, non-invasive functions, e.g., physiological monitoring and gesture recognition, by harvesting the operation power from the human body. Such a scheme is fundamentally hinged on the polarization induced current term in Maxwell's displacement current. This research is expected to have a positive impact and immediate relevance to many societally pervasive areas, e.g., biomedical monitoring, consumer electronics, and intelligent robotics.

2:10 PM

(ICACC-S5-019-2020) New zirconia-based ductile composites for biomedical applications: Opportunities and challenges (Invited)

H. Reveron*[1]

1. Univ Lyon, MATEIS UMR5510, Insa de Lyon, Ceramics and Composites Group, France

Zirconia possesses high strength and toughness for a ceramic and excellent biocompatibility However, yttria-stabilized zirconia (Y-TZP) which is increasingly being used in the dental field shows a brittle behavior and may undergo aging, compromising its mechanical performances and durability. Our current research is focusing on the development of a new type of very stable zirconia-based composite (84 vol.% Ce-TZP) containing two second-phases and exhibiting outstanding strength, toughness and ductility. The plasticity is induced by the zirconia phase transformation that occurs before failure. Moreover, an additional surface modification can be also applied for enhancing the bone tissue response. Hence, the high flaw-tolerance of these Ce-TZP based ceramics and the predictive nature of the strength, associated to a transformation-induced plasticity and a perfect stability in-vivo allow us to consider them as the "new materials of choice" not only for dental implants and intervertebral mobile prostheses (examples discussed here) but also for many other structural biomedical applications where the advantages of ceramics have been mitigated by their failure properties. Finally, contrary to most of brittle ceramics including Y-TZP, it is possible to use additive manufacturing technologies for processing porous structures with a lower stiffness without compromising the strength.

2:30 PM

(ICACC-S5-023-2020) Manipulating the Architecture of Lanthanide Doped Nanoparticles for Theranostics (Invited)

F. Vetrone*[1]

1. Institut National de la Recherche Scientifique, Université du Québec, Centre Énergie, Matériaux et Télécommunications, Canada

In the last decade, the field of rare earth doped nanoparticles has progressed from the basic understanding of the photophysical properties governing their nanoscale luminescence to their use in a variety of applications, with considerable focus in biology and medicine. This interest stems primarily from the ability to stimulate these luminescent nanoparticles with near-infrared (NIR) light as well as their diverse emission wavelengths spanning the UV to the NIR regions. Therefore, with a single NIR excitation wavelength, it is possible to observe anti-Stokes emission, known as upconversion, or single photon (Stokes) NIR emission in the three biological windows (BW-I: 700-950 nm, BW-II: 1000-1350 nm, BW-III: 1550-1870 nm) where tissues are optically transparent. Here, we present methods for controlling the luminescence of these nanoparticles through core/shell nanostructures/nanoplatforms and demonstrate how their various emissions could be harnessed for applications in biology and nanomedicine.

3:10 PM

(ICACC-S5-021-2020) Fabrication of novel complex-shaped macroporous biodegradable ceramics and hydrogel composites via various 3D printing technologies for tissue regeneration (Invited)

P. Evdokimov*[1]

1. Lomonosov Moscow State University, Chemistry Department/Materials Science Departmenet, Russian Federation

The rapid development of modern industry requires creating new types of biomaterials with enhanced performance characteristics, including structural ones. The main methods for producing biomaterials based on calcium phosphates do not allow obtaining products with a complex architecture, for example, with internal

pore channels with different and complex direction with a diameter of 100 microns. Modern methods of additive technologies allow creating products with a tailored space inside the structure. Among the wide variety of rapid prototyping methods (3D printing), the most promising approaches are stereolithography of filled photosensitive suspensions and robocasting of highly concentrated pastes. The stereolithography method has the best lateral resolution, which allows producing miniature products, and robocasting allows to create products of any size with high-speed. This talk tries to discuss several strategies for the additive fabrication of different types of personalised implants for bone tissue regeneration and the importance of pore architectonics for its osteoconductive properties. Russian Science Foundation partially supported this study under Grant No. 17-79-20427, 18-79-00256, 19-19-00587.

3:30 PM

(ICACC-S5-022-2020) Innovative solutions in order to produce multi bioceramic implants by 3D printing

C. Chaput*[1]; R. Gaignon[1]

1. 3DCERAM SINTO INC, USA

3DCERAM's expertise in ceramic 3d printing has led to significant advances in the 3d printing process. Since 2017, 3DCERAM has developed a new multi material printer, in order to print several ceramics at the same time. One of the aim applications will be the possibility to print HAP-TCP parts, with disruptive design, and targeted functionalization. 3DCERAM's unique 3D printing manufacturing process, based on laser stereolithography technology, is able to produce made-to-measure bioceramic cranial or jawbone implants. This enables the production of implants with three-dimensional shapes in hydroxyapatite and tricalcium phosphates, materials widely renowned for their osteoconductive properties. Thanks to the unique process, 3DCERAM can produce bioceramic implants with porosity structured in 3 dimensions. The 3D printing process allows control of the location and geometry of porous areas and define a consistent diameter of pores. In the same implant, we can combine porous and dense areas. With the new hybrid (multi material) printing process, 3DCERAM can print HAP-TCP implants, with new complex designs and biomedical functions. The new possibility to print multi ceramic implants will open up new perspectives for surgeons.

3:50 PM

(ICACC-S5-020-2020) Biological Blueprints Towards Next Generation Multiscale Composites (Invited)

D. Kisailus*[1]

1. UC Riverside, Chemical and Environmental Engineering, USA

There is a need for multifunctional lightweight materials that are strong, tough, and cost effective. Natural systems have evolved efficient strategies, exemplified in the biological tissues of numerous animal and plant species, to synthesize and construct composites from a limited selection of available starting materials that often exhibit exceptional mechanical properties that are frequently superior to mechanical properties exhibited by many engineering materials. These biological systems have accomplished this feat through controlled synthesis and hierarchical assembly of nano- to micro-scaled building blocks that are integrated into macroscale structures. Nature goes one step further, producing materials that display multi-functionality in order to provide organisms with advantages to ensure survival. We investigate a variety of organisms that have taken advantage of hundreds of millions of years of evolutionary changes to derive structures, which are not only strong and tough, but also demonstrate multifunctional features including damage sensing and self-cooling. We discuss our experimental findings in these hierarchical features as well as how discuss their formation. From the investigation of synthesis-structure-property relationships in these unique organisms, we are now developing and fabricating cost-effective and environmentally friendly bioinspired multifunctional engineering composites.

4:10 PM

(ICACC-S5-024-2020) Luminescent Glass-Ceramic Materials in Imaging Applications (Invited)

R. L. Leonard*[1]; D. Berkowitz[1]; C. W. Bond[1]; A. Evans[1]; A. Howansky[3]; J. A. Johnson[1]; Y. Jin[5]; A. R. Lubinsky[2]; J. McDearman[1]; A. Petford-Long[4]; A. Thomas[1]

1. University of Tennessee Space Institute, Mechanical, Aerospace, and Biomedical Engineering, USA
2. Stony Brook University, Radiology, USA
3. Stony Brook Medicine, USA
4. Argonne National Lab, Materials Science Division, USA
5. Northwestern University, Materials Science & Engineering, USA

The composite nature of luminescent glass-ceramics can yield advantages over other classes of materials in imaging applications. The versatility of glass ceramics allows for engineering of the embedded crystalline material and glassy matrix, so they may serve in a variety of roles, often simultaneously. For example, nanocrystals within a glass matrix may act as both luminescent and scattering centers; the glass matrix may host luminescent centers and also serve as a substrate for the deposition of detector electronics. The authors present results relating to their use of glass ceramics in computed radiography and indirect digital radiography applications, including emission spectra, x-ray quantum efficiency, spatial resolution, and conversion gain. Future opportunities for these materials will also be discussed. This research was supported by the Nation Science Foundation under grants DMR 1600783 and DMR 1600837.

4:30 PM

(ICACC-S5-025-2020) Fluorapatite (FAp) nano-hybrid from dicalcium phosphate dihydrate (DCPD) : Preparation and its unique properties (Invited)

M. Tafu*[1]; T. Toshima[2]; N. Okajima[1]; A. Iwaori[1]; S. Takamatsu[1]

1. National Institute of Technology, Toyama College, Japan
2. National Institute of Technology, Toyama College, Department of Mechanical Engineering, Japan

Fluorapatite (FAp) is stable mineral in presence in surface of dental enamel. FAp is more stable against acid than hydroxyapatite (HAp) and indicate antibacterials performance against various bacterium including S. mutans. We have investigated FAp obtained from reaction of dicalcium phosphate dihydrate (DCPD) and fluoride ions. From this reaction, obtained FAp has nano-scale FAp particle in original DCPD morphology. Therefore, usage of DCPD for FAp preparation is useful for FAp material having its nano-scale performance and bulk usability. In this presentation, we introduced our recently studies in three topics. (1) Improvement of reactivity of the DCPD by hybridization of nano-scaled FAp particles by using solution that contain only calcium, phosphate and fluoride ions, (2) acid resistance and fluoride release of FAp from DCPD, and (3) adsorption performance of ammonia gas on FAp from DCPD.

S6: Advanced Materials and Technologies for Rechargeable Energy Storage

Li-ion Battery: Material Design

Room: Tomoka A
Session Chairs: Vilas Pol, Purdue University; Valerie Pralong, CNRS ENSICAEN

8:30 AM

(ICACC-S6-010-2020) Computational screen and design on electrochemical energy storage materials (Invited)

J. Liu*[1]

1. Shanghai Institute of Ceramics, Chinese Academy of Sciences, China

The development of high specific capacity battery materials plays an important role in push forward for electric vehicles and large energy storage equipment. However, many battery materials have the problems of low specific capacity, poor cycling performance and high charge-discharge overpotential, which are essentially attributed to the poor electrochemical activity of materials, i.e. the low charge transfer ability (the coupling effect of material thermodynamics/dynamics changes and charge transfer quantity). Therefore, advanced computational electrochemical methods have been developed to speed up new material design. Based on these methods, we performed the microstructural design of lithium ion/sodium ion electrode materials, developing new electrochemical mechanisms, and improving the efficiency of electrochemical energy storage through combined calculation and experimental studies. Our studies reveal charge transfer coupled hydrogen transfer for electrochemical energy storage. By adjusting the layered structure to the sector structure, the electrochemical sodium storage in carbon conjugation rings is realized with increasing specific capacity. The screening of cathode oxygen evolution catalysts in lithium-O2 batteries was studied, and the charge transfer ability of catalyst surface structure (defining surface acidity) was revealed to be an important characteristic of electrocatalytic activity.

9:00 AM

(ICACC-S6-011-2020) Dry Processing Methods for Li-ion Battery Active Materials Synthesis (Invited)

M. Obrovac*[1]

1. Dalhousie University, Chemistry, Canada

Li-ion battery active materials are currently highly engineered powders having specific size distributions for good packing density and smooth rounded surfaces to reduce the amount of surface exposed to electrolyte. Additionally, such powders can have complex core/shell structures, gradient composition distributions, inclusions of other phases or have protective external conformal coating layers. The manufacturing processes currently used for making such particles can significantly contribute to battery cost. In this presentation alternate manufacturing processes based on dry methods will be presented. Dry processes have the potential to make current battery materials at a greatly reduced cost and high yield with little waste and few processing steps. In addition, dry processing may enable the manufacture of even more complex engineered particles than are currently made today.

9:30 AM

(ICACC-S6-012-2020) Effect of Doping on Mechanical and Chemical Stability of Cathode Materials: A Multiscale Modeling study (Invited)

L. Kuo[2]; R. Muecke[1]; P. Kaghazchi*[1]

1. Forschungszentrum Juelich, IEK-1, Germany
2. Freie Universität Berlin, Physikalische und Theoretische Chemie, Germany

In this talk, I will present our theoretical works to predict chemical and mechanical stability of Li-based cathode materials. In particular, effect of B and W doping on stability of NMC and NCA cathodes will be discussed. By combining Coulomb-energy analysis, density functional theory calculation, and ab initio thermodynamics, we calculated (lattice parameters) volume change and surface stability. First, the mechanism of lattice parameters change through the delithiation of cathodes will be discussed. Effect of doping on lattice parameters change will then be presented. Afterwards, relative stabilities of cathode surfaces before and after doping will be compared. It is shown that B-doping leads to the formation of α–Li_3BO_3 on the (0001) surfaces of cathodes. The large contribution of (0001) facets causes rod-like particles to be stabilized. Finally, it will be shown that the B-doped cathodes with tailored microstructures consisiting of the rod-like particles surrounding spherical-like particles are mechanically more stable than the bare cathodes.

10:20 AM

(ICACC-S6-013-2020) Stabilizing Electrode/Electrolyte Interface for Lithium Batteries (Invited)

Z. Chen*[1]

1. Argonne National Lab, USA

Lithium-ion batteries are the dominant energy storage devices for modern portable electronics, as well as for the emerging application for electric vehicles and smart grids. It has been a common sense that the success of lithium-ion technologies is rooted to the existence of a solid electrolyte interphase (SEI) that kinetically suppresses the parasitic reactions between the lithiated graphitic anodes and the carbonate-based non-aqueous electrolytes. Recently, major attention has been paid to the importance of a similar passivation/protection layer on the surface of cathode materials, aiming unlocking the full potential of cathode materials for a higher energy density. Our recent study revealed that protons generated from the parasitic reactions at the cathode surface play a critical role in the performance fade of cathode materials. Several approaches to suppress the generation of protons will be discussed.

10:50 AM

(ICACC-S6-014-2020) Alkali-earth metals-sulfur systems: Tackling the drawbacks of inefficient Ca and Mg electrolytes (Invited)

Z. Meng[1]; A. Scafuri[5]; K. Pirnat[4]; R. Dedryvere[2]; M. Morcrette[3]; L. Stievano*[1]; R. Dominko[4]; R. Berthelot[1]

1. Université de Montpellier, Institut Charles Gernardt Montpellier, France
2. University of Pau - CNRS, IPREM, France
3. LRCS, France
4. National Institute of Chemistry, Slovenia
5. Alistore ERI, France

While lithium-ion batteries (LIB) has revolutionized portable electronics and are making long-range electric vehicles a reality, they are approaching their performance and cost limits. Moreover, the increasing need in LIB might face limited resources for Li, involving possible cost volatility and geopolitical tensions. It is thus important to look at sustainable and performing alternatives, and combining cheap Mg or Ca anodes with inexpensive sulfur could produce

a game-changing technology. In theory, Ca/S or Mg/S cells exhibit high energy densities competing with LIB. However, research on such systems is facing major hurdles in designing suitable electrolytes due to the passivation of the alkali-earth metals with common electrolytes forming a blocking layer hindering the diffusion of the corresponding divalent cations. Two different approaches are proposed to tackle these problems: 1) The development of new non-corrosive and non-nucleophilic electrolytes, able to plate the alkali-earth metals efficiently at room temperature without reacting with polysulfides. 2) The use of alkali-earth metal alloys, less sensitive to passivation than pure metals, enabling the use of more classical electrolyte formulations. For both approaches, a proof of concept will be given showing that both of them have their advantages and drawbacks in terms of energy density, cycling performance and viability.

11:20 AM

(ICACC-S6-015-2020) Enhanced Lithium Transport in Epitaxial Thin Films for Lithium-ion Batteries (Invited)

M. Huijben*[1]

1. University of Twente, Netherlands

None of the current lithium-ion batteries can fully satisfy all challenging requirements for current energy storage. Essential for all high performance energy applications are processes that happen at the interfaces between the different components. Key problems include slow electrode process kinetics with high polarization and low ionic diffusion or electronic conductivity, particularly at the electrode-electrolyte interfaces. Epitaxial engineering is used to control the crystal orientation of electrode thin films, which enables a unique insight into the relation between electrochemistry and crystal directionality of such chemically complex inorganic interfaces, not obtainable in single crystals or polycrystalline samples. Here, I will show the lithium diffusion behavior in $LiMn_2O_4$ cathode, and $Li_4Ti_5O_{12}$ anode, thin films, which are epitaxially grown by pulsed laser deposition on single crystalline Nb-doped $SrTiO_3$ substrates. Control over the specific crystal orientation of the full thin film enables detailed analysis of the lithium diffusion along specific crystal planes ({001}, {110} and {111}). Single phase films show enhanced cyclability and faster charging speed, as compared to studies on polycrystalline materials.

11:50 AM

(ICACC-S6-016-2020) Processing and Properties of Anisotropic Hierarchical Porous Ceramics for Li-ion Battery Electrodes – An Experimental and Numerical Investigation

M. Azami-Ghadkolai*[1]; S. Creager[2]; R. Bordia[1]

1. Clemson University, Materials Science and Engineering, USA
2. Clemson University, Chemistry, USA

This presentation will focus on the development of processing strategies for hierarchical, anisotropic porous ceramics for Li-ion battery electrodes. We will first present a broad overview of current challenges and property requirements for next generation Li-ion batteries. Next, we will present results from our project focused on microstructural control in hierarchical and/or anisotropic porous ceramics. In this presentation, results will be presented on the processing approaches to make these designed microstructures, the quantification of the microstructure at different length scales and the performance of these materials as electrodes for Li-ion batteries. The experimental results will be complemented with numerical simulations on the performance of engineered electrodes. The numerical investigations provide clear insights into the local electro-chemical reactions and guidelines for microstructural design. This work was supported primarily by the National Science Foundation EPSCoR Program under NSF Award # OIA-1655740. Any Opinions, findings and conclusions or recommendations expressed in this material are those of the author(s) and do not necessarily reflect those of the National Science Foundation

Thermoelectric Materials

Room: Tomoka A

Session Chairs: Jon Goldsby, NASA Glenn Research Center; Palani Balaya, National University of Singapore

1:30 PM

(ICACC-S6-017-2020) Enhanced Thermoelectric Performance of Porous $SrTiO_3$ Ceramics Containing Exsolved Ni Nanoparticles (Invited)

M. Ohtaki*[1]; S. Hirata[1]; K. Suekuni[1]

1. Kyushu University, Interdisciplinary Graduate School of Engineering Sciences, Japan

Here we report an enhanced thermoelectric performance of sintered $SrTiO_3$ (STO) ceramics doped with Nb and Ni, which subsequently contained highly dispersed Ni nanoparticles formed via an exsolution reaction caused by a reducing post-treatment. Samples with a representative composition of $Sr_{0.95}(Ti_{0.8}Nb_{0.2})_{0.95}Ni_{0.05}O_3$ (STNNO) were prepared by calcining mixed raw oxide powders at 1273 K twice to form the STO phase. The resulting powder was mixed with a graphite powder and heated at 1693 K in air, and subsequently reduced at 1623 K under 5% or 20% H_2/N_2. An XRD study confirmed that all the samples have the STO structure even after the reducing post-treatment. SEM/EDS observation of the cross section of the samples revealed discretely dispersed Ni nanoparticles of ca.50 nm in diameter. While the electrical conductivity of the sample reduced in 20% H_2 was significantly higher than that reduced in 5% H_2, the Seebeck coefficient was very insensitive to the sintering temperature and reducing atmosphere. However, the lattice thermal conductivity was the lowest for the sample reduced in 20% H_2, resulting in ZT = 0.6 at 1073 K as the highest value among the STO-based oxides so far reported.

2:00 PM

(ICACC-S6-018-2020) Enhanced Power Output in Polymer-based Thermoelectric Devices through Thermal and Electrical Matching (Invited)

M. Mukaida*[1]; K. Kirihara[1]; Q. Wei[1]

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

Increasing the power outputs is of great interest to thermoelectric devices. We reported the fabrication of organic thermoelectric devices by laminating poly(3,4-ethylenedioxythiophene)/poly(styrenesulfonate) (PEDOT/PSS) film, a Ni foil and polyimide film. In this study, the shape and size of the Ni foil in the device were designed to match the thermal resistance and electrical resistance of the PEDOT/PSS film and to reduce the contact resistance between PEDOT/PSS and Ni. Because of the low thermal conductivity of the organic materials, a stable temperature difference of 50 K was achieved under natural cooling conditions without any heat sink at 100°C on high temperature side. As a result, the 0.9 cm^2 device exhibited a power density of 40 $\mu W/cm^2$ at 100°C.

2:30 PM

(ICACC-S6-019-2020) Highly dense and nanostructured thermoelectric $Ca_{2-x}Dy_xMnO_4$ ceramics

M. Allani*[1]; A. Bahezre[1]; D. Bregiroux[1]; G. Rousse[2]; C. Laberty-Robert[1]

1. Sorbonne University, LCMCP- UMR7574, Faculty of Science & Engineering, Chemistry Education and Research Unit, France
2. Sorbonne University - Collège de France, France

We aim to obtain highly dense and nanostructured calcium manganates ceramics to improve their thermoelectric properties. We prepared, by auto-combustion method, calcium manganates of Ruddlesden-Popper structure with the composition $Ca_{2-x}Dy_xMnO_4$ (x=0, 0.04, 0.1, 0.2). Their crystal structures were analyzed by XRD. The resulting powders were densified by conventional and spark plasma sintering (SPS) and their microstructure was characterized. The synthesized powders are made of nanoparticles with an average

size of 70 nm. Doping Ca_2MnO_4 by Dy has a strong anisotropic effect on the crystal structures. The relative density of the $Ca_{1.96}Dy_{0.04}MnO_4$ pellets, prepared by conventional sintering, reaches the maximum value of 85%. After Li_2O addition, dense ceramics (92% of theoretical density) with an average grain size of 540 nm were obtained. SPS allows to decrease the sintering temperature from 1200°C to 900°C and the sintering time from 10 h to 1 min. Thus, we obtained fully dense pellets with an average grain size of 140 nm. Coupling SPS with Li_2O additive decreases SPS sintering temperature from 900°C to 550°C. In that conditions, fully dense ceramics with an average grain size as small as 80 nm can be obtained. Thermoelectric properties are then measured with respect to the microstructure and the doping level. Results show that Dy-doped Ca_2MnO4 ceramics are good candidate for high temperature applications.

Sulphur Battery and Liquid Electrolytes

Room: Tomoka A

Session Chairs: Lorenzo Stievano, Université de Montpellier; Yuki Orikasa, Ritsumeikan University

3:10 PM

(ICACC-S6-020-2020) Development of High Li ion Transport Electrolyte for Fast Charging of High Energy Density Li-ion Cells (Invited)

Z. Du*[1]; X. Wu[1]; D. L. Wood[1]; I. Belharouak[1]

1. Oak Ridge National Laboratory, USA

Enabling fast charging capability of high energy density Li-ion cells could dramatically increase the widespread adoption of battery electric vehicles. However, fast charging is limited by Li ion depletion in the electrolyte and increasing Li ion transport from cathode to anode is essential. By evaluating different Li salts in the electrolyte, we find lithium bis(fluorosulfonyl)imide (LFSI) has both higher conductivity and higher Li ion transference number compared to the traditional $LiPF_6$ salt. In a 12-minute charge, the electrolyte with $LiPF_6$ salt reaches the cut-off voltage rapidly while the one with LiFSI exhibits a longer constant current charge with more capacity achieved. The LiFSI electrolyte also shows better cycling performance and less Li plating after repeated fast charging cycles.

3:40 PM

(ICACC-S6-021-2020) Concentrated Electrolytes for Li Batteries: Physicochemical Properties and Electrochemical Reaction at Graphite Electrode (Invited)

R. Tatara*[1]; K. Ueno[1]; K. Dokko[1]; M. Watanabe[1]

1. Yokohama National University, Department of Chemistry and Biotechnology, Japan

Typical liquid electrolyte solutions used for Li-ion batteries are composed of Li salt and organic solvents, which enable them to give the conduction property of Li^+ ions. Concentrated electrolyte solutions which have a salt concentration higher than ~3 mol/L, show remarkably different properties, such as reduced volatility/flammability[1], greater electrochemical window[1], enhanced rate performance[2], altered electrode reaction[3-6], and reduced interfacial reactivity[7], from conventional electrolyte solutions (~1 mol/L). These changes can be attributed to the reduction of free solvent molecules[8], which do not coordinate with Li^+ ions in the electrolyte solutions. In this study, physicochemical properties of concentrated electrolytes and its electrochemical reaction at graphite electrode will be discussed. [1] M. Watanabe et al., Chem. Rev., 2017, 117, 7190. [2] K. Dokko et al., J. Phys. Chem. B 2018, 122 (47), 10736. [3] H. Moon et al., J. Phys. Chem. C, 2014, 118, 20246. [4] R. Tatara et al., J. Phys. Chem. C, 2017, 121, 9162. [5] R. Tatara et al., J. Phys. Chem. C, 2018, 122, 18316. [6] R. Tatara et al., ChemElectroChem, 2019, DOI: 10.1002/celc.201900973. [7] R. Tatara et al., ACS Appl. Mater. Interfaces, 2019, DOI: 10.1021/acsami.9b11942. [8] K. Ueno et al., Phys. Chem. Chem. Phys., 2015, 17, 8248.

4:10 PM

(ICACC-S6-022-2020) Exploration of new titano-niobate based oxide as ionic conductor

V. Pralong*[1]; A. Neveu[1]; E. Anger[1]

1. CNRS CRISMAT, France

Looking for new material ionic conductor that could be use as electrode material even solid-state electrolyte, we decide to explore the system A-Ti-Nb-O system. We will report the synthesis and characterizations of ATiNbO5 as well as A3Ti5NbO14 with A=alkaline ion. We will discuss on the structure-properties relationship for the family of materials.

4:30 PM

(ICACC-S6-023-2020) 2D Materials and Composites for Safe Lithium Batteries (Invited)

R. S. Yassar*[1]

1. University of Illinois at Chicago, USA

Two dimensional (2D) materials are emerging materials for innovative design of Li-ion batteries that are safe and high energy density. This presentation encompasses recent progress in the PI's research team on addressing the Li-ion battery challenges via 2D materials design and integration. I first showcase a Li-metal case where graphene oxide (GO) materials were used to control the deposition of Li-metal ions during charge and discharge reactions. We demonstrated high cycling performance of Li-metal cell modified with GO in comparison to typical Li-metal cells. In another work, we studied the electrochemical cycling of Li storage in phosphorene 2D materials and showed interesting structural ordering during Li insertion in these materials and remarkable fast ion diffusion across phosphorene. Moreover, we show that the encapsulation of cathode particles with 2D materials can be an innovative approach to suppress the oxygen release in the high voltage cathodes. 2D graphene is impermeable to oxygen molecules and would be an ideal layer to coat over the surface of LiCoO2 nanoparticles. The cells containing graphene encapsulation could run at higher voltage for longer cycles.

S7: 14th International Symposium on Functional Nanomaterials and Thin Films for Sustainable Energy Harvesting, Environmental, and Health Applications

Nanotoxicity, Drug-delivery, and Tissue Engineering with Tailored Nano-bioconjugates

Room: Flagler A

Session Chair: Thomas Fischer, University of Cologne

8:30 AM

(ICACC-S7-008-2020) Estrogen-DOTA-Radio Nano-Conjugates Recognize Breast Cancer Receptors (Invited)

S. Ilyas*[1]; S. Sahnoun[2]; K. Wennhold[3]; S. Hussain[4]; K. Schomaecker[2]; S. Mathur[1]

1. University of Cologne, Institute of Inorganic Chemistry, Germany
2. University Hospital of Cologne, Clinic and Polyclinic for Nuclear Medicine, Germany
3. University Hospital Cologne, Center for Molecular Medicine Cologne (CMMC) and Cologne Translational Immunology, Germany
4. University of Cologne, Institute of Biochemistry I, Germany

Selective transport of nanoconjugates to a diseased tissue is highly desirable for prompt disease detection and therapy. Nevertheless, their chemical to biological identity change poses a significant challenge in designing and directing particles from lab to clinic. Herein, dopamine-capped Fe_3O_4 and gadolinium based core sell $SiO_2@Fe_3O_4$ NPs were grafted with an estrogen molecule and a

macrocyclic chelator followed by biological evaluation for prospective effectiveness as nanomedicine. Fe_3O_4 NPs served as efficient targeting nanoconjugates when vectorized by 17α-ethynylestradiol, via different chemical interactions. To expedite their imaging properties, 1,4,7,10-tetraazacyclododecane-tetraacetic acid, (DOTA) was attached on their surface to allow incorporation of positron emitting isotope gallium-68 and leutecium-177. The as-vectorized nanoprobes demonstrated physiological stability besides exceptional radiochemical yield of 99.4%. These nanoprobes also showed ER-mediated cellular uptake with benign toxicity to progesterone positive cell lines. Furthermore, in vivo NPs tracking showed their preferential accumulation in kidney and at tumor site as compared to other healthy organs. Given their active surface chemistry following ER-mediated accumulation at tumor site and outstanding radiolabeling efficiency demonstrate their tremendous prospective in combined imaging (PET) and therapy in preclinical settings.

9:00 AM

(ICACC-S7-009-2020) Monitoring and Probing the stability and dissolution of nano-MoO_x contrast agents for XRFbioimaging (Invited)

M. S. Toprak*[1]

1. KTH Royal Institute of Technology, Dept. of Applied Physics, Sweden

Contrast media based on nanoparticles (NPs) are used in various biomedical imaging techniques. X-ray fluorescence (XRF) bio-imaging can provide sensitive and quantitative detection of NPs. Recently we showed in a laboratory XRF system that XRF tomography could use spectrally matched molybdenum oxide (MoO_x) based NPs as contrast agents to achieve 200 μm spatial resolution in mice, beyond what can be achieved with other imaging techniques (PET, SPECT, MRI). The specific fluorescence signal allowed imaging of the MoO_x NPs as a time-series. Our attempts of in-vivo imaging has shown a significant difference on NP residence time in different regions. Understanding the behavior and fate of NPs in biological systems is still a challenge as they will have a biodistribution and in-vivo uptake controlled by their size and surface. NPs will also suffer from critical modification immediately after they come in contact with biological media. Stability of NPs in biologically relevant media has a prime importance on the in-vivo behavior of the NPs. We report on the stability of MoOx NPs using dialysis as membrane separation model and UV-Vis, DLS, FTIR, PL, and TEM as the characterization techniques. The obtained results will be discussed in detail, suggesting a mechanism of dissolution/interaction.

9:30 AM

(ICACC-S7-010-2020) Smart Mesoporous Core-shell Nanovectors as Anticancer Drug Carriers

A. Szymura*[1]; S. Ilyas[1]; M. S. Hussain[2]; S. Mathur[1]

1. University of Cologne, Institute of Inorganic Chemistry, Germany
2. University of Cologne, Institute of Biochemistry I, Germany

Overcoming the limitations of conventional cancer treatment like adverse effects of chemotherapeutic agents on healthy tissue and enhancement of their therapeutic effectiveness are occasionally the major challenges for the development of smart nanovectors today. These nanodrug carriers allow for the selective distribution and well-controlled release of anticancer drugs at the target tumor tissue site. Therefore, we present polyol process- and sol-gel-synthesized mesoporous core-shell nanovectors modified with ligands like folic acid, a diol or an imidazole derivative for the active targeting to breast cancer cells. These nanovectors were micrographically characterized and drug release studies were performed with doxorubicin in comparison to the natural flavonoide quercetin. To proof the cellular uptake of the targeting nanodrug carriers into MCF-7 breast cancer cells flow cytometry and confocal fluorescence microscopy measurements were performed. Furthermore, these smart mesoporous core-shell nanovectors can be extended as anticancer drug carriers to other types of cancer, e.g. ovarian, lung and colon cancer.

Synthesis, Functionalization, and Assembly of Inorganic and Hybrid Nanostructures I

Room: Flagler A

Session Chair: Muhammet Toprak, KTH Royal Institute of Technology

10:20 AM

(ICACC-S7-011-2020) Transition of nanomorphology in ceramic systems: Multifunctional ceramics for energy storage, microelectronics, EOIR and radiation sensors (Invited)

N. B. Singh*[1]; F. Choa[2]; B. Arnold[3]; L. Kelly[3]; K. Mandal[4]

1. University of Maryland Baltimore County, Chemistry and Biochemistry, and Computer Science and Electrical Engineering, USA
2. University of Maryland Baltimore County, Computer Science and Electrical Engineering, USA
3. University of Maryland Baltimore County, Chemistry and Biochemistry, USA
4. Indian Institute of Technology (BHU), Chemistry, India

Ceramic materials have made tremendous impact for high strength and high temperature applications. Their potential for electronic and optical systems has not been fully explored. It only recently binary, ternary and quaternary glassy and crystalline ceramic oxides, carbides and selenides are under exploration for multiple applications including high power lasers, detectors, dielectric energy storage and variety of optical devices. Material have been grown by variety of methods including Bridgman, physical vapor transport (PVT), chemical vapor transport (CVT) and flux methods in the form of bulk, thin film, nanocrystals and nanowires. For applications of nanoparticles, it is important to understand nucleation, phase transition grain growth, metastability and final morphology. We have performed experiments on multinary oxides and selenides using several growth methods to demonstrate nanoparticle and nanowire transition. Preliminary results show great promise for developing high energy storage, high power electronic and RF systems requiring high breakdown voltage, damage threshold, γ-ray detectivity, and high operating temperature IR detectors. Performance of nanoparticles, nanorods, nanowires and nanohexagons will be presented for variety of applications to demonstrate multifunctionality.

10:50 AM

(ICACC-S7-012-2020) Bioelectrochemical TiN|FDH Catalyst for CO_2 Reduction to HCOOH

F. Arena*[1]; G. Giuffredi[1]; S. Donini[1]; E. Parisini[1]; F. Di Fonzo[1]

1. Italian Institute of Technology, Italy

Among the many strategies proposed to convert CO_2 into value-added hydrocarbons involves its electrochemical conversion using BES. The advantage of this new technology lies in the possibility of exploiting the enzyme properties catalysing the reaction with a high selectivity and specificity toward products with a low overpotential applied. In this work, we present a novel BES where the FDH from Thiobacillus sp. KNK65MA is deposited on a TiN nanostructured realized by Pulsed Laser Deposition. We realize an electrode with tree-like morphology that maximizes the available surface area for catalyst absorption and enhances the bio-interface between enzyme and electrode. We quantify the amount of immobilized enzyme through enzymatic assays, demonstrating that the nanostructuration of the TiN increases the surface area available for enzyme immobilization, achieving a maximum enzyme adsorption of 59 $\mu g/cm^2$. The enzymatic electrosynthesis of formic acid from CO_2 is investigated by chronoamperometry at different applied potentials, showing a productivity for formic acid that ranges from 1.5 to 3.7 $mmol/mg_{enzyme}h$ according to the applied overpotentials. Unparalleled in previous studies, this performance achieved thanks to the high reducing activity of TsFDH and the high contact area offered by the TiN support and demonstrates the potential for a biotechnological device in terms offeaturing product specificity and stability.

11:10 AM

(ICACC-S7-013-2020) Composite Formation of Cs/$CH_3NH_3PbI_3$ Perovskite System and its Influence on Physical Properties

V. Pawar*[1]; P. Singh[1]

1. Indian Institute of Technology(BHU), PHYSICS, India

The stability issue is very critical for organic-inorganic lead halide perovskite ($CH_3NH_3PbI_3$ or $MAPbI_3$) to be utilized in solar cell applications. Whereas inorganic cesium lead halide perovskite ($CsPbI_3$) is reported to be more stable than that of $CH_3NH_3PbI_3$. In the present work, we have substituted the Cs^+ ion in the organic perovskite $CH_3NH_3PbI_3$ compound to form the $Cs_x(CH_3NH_3)_{1-x}PbI_3$ matrix with $0 \leq x \leq 0.4$ in ambient conditions i.e., at room temperature and in air via solid-state reaction route. The compound formation and its nature have been investigated through Rietveld refinement, thermo-dynamical, Raman and optical analysis. The bandgap alteration due to the solid solution and/or composite formation has also been explored.

11:30 AM

(ICACC-S7-014-2020) Spontaneous formation of Ternary Metal Oxide Nanomaterials from Cerium and High Valance Metallate precursors: VO_4^{3-} and MoO_4^{2-}

C. J. Neal*[1]; T. Sakthivel[1]; A. Jeyaranjan[1]; S. Seal[1]

1. University of Central Florida, Advanced Materials Processing and Analysis Center (AMPAC), Materials Science & Engineering, Nanoscience Technology Center (NSTC), USA

Cerium oxide nanomaterials have demonstrated great utility in the chemical and biomedical industries. In particular, the particles' ability to undergo redox cycling (Ce^{3+}/Ce^{4+}), along with formation/loss of oxygen vacancies, have afforded wide catalytic activity towards varied chemical substrates. Incorporation of higher redox state elements, such as vanadium or molybdenum, may allow chemical reactivity over an even wider chemical space. In this study, we report the spontaneous formation of nanomaterials from cerium and high valance transition metallate precursors in water. Particle composition and vacancy-character were determined via X-ray photoelectron spectroscopy. Particle size and morphology were assessed via hrTEM. Further, the influence of varying reactant ratios (e.g. 1:1, 2:1, and 3:1; cerium to molybdate/vanadate) was also assessed with respect to these properties and a strong dependence was evidenced. From here, the nanomaterials were assessed for enzyme-mimetic activities in catalase, superoxide dismutase, and hydroxyl radical assays to determine their potential for use in biomedical applications (e.g. via interaction with or production of free radical species). The synthesis method used will be further evaluated in future studies for generalizability over other metallates for the formation of cerium-metallate, ternary oxides.

Metal Oxide Nanostructures for Sensing, Batteries, and Water-splitting Applications

Room: Flagler A

Session Chair: Yakup Gönüllü, University of Cologne

1:30 PM

(ICACC-S7-015-2020) Gas sensing of NiO-SCCNTs core-shell heterostructures: Optimization by radial modulation of the hole-accumulation layer (Invited)

N. Pinna*[1]

1. Humboldt-Universität zu Berlin, Department of Chemistry, Germany

Hierarchical core-shell (C-S) heterostructures composed of a NiO shell deposited onto stacked-cup carbon nanotubes (SCCNTs) were synthesized using atomic layer deposition (ALD). A controlled film of NiO particles was uniformly deposited onto the inner and outer walls of the CNTs. The as-synthesized NiO-SCCNTs C-S heterostructures were thoroughly characterized by high-resolution transmission electron microscopy (HRTEM), energy dispersive X-ray (EDX) elemental mapping and X-ray photoelectron spectroscopy (XPS). The behavior of NiO-SCCNTs sensors with various thicknesses of the NiO shell layers was investigated for low concentrations of acetone and ethanol at 200 °C. The sensing mechanism is based on the modulation of hole-accumulation region in the NiO shell layer, during the interaction of the reducing gas molecules with the adsorbed oxygen species. The electrical conduction mechanism was further studied by developing NiO-Al_2O_3-SCCNTs heterostructures with the incorporation of Al_2O_3 dielectric layer in between the NiO and SCCNT interfaces. It suggested that the sensing mechanism is strictly related to the NiO shell layer. The remarkable performance of NiO-SCCNTs sensors towards acetone and ethanol benefits from the conformal coating by ALD, large surface area by SCCNTs and the optimized p-NiO shell layer thickness.

2:00 PM

(ICACC-S7-016-2020) Shape-controlled Ce-Ti oxide systems for photocatalytic applications in the CO preferential oxidation

E. Moretti*[1]; A. Infantes-Molina[2]; E. Rodriguez-Castellon[2]; A. Talon[1]; A. Vomiero[3]

1. Ca' Foscari University of Venice, Department of Molecular Sciences and Nanosystems, Italy
2. University of Málaga, Department of Inorganic Chemistry, Crystallography and Mineralogy, Spain
3. Lulea University of Technology, Engineering Sciences & Mathematics, Sweden

Inorganic nanostructures with well-defined shapes have aroused in recent years a wide interest both in the academic and industrial fields because of their size/shape dependent properties and their potentials of self-assembly. In particular, the investigation of CeO_2-based materials, rationally designed with a controlled morphology at the nano/microscale, is a research hotspot for environment and energy related applications. The present work aims to investigate the photocatalytic behavior of Au nanoparticles (1.0 wt% nominal loading) supported on CeO_2-TiO_2 matrices with a flower-like morphology in the CO preferential oxidation in excess of H_2 under simulated solar light irradiation (photo CO-PROX), assessing not only the role of each component in the system and on the catalytic response, but also how a peculiar morphology can affect the photocatalysis. CeO_2 samples containing different TiO_2 loadings were synthesized by a surfactant-free and environmentally friendly slow co-precipitation method. As shown by SEM microscopy, the samples appeared organized in a hierarchical structure comparable to a microscale size flower. HRTEM revealed the presence of 2-4 nm size Au NPs, homogenously distributed on the support surface. The Au/CeO_2-TiO_2 systems showed a morphology dependent behavior in the photo CO-PROX, resulting much more active than samples with a non-organized structure.

2:20 PM

(ICACC-S7-017-2020) Engineering Interfacial Modification on Nanocrystaline Hematite Photoanodes: A Close Look into the Efficiency Parameters (Invited)

D. N. Muche[1]; F. L. de Souza*[1]

1. Federal University of ABC, Center of Natural Science and Humanity, Brazil

This work describes an approach to direct evaluate the effects onto efficiency parameters by modification of hematite photoanode with thickness from 25 up to 130 nm. The starting point was given by the study of the effect of thickness for non-modified hematite photoanodes. Results shows the decreasing of η_{global} as the thickness increases caused by charge recombination due the presence of bulk. The thinnest and the thickest photoanodes were then chosen for first modification, which is the addition of Sn^{4+} in 2 different concentrations, 1 and 3% molar ratio, into the polymeric iron based solution. Photoelectrochemical investigations showed the role of Sn^{4+} as responsible for an enhanced electronic transport however with a

cost on creation of surface states. The selected modified hematite photoanode containing Sn^{4+} were submitted to Ni/Fe based material photoelectrodeposition which was responsible for passivation of such created surface states. With a fixed J_{abs}, η_{sep} and η_{cat} efficiencies showed that the detected hematite electronic problems are dominant and the tentative to improve such limitation can directly change the reactivity o the surfaces. Moreover, the results show that interfacial traps are mitigated for the ultrathin photoanodes, where the presence of Sn^{4+} and Ni/Fe significantly increases the η_{global} highlighted bysample H-01-1Sn-NiFe.

2:40 PM

(ICACC-S7-018-2020) Phase Selective Synthesis of $InFeO_3$ using Single Source Precursors

V. Nahrstedt*[1]; J. Januškevičius[2]; F. Maccari[3]; O. Gutfleisch[3]; S. Mathur[1]

1. University of Cologne, Institute of Inorganic Chemistry, Germany
2. Vilnius University, Lithuania
3. TU Darmstadt, Germany

Nanoscaled ternary oxides are of great interest with respect to their superior catalytic, magnetic and electro optical properties. Especially the ternary indium ferrite is a promising material for magneto resistive storage, although it is not well investigated so far. $InFeO_3$ is isostructural with $InMnO_3$ and often formed at high temperatures, showing phase transitions towards $LiNbO_3$-type structure at 15 GPa and 1000 °C. The formation of the indium ferrite $InFeO_3$ was achieved by thermal decomposition of heterometallic indium iron alkoxides. The phase purity was proofed by XRD measurements, SEM images revealed the generation of nanoparticles. Additionally, the magnetic properties of those particles were proofed by magnetic measurements.

Synthesis, Functionalization, and Assembly of Inorganic and Hybrid Nanostructures II

Room: Flagler A

Session Chair: Nicola Pinna, Humboldt-Universität zu Berlin

3:20 PM

(ICACC-S7-019-2020) Bioconjugated Nanoprobes: Tumor Specific Uptake and Localization (Invited)

S. Ilyas*[1]; K. Wennhold[2]; S. Hussain[3]; S. Mathur[1]

1. University of Cologne, Institute of Inorganic Chemistry, Germany
2. University Hospital Cologne, Center for Molecular Medicine Cologne (CMMC) and Cologne Translational Immunology, Germany
3. University of Cologne, Institute of Biochemistry I, Germany

Multivariate conjugation for efficient transport and tracking of nanoparticles (NPs) cross the cancer cell membrane. Specific uptake of delivering a drug or radiopharmaceutical-loaded particles is crucial for not affecting healthy non-targeted tissues or organs. The study focuses in terms of bioconjugationstrategies; e.g. click/carbodiimide, which offered an efficient way to covalently attach different ligands and targeting moieties to NPs surface. Availability of different functionalities on NPs make the conjugate attractive for the attachments of several biomolecules. The model nano-bioconjugate developed here contains folic acid as the vector unit and doxorubicin as fluorescent anti-cancer drug that facilitates localization of NPs delivery into the cells. Along standard viability trends for the cancer cells at high doses the nanoconjugates showed efficient intracellular uptake and delivery in folate receptor overexpressing cell lines. Mice models demonstrated preferential nano-accumulation at the tumor site validating robust drug delivery chemical design. The NPs trivially localized in healthy organs which certainly directs an elevated therapeutic index NPs used. Different analysis methods revealed distinctive variations after the attachments of different ligands and molecules on particles. The talk will present how NPs can be transformed into site-selective nanovectors for breast cancer research.

3:50 PM

(ICACC-S7-020-2020) Superhydrophobic Functional Surfaces

A. K. Schmidt-Verma*[1]; A. Renner[1]; S. Mathur[1]

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

Control over surface properties and chemical structure is mandatory for the development of new materials which can help to increase e. g. solar energy conversion or reduce maintenance costs of already established devices. In this regard, superhydrophobic ceramic coatings are crucial for eco-friendly materials processing due to their self-cleaning properties, offering a green alternative to the usage of aggressive cleaning agents. Although the potential of commercial product is huge and their market truly global, the wide range of possible application is limited by the low temperature stability of these coatings. In this presentation, a superhydrophobic coating with high temperature stability will be presented. Spinel nanoparticles were prepared through sol-gel method on glass substrates from the bimetallic alkoxide $[Mg\{Al(O^iPr)_4\}_2]$ followed by subsequent chemical functionalization. The coating of functionalized magnesium aluminate nanoparticles produced transparent, superhydrophobic films with contact angle for water close to 180° after temperature treatment.

4:10 PM

(ICACC-S7-021-2020) Cost efficient oxygen generation through alkaline water electrolysis using Ni on SnO_2 mesoporous support-based electrocatalysts

F. Neatu*[1]; S. Neatu[1]; V. Diculescu[1]; M. M. Trandafir[1]; N. Petrea[2]; S. Somacescu[3]; F. Krumeich[4]; A. Knorpp[4]; J. van Bokhoven[4]; M. Florea[1]

1. National Institute of Materials Physics, Romania
2. Scientific Research Centre for CBRN Defence and Ecology, Romania
3. "Ilie Murgulescu" Institute of Physical Chemistry, Romanian Academy, Romania
4. ETH Zurich, Institute for Chemical and Bioengineering, Switzerland

The increasing worldwide energetic demands in the last decades push humankind to seek for alternatives to the actual environmental unfriendly carbon-based energy production processes.[1] Alkaline water electrolysis, fuel cells and metal-air batteries are among the most studied energy conversion and storage systems.[2] Herein, the synergistic behavior of Ni species and bimodal mesoporous SnO_2 is investigated in oxygen evolution reaction under alkaline conditions without any other modification of the compositional phases or using noble metals. A hydrothermal method to prepare mesoporous undoped SnO_2 with very high surface area (>130 m^2g^{-1}) and a deposition-precipitation methodology of well-dispersed Ni-species on undoped SnO_2 is reported. The powders have been characterized by BET, TG-DTA, XRD, TEM, Raman, TPR-H_2, and XPS. The best NiSn composite generates, under certain experimental conditions, a very high TOF value of 1.14 s^{-1} and a mass activity higher than 370 A g^{-1}, which are remarkable results considering the low amount of Ni deposited on the electrode (3.78 ng). This performance is due the dual role of SnO_2: i) as support for active and well dispersed Ni species and ii) as an active player through oxygen vacancies generated upon Ni deposition.

4:30 PM

(ICACC-S7-022-2020) Oxide based high temperature thermoelectric devices: Multilayer fabrication and performance

F. R. Caliari*[1]; S. Sampath[1]

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

Current thermoelectric materials are highly toxic, expensive and scarce. Those high ZT materials lose their TE properties upon exposition to high temperature air atmosphere, demanding complex steps on the TEG fabrication. Transition metal oxides (TMO) are good candidates for thermoelectric (TE) applications in this context. Thermal spray (TS) stands out as an alternative process route,

enabling the processing of these high temperature materials under either reducing or oxidizing environment, and thus providing in-situ reactions and control of the oxide stoichiometry. In this study plasma sprayed TiO_{2-x} and Ca2Co2O5 are used as n-type and p-type materials. In addition, multilayer strategy allows unique capabilities of plasma spray to assemble thermoelectric system which can directly be applied on heat engine components for waste heat power harvesting.

4:50 PM

(ICACC-S7-023-2020) Elegant Fabrication of Boron Nitride Aerogels Consisting of Varied Superstructures

J. Pan*[1]; J. Wang[1]

1. Chinese Academy of Sciences, Institute of Metal Research, China

Among the most important inorganic constituents, boron nitride (BN) and its corresponding nanostructures have garnered increasing attention. As a porous material with a nanoscale skeleton, aerogel allows the bridging of nano- and macro-world, and implies unexpected properties arising from the combination of multidimensional features. Accordingly, the existence of BN in aerogel form is expected to exhibit even more extraordinary properties, whereas the advance of BN aerogel is extremely slow because of the chronic difficulty in fabrication. Herein presented is the elegant preparation of BN aerogels with tailorable microstructures and some preliminary explorations around their properties. Given the significance of fabrication and the dependence between microstructures and functional properties, this report is also meant to engage the larger community by asking: Can the BN aerogels with unique structural hierarchy find more other applications? And is it possible to apply the fabrication principle for the design of other materials with complex structures?

S8: 14th International Symposium on Advanced Processing and Manufacturing Technologies for Structural and Multifunctional Materials and Systems (APMT14)

Advanced Sintering Technologies II

Room: Coquina Salon A

Session Chairs: Jon Binner, Loughborough University; Tadachika Nakayama, Nagaoka University of Technology

8:30 AM

(ICACC-S8-011-2020) Pressureless flash sintering of α-SiC using solid state sintering aids (Invited)

A. Gibson[1]; Y. Li[1]; R. I. Todd*[1]

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

In flash sintering, high density can be achieved in significantly shorter times and with lower furnace temperatures than in conventional sintering by passing an electric current through the sample during densification. Most work on flash sintering to date has been on oxides, but rapid sintering using lower furnace temperatures is of potentially great benefit in non-oxide ceramics such as SiC, which require very high furnace temperatures (>2100 °C) for conventional sintering. This presentation describes the pressureless flash sintering of a-SiC to high density using boron- and carbon-containing solid state sintering aids. Sintering can be achieved in several minutes with furnace temperatures of only ~1500 °C. The electrical response of the specimens is described and is shown to be more dependent on extrinsic factors than is the case with oxide ceramics, where the behaviour is dominated by the innate conductivity of the ceramic. Microstructural development is investigated and the possibility of producing dense, fine grained microstructures with high hardness is demonstrated. The practical challenges of electrical supply and thermal management are discussed.

9:00 AM

(ICACC-S8-012-2020) Aligning α-Alumina Platelets via Uniaxial Pressing of Ceramic-filled Thermoplastic Polymer Blends for the Improvement of Final Sintered Transparency

W. J. Costakis*[1]; A. Schlup[1]; R. Trice[1]; J. P. Youngblood[1]

1. Purdue University, Department of Materials Engineering, USA

Transparent alumina is a candidate material for ballistic applications were visible or infrared wavelength transmission is required. However, the transparency of polycrystalline alumina can be limited due to its rhombohedral crystal structure being inherently birefringent. Birefringence causes light scattering at misaligned grain boundaries and is detrimental to the transparency. It has been shown experimentally, that the application of a high magnetic field during processing can lead to crystallographic alignment and the reduction of birefringent light scattering. This alignment method is effective but may be limited in terms of scalability. It is proposed that the use of shear and elongational forming processes can be a practical means to align axisymmetric α-alumina platelets for the improvement of final sintered transparency. Thermoplastic polymer and α-alumina platelet blends where developed and formed into sheets through uniaxial warm pressing. The effects of α -alumina platelet diameter, solids loading and rheological properties on the final platelet orientation were investigated via rocking curve analysis. Optimal samples produced with 30 vol.% 11μm diameter alumina platelets demonstrated an average orientation parameter (r) and grain misalignment alignment (FWHM) of 0.25 and 11.16°, respectively.

9:20 AM

(ICACC-S8-013-2020) Study of 3D printing of transparent ceramics

G. Zhang*[1]; D. Carloni[1]; Y. Wu[1]

1. Alfred University, Department of Materials Science, Kazuo Inamori School of Engineering, USA

Transparent YAG ceramics have been fabricated by a 3D printing method. Printed green body was shaped into gear shapes by using aqueous viscous slurry. After a slow debinding process, the green body could be sintered into transparent ceramics by a vacuum sintering. The work demonstrated transparent ceramics can be prepared using a simplified process without traditional cold isostatic pressing procedure. It also enables flexibility of designing and processing any desired shape for transparent ceramic without a mold. It will serve as s potential method of making customized transparent ceramic components and it is also useful for fabricating ceramics structure with a full densification.

9:40 AM

(ICACC-S8-014-2020) Microstructures Ceramic Designing by Voronoi Model and Fractals Geometry

V. Mitic*[1]; G. Lazovic[2]; D. Rancic[3]; I. Antolovic[3]; Z. Nikolic[4]; H. Fecht[5]

1. Serbian Academy of Sciences /Faculty of Electronic Engineering University Nis, Institute of Technical Sciences, Serbia
2. University of Belgrade, Faculty of Mechanical Engineering, Serbia
3. University of Nis, Faculty of Electronic Engineering, Serbia
4. University Nis, Serbia
5. University Ulm, Germany

The Voronoi model application idea has been based on the evident similarity of observed surface land morphology from the space distances watching with the looking grain surfaces structures on microstructure level. The Voronoi diagram is a collection of geometric objects is a partition of space into cells, each of which consists of the points closer to one particular object than to any others, including models of crystal, cell and grain growth Considering morphology shapes properties, method of Voronoi tessellation has been used for grains surface modeling. . In the experiments, we apply statistical approach to determine the influence of different temperature levels of sintering (1320 -1380°C) on the size

of contact area for different additives doped BaTiO3 –. All these steps by fractals are part of a new grain and pore structure characterization methods, We originally combine Voronoi model and fractals, which, give much more natural approximation to physical advanced technology structures than Euclidean geometry. This means that processes' parameters may have fractal behaviour in time, for ex. changing of energy and motion of particles (Brownian walk), also.

10:20 AM

(ICACC-S8-015-2020) Numerical simulation of water molecule coordinates in $Sr_2CaCu_2O_y$ superconductor (Invited)

H. Suematsu*[1]; K. Kawai[1]; A. Fujimoto[2]; T. Do[1]; T. Nakayama[1]; K. Niihara[1]

1. Nagaoka University of Technology, Extreme Energy-Density Research Institute, Japan
2. National Institute of Technology, Numazu College, Japan

$Sr_2Ca_{(n-1)}Cu_nO_y(0^{(Sr)}2(n-1)n)$ superconductors have unique characteristics: they absorb carbon dioxide and water molecules to elongate the c-paramters and still exhibit superconductivity[1]. Furthermore, after the water molecule absorption, the critical current density and irreversibility magnetic field increase[2]. However, their water molecule absorption sites have not been clarified. In oxide catalyst research, to visualize the organic molecule absorption sites, MOPAC has been used. In this work, MOPAC calculations were carried out to investigate the water molecule absorption sites in a $0^{(Sr)}212$ crystal. One to four water molecules were inserted in between the SrO double layers in the $0^{(Sr)}212$ crystal. The heat of formation was calculated with water molecule(s) sitting at a distance along <110> from the center of the SrO double layers. This calculations were repeated with increasing the distance. The maximum c-parameter elongation of 0.147nm was obtained for four water molecules absorption. This value is smaller than but comparable to that of the experimentally obtained c/2 parameter elongation of 0.325 nm[1]. As far as the author's knowledge, this is the first attempt to calculate molecule intercalation in an oxide crystal.

10:50 AM

(ICACC-S8-016-2020) Microstructure and anisotropic mechanical properties of $B_{6.5}C$-TiB_2-SiC-BN composites fabricated by reactive hot pressing (Invited)

W. Wang*[1]; Q. He[1]; H. Wang[1]; Z. Fu[2]

1. Wuhan University of Technology, China
2. Wuhan University of Technology, State Key Lab of Advanced Technology for Materials Synthesis and Processing, China

$B_{6.5}C$-TiB_2-SiC-BN composite ceramics were prepared by a novel solid state reaction using TiCN, B and Si as the raw materials. The final products with fine microstructure, homogeneous distribution and excellent mechanical properties were obtained via hot pressing at 1950 °C. Due to the orientation growth and structural characteristics of TiB_2 and h-BN grains, the anisotropy of mechanical properties of the composites was caused. When the test surface is perpendicular to the applied pressure direction, superior performance BN-containing composites with a hardness, bending strength, and fracture toughness of 22.40 GPa, 801 MPa and 5.31 $MPam^{1/2}$ were obtained. In addition, the formation of twin structures in $B_{6.5}C$ and SiC grains is beneficial to the mechanical properties.

11:20 AM

(ICACC-S8-017-2020) Electric field-assisted softening and crystallization in glasses

S. S. Parihar*[1]; K. T. Strong[2]

1. Sandia National Laboratories, USA
2. Sandia National Laboratories, Material Mechanics and Tribology, USA

Application of electric field can result in appreciable decrease in softening point of glasses. Electrical field can also change the nucleation and crystallization behavior of glass. In this study, a group of lithium containing glasses were examined under different temperatures, and electric field conditions. Effect of electric field on softening temperature, nucleation temperature, and morphology of crystalline phases will be presented. Effect of glass composition on the efficacy of the electric field in softening temperature reduction will be discussed. Sandia National Laboratories is a multi-program laboratory managed and operated by National Technology & Engineering Solutions of Sandia, LLC (NTESS), for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525. SAND NO. 2019-####.

11:40 AM

(ICACC-S8-018-2020) Sintering of advanced ceramics by plastic deformation as dominant mechanism

W. Ji*[1]; Z. Fu[2]

1. Wuhan University of Technology, China
2. Wuhan University of Technology, State Key Lab of Advanced Technology for Materials Synthesis and Processing, China

Dense and fine grain structure is the goal of ceramic sintering. However, common sintering processes with high sintering temperature and long soaking time lead to inevitable grain growth. A new ceramic sintering approach employing plastic deformation as the dominant mechanism is proposed, at low temperature close to the onset point of grain growth and under high pressure. High performance boride ceramics with full density and limited grain growth were fabricated based on the technology. This idea and method provide both time and energy efficient ways for borides, and also facilitate preparation of other advanced ceramics such as nano-ceramics for practical applications.

Advanced Manufacturing and Processing I

Room: Coquina Salon A

Session Chairs: Jianfeng Yang, Xi'an Jiaotong University; Weimin Wang, Wuhan University of Technology

1:30 PM

(ICACC-S8-019-2020) Development of hybrid machining technique to process complex shape ceramics with very fine details (Invited)

F. J. Cambier*[1]; F. Petit[1]; C. Duterte[2]; A. Biernaux[2]

1. Belgian Ceramic Research Centre, Belgium
2. Optec SA, Belgium

Ceramics (mainly zirconia) are more and more used in the luxury applications. Particularly, zirconia made bezels is almost common in the watch industry. As a consequence, companies are continuously looking for new designs and for techniques allowing both to reduce the manufacturing costs while achieving more and more detailed and/or complex parts. Most of such complex parts (but not all) can be produced by injection molding, so far the number of parts is enough high, because the need of amortizing a very expensive mold. At the prototype stage, additive manufacturing techniques (i.e. stereolithography, SLS, binder jetting, etc.) are worldly envisaged to produce highly complex parts, however such methods are not yet really industrialized. By comparison subtractive technique is widely sprayed through industry, for instance, teeth crowns are fabricated by classical milling of a pre-sintered ceramic, starting from a digital model. More recently, machining by laser of oxide ceramics at the green state has been developed thanks to a proprietary process. All the techniques cited above have advantages and drawbacks which will be reviewed. To optimizing both accuracy and create new designs, hybrid methods are used. The presentation will show the development of both a new technique and new equipments allowing achieving very original designs.

2:00 PM

(ICACC-S8-020-2020) Advanced ceramic and composite electrodes for supercapacitors with high active mass loadings (Invited)

I. Zhitomirsky*[1]

1. McMaster University, Canada

The goal of this study was the development of advanced ceramic and composite electrodes for high-power supercapacitors with high active mass loading, high areal and gravimetric capacitances, good cyclic stability and low impedance. The approach was based on nature inspired strategies for the surface modification and colloidal processing of nanomaterials. Chelating organic molecules with strong polydentate bonding to metal oxide surface were used as capping and dispersing agents, which allowed excellent control of nanoparticle size and dispersion. The strategies for the dispersion of CNT and graphene were based on the use of commercial bile salts and organic dyes. Enhanced performance was achieved by the use of chelating polymers and complexes for the colloidal processing of multicomponent systems. Various heterocoagulation techniques and liquid-liquid extraction methods were developed for the design of advanced nanocomposites. New strategies were used for the fabrication of MnO_2-CNT, Mn_3O_4-CNT, FeOOH-CNT composites for negative and positive electrodes. An important discovery was the high capacitive properties of multiferroic $BiMn_2O_5$ and V_2O_3 material with metallic conductivity. Efficient supercapacitor electrodes and devices were developed with areal capacitance as high as 8 F cm^{-2}. The electrodes showed good power-energy characteristics, low impedance and cyclic stability.

2:30 PM

(ICACC-S8-021-2020) Novel electrical disintegration for selective dismantling between multi-material (Invited)

C. Tokoro*[1]

1. Waseda University, Japan

Nowadays, demand for not only availability of lightweight materials and reliable jointing technologies, but also cost-efficient dismantling technology suitable for multi-material are increasing. We developed novel electrical disintegration which enabled selective heating, selective reaction, selective peeling and selective disintegration by precise control of discharge path, waveform of voltage/current, and reputation time/frequency. We created a test pieces which consisted of Fe plate, Al plate and/or CFRP (carbon fiber reinforced plastics) plate and bonded by three kinds of bonding agent of epoxy-type, urethane-type or silicon type, and confirmed these bonding could be completely separated by the novel electrical disintegration. Fe and Al plates were not damaged and enough to be reused after the disintegration. Furthermore, the novel electrical disintegration technology could be applied to the selective separation of other multi-materials. For example, positive electrode particles were successfully separated from aluminum foil in lithium ion battery, and Cu/Ag wire could be separated from the EVA (ethylene-vinyl acetate) sheet in photovoltaic cell. We believe these separation characteristics by novel electrical disintegration could create a new reuse and recycling loop toward building sound material-cycle society.

3:20 PM

(ICACC-S8-022-2020) Deposition Behavior of Cesium Molybdate on Stainless Steel 316 (Invited)

T. Do[1]; X. H. Hoang[1]; T. Nakayama*[2]; H. Suematsu[3]

1. Nagaoka University of Technology, Nuclear System Safety Engineering, Japan
2. Nagaoka University of Technology, Japan
3. Nagaoka University of Technology, Extreme Energy-Density Research Institute, Japan

After the Fukushima Daiichi nuclear accident, the study of the behavior of cesium molybdate (Cs_2MoO_4) inside the reactor received great attention. When Cs_2MoO_4 exists as a gas phase, both Cs and Mo are be diffused and implanted into the structure material. If Cs_2MoO_4 exists as a solid phase, it will deposit on the surface of the reactor coolant system (RCS) and it may cause some interactions with the material of RCS such as stainless steel. In this study, the reaction between Cs_2MoO_4 and SUS in both cases will be analyzed. Cs_2MoO_4 was heated at 1530 K in a siliconit furnace. SUS316 pieces were placed at different positions corresponding with the temperature at 1550, 1500, 1230, 730 and 550 K. The experiment was carried out in Ar gas for 20 minutes. A similar experiment but without Cs_2MoO_4 was performed to have the references for the SUS oxide layers. Surfaces of SUS316 were examined by XRD and micro-Raman. Cr_2O_3 was detected in the case of heating SUS without Cs_2MoO_4. However, Cr_2O_3 was not identified in the case of heating with Cs_2MoO_4. This result suggests that there could be some reactions between Cs_2MoO_4 and SUS316 at high temperature. The results will be used for the understanding of the nuclear severe accident

3:50 PM

(ICACC-S8-023-2020) NDK and HDK composite networks for optimization of dielectric behavior

K. B. Häuser*[1]; J. R. Binder[1]; P. Agrawal[2]; R. Jakoby[2]

1. Karlsruhe Institute of Technology, Institute for Applied Materials (IAM), Germany
2. Technical University Darmstadt, IMP, Germany

HDK materials, such as $Ba_{1-x}Sr_xTiO_3$ (BST), play a central role in devices such as ceramic varactors or resonators, oftentimes due to their tunability. Tunability denotes a DC-bias controllable permittivity, which allows for fast matching of capacity or resonance frequency. Unfortunately, these materials often feature relatively high dielectric loss and high base permittivity, leading to extensive heat development in high power applications and limited efficiency when trying to couple into a resonator. Composites made out of these HDK materials and NDK materials, for example $Mg_3B_2O_6$ (MBO), feature lower permittivities and dielectric loss. This comes at the price of reduced tunability, since the displacement of the electric field into the NDK material decreases the effective tuning field strength in the HDK material. This leads to a percolating behavior of tunability over NDK percentage, since the formation of NDK pathways almost nullifies the tunability of the composite. Therefore, the creation of structured HDK/NDK composites has great potential to improve device properties, if the percolation of the NDK material is prevented. These can be achieved via realization of core-shell granulates, which are made with a physical structuring process during spray drying, using granular convection. Further improvement can be made with introduction of a third, conductive phase, leading to a microcapacitor network.

4:10 PM

(ICACC-S8-024-2020) Structural change analysis of cerium and yttrium minerals in weathered residual rare earth ore by mechanochemical reaction

T. Kato*[1]; C. Tokoro[2]

1. Waseda University, Graduate School of Creative Science and Engineering, Japan
2. Waseda University, Japan

Prolonged high intensity grinding can modify the crystal structure of solid substances and/or induce chemical reaction, called as mechanochemical reaction. Since such reactions can exert positive influences on hydrometallurgical processes, the hydrometallurgical process based on mechanochemical reaction to enhance metal dissolution from minerals/ores have been recently attracting much attention. Recently, the mechanism of mechanochemical reaction has been investigated using solid analysis and simulations by many researchers. On the other hand, the structural changes caused by mechanochemical reactions are not yet sufficiently clarified because the ground samples are amorphous. The objective of this study was to clarify structural changes of cerium and yttrium minerals in weathered residual rare earth ores

by mechanochemical reaction. The ore was ground by planetary ball milling with and without solid sodium hydroxide. To achieve this, we performed x-ray absorption fine structure (XAFS) analysis for ground samples at the cerium L_{III}- and K-edges and yttrium K-edge. Based on extened x-ray absorption fine structure analysis, it was revealed that the structural change of cerium and yttrium minerals in the weathered residual rare earth ores by mechanochemical reaction.

4:30 PM

(ICACC-S8-025-2020) Design of Lightweight and Durable Conductor for Electrical Propulsion

A. J. Goretski*[1]; A. S. Almansour[2]; A. Gorven[3]

1. Mississippi State University, USA
2. NASA Glenn Research Center, Mechanical Engineering, USA
3. Boise State University, Mechanical Engineering, USA

Development of high conductivity, lightweight and durable conductive composite wires are required for enabling Electrified Aircraft Propulsion (EAP). Therefore, modeling, designing, fabricating, testing and characterizing the material behavior of metalized yarns were done here in the interest of creating lightweight, durable power transmission cables. Electroplating was used to create the metal conductor coating on yarns. A multi-yarn frame was designed and additively manufactured using 3D printing of acrylonitrile butadiene styrene (ABS) to help create multiple composite wires and ensure that the plating solution would not react with the yarns' frame and focus the ion transfer to the yarns. The process of plating required experimentation with time and electrical current to reach the desired thickness and uniformity of the coating. Electroplating processing parameters were optimized and tensile testing was conducted along with electrical conductivity and acoustic emission monitoring on multiple wires from each of the plating batches, with and without heat treatment of the samples. Finally, imaging with scanning electron microscopy (SEM) was performed to assess the samples' microstructures.

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

Alloys and Compounds : New Concepts and Emerging Technologies for Enhanced Product Performance

Room: Tomoka B

Session Chairs: Hyuksu Han, Hongik University; Sungwook Mhin, Korea Institute of Industrial Technology

9:00 AM

(ICACC-S11-011-2020) Microstructural segmentation and phase fraction of carbon steel weldment analysis using deep learning

S. Lee*[1]; J. Jang[1]; D. Van[1]; J. Park[1]; J. Kim[1]

1. Korea Aerospace University, Republic of Korea

Phase fraction of microstructure in the weldment of carbon steel is known to have a great influence on the mechanical properties. Accordingly, a variety of methods for quantitative measurement of phase have been devised, and technical standards such as ASTM E562 and ISO 9042, and IIW have selected point analysis as the most efficient method for measuring fraction of specific phases in metal microstructures. However, this method is time-consuming and requires professional techniques. Also, large difference between measurement and analysis results varies considerably depending on operators. In this study, the phase fraction of acicular ferrite in microstructure of carbon steel weldment was measured using deep learning image segmentation. A microstructure image dataset was constructed from a weld specimen made of A516-60 carbon steel through SAW process. ResNet, one of the latest and most efficient CNN-based classification model by Microsoft, based Fully Connected Network (FCN) was trained to perform the segmentation of microstructures. In addition, the phase fraction of the acicular ferrite predicted by the model was measured and compared with the actual phase fraction. The deep learning model presented in this paper is expected to segment chosen phases and quantitatively measure fractions in microstructures with less time and effort than conventional methods.

9:20 AM

(ICACC-S11-012-2020) Direct observation of alumina slurry under applying shear field by optical coherence tomography (Invited)

J. Tatami*[1]; H. Takaba[1]; M. Iijima[1]; T. Takahashi[2]

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

Dispersion of fine ceramic particles in a liquid is a key technology in ceramic processing. In this study, we directly observed the alumina slurry under shear field using optical coherence tomography. Aqueous alumina slurries with different pH value were prepared by a ball milling technique. Swept-source optical coherence tomography (SS-OCT) was used to observe the internal structure of the slurry. The SS-OCT was set below a rotary rheometer consisting of a stainless steel cone and a sapphire plate. At this time, the shear rate was increased from 0 to 150 s^{-1} at 1 s^{-1} per second. In the slurry with low pH value (high zeta potential), it was observed that many bright spots were fluctuated resulting from the Brownian motion of the fine particles. The movement did not change by increasing the shear rate. On the other hand, the internal structure of the slurry having pH value near isostatic point was stationary before applying shear rate. As the shear rate increased, it collapsed from near the stainless steel cone, and the size of the bright spot decreased, which means that the network structure of the agglomerates was change into dispersion of the fine particles.

9:50 AM

(ICACC-S11-013-2020) High temperature fluidized bed spray coating – coating and calcination / reduction / oxidation or phase transformation in one step

A. Teiwes[1]; K. Weber*[1]

1. Glatt Ingenieurtechnik GmbH, Germany

The goal of particle coating processes is to improve product performance by changing physical and chemical particle properties, adding surface activity to carrier particles for e.g. heterogeneous catalysis or adsorption. A combination of coating and high temperature thermal treatment in only one process step has not been available so far. Glatt Ingenieurtechnik GmbH has developed a very flexible high temperature fluidized bed system with adjustable temperatures up to 700 °C and simultaneous injection of liquids. So a particle coating and a chemical reaction may be done simultaneously or in sequence, as required. The concepts behind the powerful technology will be presented as well as exemplary use cases. Based on a concrete example, it will show the novel processes flexibility. The influence of different process and formulation parameters on coating mechanisms, yield and material properties will be shown for the application of manganese oxide, formed from manganese nitrate, coated on alumina oxide carriers. As analytical methods XRPD, REM, DSC/TGA and ICP-OES were used to particularize the newly formed material and find the best process parameter.

10:30 AM

(ICACC-S11-014-2020) Development of room-temperature formable Mg alloy sheets with high strengths (Invited)

T. Nakata*[1]; H. Ohashi[1]; S. Kamado[1]

1. Nagaoka University of Technology, Mechanical Engineering, Japan

We will report the recent development of room-temperature formable Mg alloy sheets with high strengths. A Mg-Al based alloy sheet exhibits large Index Erichsen value of 7.1 mm and moderate

0.2 % proof stress of 120 MPa. Upon an aging, the 0.2% proof stress increases to 150 MPa. Also the alloy sheet shows small yield anisotropy, which is a major hinderance of acceptance of wrought Mg alloys. Such properties could be obtained due to the formation of weak basal texture and fine grain structure. We have also developed a Mg-Zn based alloy sheets which can be substantially strengthened by an aging. A Mg-6Zn (mass%) based alloy sheet shows good Index Erichsen value over 7 mm after a solution-treatment. Also, after the aging, the Mg-6Zn based alloy sheet exhibits high 0.2 % proof stress of 263 MPa due to dispersion of fine b′ phases. These alloy sheets show comparable strengths and room-temperature formability as those in 6xxx series Al alloy sheets, indicating that our finding will broaden the application of wrought Mg alloy sheets in automotive industry.

11:00 AM

(ICACC-S11-015-2020) Effect of Gd and Y contents on the age-hardening response and tensile properties of extruded Mg-Gd-Y based alloys

Y. Mori*[1]; T. Nakata[1]; S. Kamado[1]

1. Nagaoka University of Technology, Mechanical Engineering, Japan

Mg-Gd-Y based alloys are expected to be applied for impellers of turbochargers, because they are strong and keep their high strengths even at elevated temperatures. The age-hardening response and tensile strengths of cast Mg-Gd-Y based alloys can be improved by increasing Gd and/or Y contents. However, strengthening of extruded Mg-Gd-Y based alloys cannot be simply achieved via the additions of Gd and/or Y elements. In this work, we have tried to optimize Gd and Y contents for the development of ultra high strength Mg-Gd-Y extruded alloys, and found that Gd and Y contents over 4.2mol% does not lead significant increase in the strengths and decrease in the quantities of age-hardening. This is because, during the extrusion, excessive additions of Gd and Y elements lead to the formation of coarse $Mg_5(Gd,Y)$ phases at grain boundaries. Such dynamic precipitation results in low dissolution of the alloying elements into Mg matrix and decrease of age-hardenability after the extrusion. These results will help to optimize the contents Gd and Y elements for extruded Mg-Gd-Y based alloys with high strengths.

11:20 AM

(ICACC-S11-016-2020) Enhancing room-temperature formability of a Mg-Al alloy sheet via micro-alloying

H. Ohashi*[1]; T. Nakata[1]; S. Kamado[1]

1. Nagaoka University of Technology, Mechanical Engineering, Japan

Excellent room-temperature formability is critical for the practical application of rolled Mg alloy sheets in automotive industry. However, rolled Mg alloy sheets have limited formability at room temperature. This is because, during rolling processing, the Mg alloys easily form strong basal texture where their (0001) planes strongly align parallel to the sheet planes. Therefore, weakening of the basal texture is necessary. The texture feature of rolled Mg alloy sheets is strongly influenced by the alloy composition and thermomechanical conditions. In this work, we have investigated the effect of Zn and Ca additions on grain structure, texture, and mechanical properties of a Mg-3Al-0.4Mn (mass%) alloy sheet. The Mg-3Al-0.4Mn alloy sheet forms strong basal texture and thus shows poor Index Erichsen value of ~3mm. Sole Zn addition does not result in the improvement of room-temperature formability; however, Ca addition has modified strong basal texture, and the Ca-containing sheets exhibit good Index Erichsen value of ~7mm. Also, the Ca addition contributes to the formation of fine disk-shaped precipitates after an aging, leading to moderate 0.2 % proof stress of 150 MPa.

11:40 AM

(ICACC-S11-017-2020) Microstructure and Characteristics of Mo-Cu-X-N Coatings Deposited by Alloying Coatings

S. Kim[1]; H. Yoon[1]; K. Moon*[1]

1. KITECH, Republic of Korea

Mo-N based coatings have been studied for enhancing physical characteristics of thin films. In the case of Mo-X-N coatings, the microstructure and physical properties can be affected by the content of the third element. In our previous work, Mo-Cu-N coatings were successfully fabricated with varying the Cu content from 4.5 at% to 26 at% by the co-sputtering method. Thus, the microstructure and physical properties of the coatings were analyzed by EDS, SEM, XRD, AFM, nano indentation and scratch test techniques. From observed results, Mo_xN bonds were made in a nitrogen atmosphere, and Cu elements were present at grain boundaries. In addition, coatings with a Cu content below 11 at% had a dominant Mo_xN peak and it was difficult to identify the Cu peak in the XRD results. Related to this, the mechanical properties were found to be relatively good at certain Cu contents (4.5 at%, 7.5 at%, 11 at%). So, in this study, it had been tried to make alloying targets with the similar compositions prepared by two elemental target process. The properties between the films prepared by alloying targets and two elemental targets were compared.

Sustainable Energy Concepts and Applications I

Room: Tomoka B

Session Chairs: Heechae Choi, University of Cologne; Hyuksu Han, Hongik University

1:30 PM

(ICACC-S11-018-2020) Recycling of Used Li-ion Batteries by New Lithium Separation Membrane using Ceramics Ionic Conductor (Invited)

T. Hoshino*[1]

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

The world is increasingly turning to the use of Li-ion batteries in electric vehicles; therefore, there is a growing need for lithium (Li). I propose a method for recovering Li from used Li-ion batteries by using innovative electrodialysis with a Li ionic conductor functioning as a Li separation membrane (LISM). This innovative method involves the use of an LISM whereby only Li ions in a solution of used Li-ion batteries permeate from the positive electrode side to the negative electrode side during electrodialysis; the other ions, including Co, Al, and F, do not permeate the membrane. $Li_{0.29}La_{0.57}TiO_3$ was selected as the LISM. The positive side of the dialysis cell was filled with used Li-ion battery solution, and then the negative side was filled with distilled water. In this study, the platinum (Pt) electrodes are bonded to the right and left faces (the two main faces) of the LISM, respectively. The applied dialysis voltage was 5 V, and the electrode area was 16 cm^2 and 4 cm^2 as new LISM. The Li recovery ratio increased with electrodialysis time. Furthermore, Li permeation speed was not depended on the electrode area, and I succeeded in the development of new LISM with small area electrode. Thus, this electrodialysis method is suitable for the Li recycling of used Li-ion batteries.

2:00 PM

(ICACC-S11-019-2020) Laser-induced structural modification of $Na_2FeP_2O_7$ glass-ceramics for all-solid-state sodium ion battery

M. Hiratsuka*[1]; T. Honma[2]; T. Komatsu[3]

1. Nagaoka University of Technology, Materials Science and Technology, Japan
2. Nagaoka Univeristy of Technology, Department of Materials Science and Technology, Japan
3. Nagaoka University of Technology, Japan

In recent years, the rapid use of lithium ion secondary batteries (LIBs) has been expanded. The problems concern material cost and safety performance of LIBs have become serious. Therefore, it is necessary to develop next-generation secondary batteries such as all-solid-state batteries without rare metals. We reported that $Na_2FeP_2O_7$ (NFP) exhibits good active material performance with 88 $mAhg^{-1}$ (90% of theoretical capacity ratio) for sodium ion batteries (SIBs) so far. Furthermore, Yamauchi et al. successfully prepared all-solid-state SIBs composed of $Na_2FeP_2O_7$ glass-ceramic cathode and β″-alumina solid electrolyte by conventional heat-treatment. However, in order to reduce the resistance at the cathode-solid electrolyte interface, it is required to form a stronger bonding interface between the cathode and electrolyte. In order to develop oxide base all-solid-state SIB, laser-induced structural modification technique is interesting to apply NFP glass ceramics because of Fe^{2+} in NFP can absorb laser light and effectively and convert light energy to thermal energy. The aim of the study, we applied laser-induced structural modification to NFP glass-ceramics and demonstrated to make NFP/β″-alumina solid electrolyte interface by continuous wave laser scanning in shortly.

2:20 PM

(ICACC-S11-020-2020) Investigation of pyroelectric power generation with sintered ceramics (Invited)

T. Nakayama*[1]

1. Nagaoka University of Technology, Japan

In power regeneration from a heat source with temporal temperature change (dT / dt), the research is being carried out focusing on pyroelectric effect of ferroelectric. The new thermal / electric combined cycle that applies external charge according to the temporal temperature change is expected to further increase the amount of power generation compared with the conventional cycle. However, there are few experimental examples. In this study, La-doped Pb(Zr, Sn, Ti)O3 (PLZST) ceramics was applied as a pyroelectric material, and power generation characteristics and electric characteristics were measured. PLZST becomes paraelectric phase (PE) after passing through ferroelectric rhombohedral (FER), antiferroelectric tetragonal (AFET) with increasing temperature. In addition, the phase transition from FE to AFE is promising as a pyroelectric sensor material. The phase transition threshold and electric characteristics were adjusted by controlling the Sn: Ti ratio of PLZST. In addition, the power generation amount at that time was compared. As a result, it was found that electric power can be regenerated over a wider temperature range by controlling Sn: Ti ratio.

3:10 PM

(ICACC-S11-021-2020) Materials and Devices for Direct Thermal-to-Electrical Energy Conversion (Invited)

M. Takeda*[1]

1. Nagaoka University of Technology, Department of Mechanical Engineering, Japan

Thermoelectric (TE) effects allow the direct conversion of heat into electrical energy, which can be utilized for waste heat recovery. Materials that possess high Seebeck coefficient, high electrical conductivity and low thermal conductivity are preferable for this purpose. Boron-rich solids are promising materials for thermoelectric conversion. Particularly, metal-doped beta-rhombohedral boron and boron carbide have been studied intensively as thermoelectric material for high temperature application. They are semiconductor and contain B_{12} icosahedral clusters as building unit in their crystal structure. On the other hand, we demonstrated that divalent hexaborides, which contain B_6 octahedral clusters, are also promising candidate for a new class of TE material. Investigation of boron-rich metal borides that contain B_{12} or B_6 clusters is necessary to improve the thermoelectric performance or to discover new candidates. In the present study, B_{12} or B_6 containing metal borides were synthesized and their thermoelectric properties were evaluated. Review of the thermoelectric properties of boron-rich solids and experimental results on the metal borides will be presented. Additionally, I will also present our TE power generator: "Flexible TE device".

3:40 PM

(ICACC-S11-022-2020) Development of non-contact direct electrocaloric measurement method using high speed infrared camera (Invited)

M. Baba*[1]; M. Ejima[1]; N. Ishibashi[1]; S. Fukuda[1]; M. Takeda[1]

1. Nagaoka University of Technology, Department of Mechanical Engineering, Japan

Electrocaloric effect (ECE) is a phenomenon in which a ferroelectric material shows temperature change under an applied electric field. A solid state cooling device that combine ECE and thermal switches is attracting attention, because they can be utilized for small cooling system. Direct and indirect ECE measurement methods are evaluate method of ECE. The direct method measures temperature change (≈ 1 K) directly, and the indirect method calculates temperature change of ECE based on the Maxwell relation. In the direct method, high-sensitive measurements are necessary because of small temperature change of the ECE. In the indirect method, measured polarization-electric field (D-E) curves are necessary to calculate temperature change. In other words, the accuracy of indirect method depends on the accuracy of the measured D-E curves. Therefore, comparison of measurement results obtained by these two methods is necessary. In this study, we developed high-precision ECE measurement method. In the direct method, the non-contact direct ECE measurement (NCDM) method that realize low electrical and temperature noises was developed. Temperature change of ECE is measured using $BaTiO_3$ ceramics by NCDM method. In the indirect method, high-precision measurement of D-E curves using unipolar and bipolar electric fields is carried out, and temperature change of ECE is calculated.

S12: On the Design of Nano-Laminated Ternary Transition Metal Carbides/Nitrides (MAX Phases) and Borides (MAB Phases), and their 2D Counterparts (MXENES, MBENES)

Oxidation Behavior of MAX Phases

Room: Coquina Salon F
Session Chairs: Deniz Cakir, University of North Dakota; Konstantina Lambrinou, SCK-CEN

8:10 AM

(ICACC-S12-010-2020) Effect of Impurities on the Morphology and Spallation of the Oxide Formed by Oxidation of Cr_2AlC (Invited)

Y. Chen[1]; Z. Zhan[1]; D. Holta[1]; J. Smialek[2]; T. Ouisse[3]; M. Radovic*[1]

1. Texas A&M University, Materials Science & Engineering, USA
2. NASA Glenn Research Center, USA
3. Grenoble INP, France

Cr_2AlC is one of the MAX phases with the potential of forming protective alumina oxide layer when exposed to high temperature in oxidizing environment. However, formation of alumina layer on

the surface, results in Al depletion in Cr_2AlC and formation of Cr_7C_3 sub-layer between the Al_2O_3 scale and the Cr_2AlC substrate. In this study, a bulk polycrystalline Cr_2AlC samples were synthesized with different ratios of Cr_2C_3, Cr and Al starting materials to produce samples with different amount of chromium carbide impurities, using Cr powders of different purity. Those polycrystalline samples, as well as Cr_2AlC singlecrystals were exposed to oxidation in 1200 ºC for different times. The results of this study show that formation of Cr_7C_3 sub-layer can be avoided in Cr_2AlC samples free of chromium-carbide impurities or in samples with excess Al. In addition, we have shown that small amounts of impurities in starting Cr powders (such as Fe) as well as absence of Cr_7C_3 sub-layer leads to the wrinkling of alumina scale and its premature spallation. The results of this study suggest that careful control of the composition of Cr_2AlC can result in formation of protective and stable alumina oxide scale, as that is the case during oxidation of Ti_2AlC.

8:40 AM

(ICACC-S12-011-2020) Oxidation behavior of V_2AlC coatings in air

C. Azina*[2]; S. Mráz[1]; M. M. Yildizhan[2]; J. Rosén[2]; J. Schneider[1]; P. Eklund[2]

1. RWTH Aachen University, Materials Chemistry, Germany
2. Linkoping University, Dept. of Physics, Chemistry, and Biology, Sweden

The oxidation resistance of several MAX phases has been discussed the past few years and the formation of an, oftentimes, protective Al_2O_3 scale was demonstrated in the case of Al-containing MAX phases. While the oxidation behavior of Cr_2AlC and Ti_2AlC has been widely investigated, little information can be found on V_2AlC as oxidation above 700 °C has been shown to be destructive. Here, we investigate the oxidation resistance of V_2AlC coatings at various temperatures up to 900 °C in air. Near phase-pure films were obtained by sputtering of a V_2AlC compound target on MgO (111) substrates. Oxidation tests have been carried out at several temperatures ranging between 400 and 900 °C. The films were systematically analyzed to track the microstructural and compositional variations of the MAX phase with respect to temperature. The films were shown to form a protective coating up to 800 °C, at which after 5 min of annealing, the MAX phase completely decomposed into a multi-oxide film.

9:00 AM

(ICACC-S12-012-2020) Thermal stability enhancement of Cr_2AlC coatings on Zr by utilizing a double layer diffusion barrier

S. Mráz*[1]; M. Tyra[1]; M. to Baben[2]; M. Hans[1]; X. Chen[1]; F. Herrig[1]; K. Lambrinou[3]; J. Schneider[1]

1. Materials Chemistry, RWTH Aachen University, Germany
2. GTT Technologies, Germany
3. SCK-CEN, NMS, Belgium

Decomposition of Cr_2AlC deposited onto a Zr substrate and vacuum-annealed is observed at 800°C as Al diffuses from the MAX phase into the Zr substrate. A double layer of ZrN and AlN has been predicted by CALPHAD calculations to act as diffusion barrier between the Zr substrate and Cr_2AlC. Experimental thermal stability investigations corroborate this prediction by confirming that the proposed double layer diffusion barrier coatings suppresses the decomposition of Cr_2AlC at temperatures of up to 1000°C.

9:20 AM

(ICACC-S12-013-2020) Oxidation resistance of $(Ti_x,Nb_{1-x})_2AlC$ MAX phases

E. Epifano*[1]; G. A. Hug[2]; A. Jancowiak[3]

1. ONERA, DMAS/LEM, France
2. ONERA, LEM, France
3. ONERA, France

In order to increase the temperature of aeronautical engines and hence their thermal efficiency, new high-temperature resistant materials are currently under study. A promising option seems the γ-TiAl intermetallic with a Ti_2AlC protective layer. The Ti_2AlC compound is one of the so-named MAX phases, a class of thermodynamically stable carbides or nitrides. MAX phases are of great interest in various domains because of their unique properties, between ceramics (high melting point, corrosion resistance…) and metals (excellent conductivities, good machinability…). Among the MAXes, Ti_2AlC is the most investigated for the aerospace applications because of its good properties and low weight. Recent experiments have shown that doping the Ti_2AlC phase with Niobium increases the high temperature oxidation resistance, by enhancing the formation of a protective alumina layer. In this work, several $(Ti_x,Nb_{1-x})_2AlC$ compounds, with x ranging from 0.05 to 0.20, are investigated in order to determine the optimum Nb concentration to improve the oxidation resistance. Oxidation tests will be performed in air, at 1273 K, and the evolution of the sample microstructure will be characterized by Scanning Electron Microscopy. Moreover, Density Functional Theory calculations will be performed in order to understanding the atomic-scale mechanisms induced by the Nb presence that enhance the oxidation resistance.

Theoretical and Multifunctional Application of MAX Phases

Room: Coquina Salon F

Session Chairs: Jesus Gonzalez-Julian, Forschungszentrum Juelich; Mihaela Florea, National Institute of Materials Physics

10:00 AM

(ICACC-S12-014-2020) Structural characterization of the phase transition of Cr_2AlC under ion irradiation

T. Cabioch*[1]; F. Brenet[1]; J. Nicolaï[1]; M. Beaufort[1]

1. Institut PPRIME, France

The phase transition from the MAX Phase structure to the so-called gamma phase structure (Solid solution (2Cr,Al)C with the NiAS structure) is here studied for various Cr_2AlC samples (bulk samples and thin films). Bulk samples were irradiated with 4 MeV gold ions at room temperature and thin films were irradiated with 50 keV He ions at room temperature and 350°C. The phase transition was then characterized by using transmission electron microscopy and electron diffraction but also X-Ray diffraction, electron energy loss spectroscopy and resistivity measurements. On the basis of the results so far obtained, we discuss the formation of antisites on the Cr and Al positions and the progressive rearrangement of C atoms.

10:20 AM

(ICACC-S12-015-2020) On the feasibility of MAX phase coatings on Zircaloy cladding for Enhanced Accident Tolerance Fuels in LWRs (Invited)

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

1. Insitute of Metal Research, Chinese Academy of Sciences, Shenyang National Laboratory for Materials Science, China

The safe, reliable, and economic operation has always been a top priority for nuclear power. For the current fleet of commercial LWRs, the development of nuclear fuel and claddings with enhanced accident tolerance has been launched worldwide, so called Enhanced Accident Tolerant Fuels (E-ATF). Among all the strategies, modification of Zircaloy cladding surface with advanced ceramic coatings appears to be the one promising short-term concept. In the current presentation, the early studies on nano-laminated ternary carbides/nitrides as protective coatings were revealed. Then, the gradient ceramic coatings with low thermal neutron capture cross section and optimal steam oxidation resistance were synthesized by PVD method with temperature friendly to Zircaloy cladding. The mechanical and chemical compatibility between integrated coating and substrate in the as-deposited state as well as under LOss of Coolant Accident scenario were investigated. Finally, the HT steam oxidation and hydrothermal corrosion tests were performed to evaluate the feasibility of integrated ceramic coatings in accident-tolerant fuel/clad system.

10:50 AM

(ICACC-S12-016-2020) Compatibility of the Zr_2AlC MAX phase with liquid lead-bismuth eutectic

K. Lambrinou*[1]; B. Tunca Altintas[1]; T. Lapauw[3]; C. Callaert[2]; J. Hadermann[2]; R. Delville[1]; E. Caspi[4]; M. Dahlqvist[5]; J. Rosen[5]; M. Amalraj[6]; K. Pradeep[7]; J. Schneider[6]; J. Vleugels[3]

1. SCK-CEN, Nuclear Materials Science Institute, Belgium
2. University of Antwerp, Department of Physics, Belgium
3. KU Leuven, Department of Materials Engineering, Belgium
4. Nuclear Research Centre-Negev, Physics Depratment, Israel
5. Linkoping University, Department of Physics, Chemistry, and Biology, Sweden
6. RWTH Aachen University, Materials Chemistry, Germany
7. Indian Institute of Technology, Department of Metallurgical and Materials Engineering, India

Nano-laminated ternary carbides (MAX phases) are considered possible coating materials for stainless steel fuel clads intended for use in Gen-IV lead-fast reactors (LFRs). MAX phase coatings aim at preventing the interaction of the substrate stainless steel clad with the heavy liquid metal, which might lead to steel degradation due to dissolution corrosion. This work reports on the compatibility of the Zr_2AlC MAX phase with oxygen-poor ($C_O < 10^{-8}$ mass%) static liquid lead-bismuth eutectic (LBE) after 1000 h at 500°C. The low neutron cross-section of Zr makes Zr_2AlC an appealing fuel clad coating material candidate. The observed LBE/Zr_2AlC interaction was local and resulted in the in-situ formation of a $Zr_2(Al,Bi,Pb)C$ MAX phase solid solution in the LBE-attacked areas. This work proposes a mechanism for the in-situ formation of the $Zr_2(Al,Bi,Pb)C$ solid solution, the existence of which was confirmed by neutron powder diffraction in a bulk ceramic produced by reactive hot pressing of ZrH_2, Al, C and eutectic Pb-Bi starting powders. Out-of-plane ordering resulting in the alternation of Al-rich and Pb/Bi-rich layers in the MAX phase structure was revealed by TEM, and was also supported by density functional theory calculations. Grains of the parasitic ZrC phase in the Zr_2AlC ceramic were not susceptible to LBE attack, except for twin boundaries in these grains that were preferentially decorated by Pb/Bi atoms.

11:10 AM

(ICACC-S12-017-2020) Ab initio study of vacancy stability and migration in disordered MAX phase alloys

P. Singh*[1]; D. Sauceda[1]; R. Arroyave[1]

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

MAX phase has attracted increased attention due to unique combination of ceramic and metallic properties. We perform systematic density functional study to explore the alloying effect on structural-stability, energy-stability, electronic-structure and vacancy migration on disorder MAX phase with point defects. The vacancy (V_M,V_A,V_X) and antisite (M-A; M-X) defects are considered for M (=Cr, Nb, Ti) and A (=Sn, Al, Pb, Bi) site disorder in $(ZrM)_2(AA')C$, where M=Cr, Nb, Ti and AA'=Al, AlSn, PbBi. Our calculations suggest that the chemical disorder improves the vacancy formation energies, whereas the vacancy migration energies are significantly lowered compared the ordered MAX phase. We believe that our study will provide a fundamental understanding and ways to manipulate the key properties of disordered MAX phase that will lead to desirable properties and the discovery of new MAX phases.

11:30 AM

(ICACC-S12-018-2020) Exploring the Effect of Cleavage-Stress and Shear-Stress on Structural, Electronic and Mechanical Properties of MAX Phase

D. Sauceda*[1]; P. Singh[1]; R. Arroyave[1]

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

The MAX phase alloy undergoes cleavage and shear upon application of stress. The intrinsic resistance to cleavage and shear deformation is important and provides valuable insights into the brittle/ductile nature of materials. To understand relative propensities of MAX phase alloys to undergo cleavage or shear and its effect on electronic, structural and mechanical properties, we perform a detailed investigation using first-principles density-functional theory on M_2AC, where M= Sc, Ti, V, Cr, Mn, Fe, Co, Cu, Mo, Nb, Ni, Y, Zr, Ta, Hf, Ta and A=Al, Si. We focus on simple-alias cleave/shear deformation of M-A layer (as the weaker metallic bonds between M-A make the deformation easier) and provide electronic structure origin of mechanical deformation behavior. We believe that the understanding of the cleavage and shear behavior within the 211 chemistry (M_2AX) will provide valuable information about the parameterization of micro-mechanical models for deformation in these materials.

Current Progress in Mxenes I

Room: Coquina Salon F

Session Chairs: Martin Magnuson, Linkoping University; De-en Jiang, University of California, Riverside

1:20 PM

(ICACC-S12-019-2020) Multilayered MXenes and Clays: The Interlayer Space, Cations, Polar and Nonpolar Solvents

M. Barsoum*[1]; M. Carey[1]; V. Natu[1]; L. Verger[1]

1. Drexel University, Materials Science and Engineering, USA

The 2D early transition metal carbides known as MXenes - obtained by etching the A-layers from the MAX phases - discovered in 2011 - have generated substantial interest in the scientific community because of their potential in an ever-expanding host of applications. In many of these applications, what is occurring in the interlayer space of multilayers is critical, whether because it leads to delamination, or their dispersion in various solvents. Unlike hydrophobic graphene, MXenes are hydrophilic and behave as 2D metals or "conductive clays" a hitherto unknown combination. In this talk I will focus on the "conductive clay" aspects of MXenes. There is a large body of work on clays that was carried out decades ago. Over the years we have made exploited this work to understand what is happening in multilayered MXenes Using primarily XRD diffraction, the relationship between etchant used and the swelling of the interlayer space in $Ti_3C_2T_x$ multilayers will be elucidated. Further how to disperse and stabilize $Ti_3C_2T_x$ in polar and non-polar solvents is discussed. In short, the relationship between intercalated cations and interlayer space and the similarities and differences between MXenes with clays will be touched upon.

2:00 PM

(ICACC-S12-020-2020) Oxidation and stabilization of 2D MXene nanosheets (Invited)

M. Green*[1]; M. Radovic[2]; J. Lutkenhaus[1]

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

MXenes, such as $Ti_3C_2T_x$, are fascinating 2D nanomaterials with an attractive combination of functional properties suitable for applications such as batteries, supercapacitors, and strain sensors. However, practical uses of MXenes, such as $Ti_3C_2T_x$, remains challenging as these nanosheets are known to oxidize and degrade quickly from reacting with water and dissolved oxygen. Here, we examine oxidation of MXene nanosheets in various media (air, liquid, and solid) via multiple types of measurements to assess their shelf stability. The oxidation rate of MXene nanosheets were observed fastest in liquid media and slowest in solid media and can be accelerated by exposure to UV light. We also demonstrate an effective method to retard the oxidation of colloidal $Ti_3C_2T_x$ MXene nanosheets by introducing antioxidants such as sodium L-ascorbate. The success of the method is evident in the conductivity and colloidal stability of $Ti_3C_2T_x$. Even in the presence of water and oxygen, the electrical conductivity of $Ti_3C_2T_x$ nanosheets treated with sodium L-ascorbate was orders of magnitude higher as

compared to untreated ones after 21 days. Our findings have the potential to be generalized to protect other types of MXenes as well and solve the most pressing challenge in the field of MXene engineering.

2:30 PM

(ICACC-S12-021-2020) New pathways in the synthesis of MXene plates, crumpled sheets, spheres, and scrolls

S. Kellici*[1]

1. London South Bank University, School of Engineering, United Kingdom

MXene, a low dimensional material is composed of early transition metal carbides or nitrides. MXene is derived from 3D MAX parent phase laminar materials (ceramics), where $M_{n+1}AX_n$, M = early transition metal, A = group 3A or 4A, X = C or N, n = 1, 2, or 3. A is etched to give MXenes: M_2C, M_3C_2, and M_4C_3. In this work, we employed calixarenes to template for the first time morphological changes into plates, crumpled sheets, spheres, and scrolls. Calixarenes are versatile macrocycles with inherent architecture. The as–synthesized materials have potential applications in energy storage, optoelectronics, heavy–metal adsorption, catalysis, tribology, bio–sensing.

2:50 PM

(ICACC-S12-022-2020) Electrically conductive MXene coated glass fibers for damage-sensing epoxy composite applications

C. B. Hatter*[1]; Y. Gogotsi[1]

1. Drexel University, Materials Science and Engineering, USA

Polymer composites offer a wide variety of uses in fields such as aersospace, additive manufacturing, and the automotive industry. Fiber-reinforced polymer composites, specifically, are an important class of materials due to their mechanical strength, lightweight, and fatigue performance. Two-dimensional (2D) transition metal carbides (MXenes) have rich chemistries offer metallic conductivity and hydrophilicity coupled with good mechanical properties. MXenes has been shown to have electrically conductivities upwards of 10,000 S/cm and mechanical stability with Young's modulus of 330 GPa, one of the highest among solution processed 2D materials. Most MXene-polymer composite studies to-date have investigated their use as electrodes in energy storage systems as well as electromagnetic interference shielding, however no studies have explored incorporating MXenes into fiber-reinforced composite systems for damage-tracking capabilities. Here we present a facile coating method for attaching $Ti_3C_2T_x$ MXene flakes to electrically insulating glass fibers for potential use in epoxy composites. In-situ electrical resistance monitoring was performed during tensile testing of MXene-coated fibers to assess damage-sensing capabilities. Furthermore, this new multifunctional system could provide tracking of mechanical failures within composite systems.

Design and Characterization of MAB Phases

Room: Coquina Salon F

Session Chair: Micah Green, Texas A&M University

3:30 PM

(ICACC-S12-023-2020) Perspectives on processing $AlFe_2B_2$ and related MAB phases for magnetocaloric applications (Invited)

R. Barua*[1]; B. T. Lejeune[2]; S. Vallone[4]; R. T. Ott[3]; K. G. Sandeman[4]; B. Frandsen[5]; M. Kramer[3]; L. Lewis[2]

1. Virginia Commonwealth University, Department of Mechanical & Nuclear Engineering, USA
2. Northeastern University, Department of Chemical Engineering, USA
3. Ames Laboratory (USDOE), Materials Sciences and Engineering, USA
4. Brooklyn College, CUNY, Department of Physics, USA
5. Brigham Young University, Department of Physics, USA

Material processing schemes play a critical role in guiding the development of emerging magnetocaloric materials for energy-related applications, mainly magnetic refrigeration and thermomagetic energy conversion. Within this context, the ternary MAB phase $AlFe_2B_2$ is attractive due to its low cost, favorable heat transfer properties, tunable magnetic transition temperature (T_c) and promising room temperature magnetocaloric effect (MCE). In this study, the magnetocaloric potential of single-phase $AlFe_2B_2$ samples synthesized via conventional metallurgical routes (casting) and additive manufacturing technology (laser engineered net shaping (LENS)) was evaluated and compared. Suction-casting and subsequent heat treatment at high temperatures allows fabrication of $Al_{1.2}M_xFe_2B_2$ (M=Ga/Ge, x≤0.1) samples, where-in introduction of ~2 at % of Ga/Ge results in 30 K increase in T_c and two-fold improvement in MCE. The enhanced functional response of the $Al_{1.2}(Ga,Ge)_xFe_2B_2$ samples is attributed to chemical bonding and electronic effects arising due to local disorder caused by antisite occupancy of Fe on Al sites within the $AlFe_2B_2$ lattice. These results provide fundamental insights regarding the phase stability of the ternary Al-Fe-B system, and guide 3D-printing of functionally-graded $AlFe_2B_2$ regenerators with porous architecture for modular insertion into magnetocaloric heat-pump device prototypes.

4:10 PM

(ICACC-S12-024-2020) Synthesis and Characterization of MAB Phases by Novel Manufacturing Methods

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

1. University of North Dakota, Mechanical Engineering, USA

Traditionally, non-oxide ceramics are manufactured by sintering pressed pellets in inert environment to prevent oxidation. For manufacturing perspective, it is quite expensive to maintain inert environment. For large scale deployment, it is imperative to design and develop alternative manufacturing methods. An alternative is to manufacture non-oxide ceramics by encapsulating it in molten salt to present oxidation. Recently non-oxide ceramics like B-containing MAB Phases have emerged as potential material for different structural application. In this presentation, we will report the synthesis and characterization of MAB phases by using salt method. As a a part of this study, we will compare different manufacturing methods for synthesizing MAB phases.

S13: Development and Applications of Advanced Ceramics and Composites for Nuclear Fission and Fusion Energy Systems

Novel Ceramics and Composites for Nuclear Systems II

Room: Coquina Salon H

Session Chair: Caen Ang, University of Tennessee

8:30 AM

(ICACC-S13-010-2020) 3D-printed silicon carbide for nuclear energy applications

G. Vasudevamurthy[1]; K. Terrani*[1]

1. Oak Ridge National Lab, USA

An additive manufacturing process was developed at Oak Ridge National Laboratory in the past two years that allows for 3D printing of SiC. The material is polycrystalline and free of impurities and secondary phases. This microstructure is ideal when considering applications that involve displacement damage in irradiation environments. This talk describes the properties and testing activities for this material system as it pertains to nuclear fission and fusion energy applications.

8:50 AM

(ICACC-S13-011-2020) Thermophysical properties of sintered yttrium dihydride

A. P. Shivprasad*[1]; V. K. Mehta[2]; J. T. White[1]; M. W. Cooper[1]; T. A. Saleh[1]; J. R. Wermer[3]; E. P. Luther[3]; H. R. Trellue[2]; D. Rao[4]

1. Los Alamos National Lab, Materials Science and Technology Division, USA
2. Los Alamos National Lab, Nuclear and Nonproliferation, USA
3. Los Alamos National Lab, Sigma Division, USA
4. Los Alamos National Lab, Civilian Nuclear Programs, USA

One current challenge to the nuclear industry is the ability to produce nuclear energy in remote locations with microgrids, at which localized power generation might be necessary. Owing to their smaller size, cost, and power output, microreactors are being designed to meet these needs. Proposed microreactor designs may comprise metal hydride moderating material, to help reduce size and fuel mass requirements in the core by increasing the number of low energy neutrons and optimizing fission in the core. Yttrium dihydride (YH_2) contains a large amount of hydrogen for such moderation, has high thermal stability at temperatures even exceeding 1500 K, and is thus a promising candidate for this application. Despite these advantages, it is difficult to produce YH_2 in geometries required for reactor design concepts. In this study, YH_2 pellets were fabricated using powder metallurgical methods in an effort to produce material to near net shape. Pellets were analyzed for thermal diffusivity, coefficient of thermal expansion, and heat capacity to determine thermal conductivity. Results presented in this work will relate the thermophysical properties of the sintered pellets with those of hydrided monoliths in literature. Heat capacity will also be discussed in terms of Debye and Einstein temperatures.

9:10 AM

(ICACC-S13-012-2020) Fabrication and Characterization of Massive Crack-free Single-Phase Yttrium Hydride for High Temperature Moderator Application

X. Hu*[1]; K. Terrani[1]

1. Oak Ridge National Lab, USA

The use of metal hydrides, especially the zirconium hydride (ZrHx), as high-performance moderators in advanced reactors has long-term precedent. However, the application of ZrHx in nuclear system requires careful management of the moderator temperature to avoid hydrogen desorption at elevated temperatures. Yttrium hydride (YHx) is more attractive for high temperaturemoderator applications, attributed to its much lower equilibrium hydrogen partial pressure at elevated temperatures. A fully programmable hydriding system with continuous hydrogen partial pressure and flow control to facilitate processing of massive YH x has been developed at ORNL. In this presentation, the working principle of the hydriding system will be introduced. Characterization of the produced YHx includes X-ray powder diffraction to identify the present phases, LECO H and O analysis and vacuum hot gas extraction to quantify hydrogen, and X-ray Computed Tomography to visualize the possible cracks. The results indicate that crack-free single phase YHx with >10 cm^3 has been successfully produced. We will also report the physical (i.e., lattice constant, density), thermal (i.e., thermal diffusivity, heat capacity, and CTE), and mechanical (i.e., hardness, elastic modules, equibiaxial flexural strength) properties of the resulting YHx as a function of hydrogen content.

9:30 AM

(ICACC-S13-013-2020) A new neutron shielding material W-WB

M. Athanasakis[1]; C. McFadzean[1]; S. A. Humphry-Baker*[1]

1. Imperial College London, Materials, United Kingdom

Compact spherical tokamaks offer a rapid development pathway for fusion energy. Their success requires more space-efficient shielding materials. A critical component is the superconducting magnet coils within the central column, which must be protected from neutrons and gamma rays. Computational studies show that tungsten boride (WB) has expectational neutron-attenuation efficiency, however suitable engineering forms for it are yet to be engineered. We have begun a research programme on W-WB composites, which may combine the attenuation characteristics of WB, with the known thermophysical strengths of W. In this first report, we present the material's high temperature mechanical properties and oxidation resistance. Mechanical properties are tested in flexion and compression up to 2000°C. The material shows a ductile-brittle transition temperature (DBTT) of ~1000°C, and a peak flexural strength at ~1200°C of ~1GPa, which is significantly higher than rolled W. Next, oxidation resistance results are shown using thermogravimetry up to 1100°C. These results are encouraging: the presence of WB particles causes a transition from linear to parabolic kinetics, when compared to pure W which is known to have limited oxidation resistance. The protective nature of the oxide scale is due to the formation of a passivating boria scale. As a result of these promising results, further work is envisaged. Particularly needed are irradiation damage studies.

9:50 AM

(ICACC-S13-014-2020) Design and Strategy for Next Generation Silicon Carbide Composites for Nuclear Energy

Y. Katoh*[1]; T. Koyanagi[1]; Y. Yang[1]; B. Jolly[1]; C. Ang[2]; T. Nozawa[3]; L. Snead[4]

1. Oak Ridge National Laboratory, USA
2. University of Tennessee, Nuclear Engineering, USA
3. National Institutes for Quantum and Radiological Science and Technology, Japan
4. Stony Brook University, USA

Silicon carbide-based ceramic composites technology is making steady advance toward deployment in nuclear systems with the initial design rules for composite core components for high temperature reactors published as a part of the 2019 ASME Code Sc. III. As performance data establish, the limitations on the current generation of nuclear grade SiC composites are recognized. These limitations are mainly for high dose neutron irradiation tolerance, corrosion in oxidizing environments, and the ability to retain hermeticity in the operating environment. To overcome these limitations, improved quality SiC fibers, alternative fiber-matrix interphases, modified or alternative matrices, incorporation of hermetic layers, and environmental barrier coatings are being considered and developed. This paper discusses various options for improving the performances of the next generation of SiC composites for nuclear energy applications, with the main emphasis on the design and development of alternative interphase layers for advanced composites. Research sponsored by the U.S. Department of Energy, Office of Fusion Energy Sciences and Office of Nuclear Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

Mechanical Properties: Test Methods, Codes and Standards, and Design Methodology

Room: Coquina Salon H
Session Chair: Yutai Katoh, Oak Ridge National Laboratory

10:30 AM

(ICACC-S13-015-2020) Inter-laboratory round robin study on hoop tensile properties of SiC/SiC composite tubes

T. Nozawa*[1]; H. Sato[2]; K. Furumoto[3]; R. Ishibashi[4]; S. Yamashita[5]; T. Kawanishi[5]; T. Fukahori[5]

1. National Institutes for Quantum and Radiological Science and Technology, Japan
2. Toshiba Energy Systems & Solutions Corporation, Japan
3. Mitsubishi Nuclear Fuel Co., Ltd., Japan
4. Hitachi GE Nuclear Energy, Ltd., Japan
5. Japan Atomic Energy Agency, Japan

SiC/SiC composites are one of promising candidates for advanced fuel and core components of light water reactor with enhanced accident tolerance due to superior thermo-mechanical properties by

neutron irradiation and less hydrogen generation with reaction of water vapor at high-temperatures during severe accident. To judge feasibility of the SiC/SiC composite cladding as a realistic option for the future LWR components, evaluation of baseline mechanical properties, e.g., hoop tensile strength as one of important parameters for the ATF design, is essential. The key test methodology for CMC tube hoop tensile properties at ambient temperature was standardized as ASTM C1819 so far. However critical information about repeatability and reproducibility of this developmental standard is somehow lacking. In this study, the precision and bias of the test methodology were therefore evaluated by the interlaboratory round robin study. Two different types of composite tubes, CVI and NITE SiC/SiC tubes, were tested by six laboratories according to the ASTM C1819 with their in-house testing test equipment, using the supplied common fixtures and elastomeric inserts. Statistical nature of SiC/SiC composites tubes was then evaluated by Weibull statistics as well as ASTM E691, standard practice for conducting an interlaboratory study to determine the precision of a test method.

10:50 AM

(ICACC-S13-016-2020) SiC-SiC CMCs and Graphite for Nuclear Applications: Newly Published Parts of the ASME BPV Code, Section III, Division 5

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 Laboratory, USA

High-temperature reactors (HTRs) planned by US DOE use SiC-SiC CMCs and graphite to enhance fuel performance and improve accident tolerance because these materials are tolerant to the relevant irradiation and chemical environments. As nonconventional materials, SiC-SiC CMCs and graphite are of special concern because the mission of the US Nuclear Regulatory Commission (NRC) is to license and regulate the nation's civilian nuclear reactors, reactor designs and reactor materials. NRC is legally required to use consensus codes and standards as integral parts of the regulatory process. Therefore, the ASME Boiler and Pressure Vessel (BPV) Code Section III "Rules for Construction of Nuclear Components" (including accepted materials) is included in the NRC regulations. Division 5 on HTRs of Section III has supported a working group on graphite and ceramic composites for many years. For SiC-SiC CMCs to be incorporated into future HTRs, they must be included in ASME BPV Code as acceptable materials. In 2019 new portions of ASME BPV Code on design and qualification components involving graphite and SiC-SiC CMCs as well as appendices regarding the testing, classification, composition, structure, manufacture, and properties of SiC-SiC CMCs for nuclear applications were finalized and published.

11:10 AM

(ICACC-S13-017-2020) Compressive Strength of CMC Tubes Used as Components in Nuclear Applications: ASTM Draft Standard Using Axial Compression Loading

M. G. Jenkins*[1]; J. E. Gallego[1]

1. Bothell Engineering and Science Technologies, USA

US DOE has plans to use advanced materials for the core and the reactor unit components in various advanced reactor concepts. Ceramic matrix composites (CMCs), in particular silicon carbide (SiC) fiber SiC-matrix (SiC-SiC) composites, could greatly expand the design window for various components in terms of operating temperatures, applicable stresses, and service lives, compared to heat-resistant metallic alloys, while significantly improving accident tolerance and safety margins. Potential CMC tubular components include fuel rods, control rod sleeves, and control rod joints. Possible failure modes for these components include axial and hoop tension, axial flexure, axial and diametral compression, and axial shear. A draft ASTM standard test method has been developed and submitted for full-consensus ballot to determine the longitudinal compressive strength of ceramic matrix composite tubes subjected to axial compression. Modeling and empirical tests of composite tubes provided validation of the parameters specified in the test method. The draft standard test method addresses the following experimental issues -- test specimen geometries/preparation, test fixtures, test equipment, interferences, testing modes/procedures, data collection, calculations, reporting requirements, precision/bias.

11:30 AM

(ICACC-S13-018-2020) Development of Novel Flexure Test Methods for Nuclear Grade SiC/SiC Composite Tube

O. Adams[1]; J. Bao[1]; D. McCleeary[1]; X. Huang*[1]

1. University of South Carolina, Mechanical Engineering, USA

Silicon Carbide (SiC) matrix, SiC fiber reinforced ceramic matric composite (SiC/SiC) is a candidate for accident tolerant fuel cladding. The authors have developed flexure test methods to study the mechanical response and damage modes of the SiC composite tube. Further development of our flexure test methods has been carried out to enable flexure test (1) with internal pressure; (2) under temperature gradient; (3) with in-situ leak monitoring. In the flexure test with internal pressure, pressurized hydraulic fluid was sealed inside the tube during flexure test. This results in combined internal pressure and flexure loading. To create temperature gradient, the tubular sample was cooled from inside and heated from outside. Liquid nitrogen was pumped through the ID and hot water was used to heat the OD of the tube, an ID-to-OD temperature difference of over 100 C can be maintained. This technique allowed us to generate a compressive stress on the OD side of the tube and to study its flexure response. To enable flexure test with in situ Helium leak detection, a soft jacket with pressurized helium was placed around the tube during the flexure test, the helium leaked through the tube wall was monitored in situ by mass spectrometer during the flexure test. Implementation details of test methods as well as the typical test results obtained on nuclear grade SiC/SiC composite tube will be presented.

11:50 AM

(ICACC-S13-019-2020) Probabilistic Failure Analysis of SiC/SiC Composite Fuel Cladding under Multi-Axial Loading

J. Le*[1]; C. Hu[1]; J. Labuz[1]; T. Koyanagi[2]

1. University of Minnesota, USA
2. Oak Ridge National Laboratory, USA

Owing to excellent mechanical properties under various thermal, chemical and neutron radiated conditions, SiC/SiC composites have been recognized as a promising material for accident-tolerant fuel cladding in light water reactors. Recent experiments showed that the failure strength of SiC/SiC composites exhibits a considerable degree of variability. Understanding the strength statistics is of paramount importance for reliability-based analysis and design of nuclear structural components. In this study, we develop a mechanistic reliability analysis framework. A multiaxial stress failure envelope at the proportional limit is first determined based on laboratory experiments and existing literature. The failure probability of a material representative volume element is calculated by the second-order reliability method (SORM). Due to the damage localization mechanism, the failure statistics of the entire fuel cladding can be modeled by a finite weakest-link model. The main feature of the model is that it predicts a strong size effect on the failure statistics, which provides a robust means for extrapolation of laboratory test results to full scale design. Based on the recent study on the time-dependent stress distribution of SiC/SiC composites in fuel cladding, we determine the time evolution of the failure risk, which provides valuable information for assessment of SiC/SiC cladding.

Ceramic Fuel Materials, Technologies, and Characterization; TRISO Fuels

Room: Coquina Salon H
Session Chair: Kurt Terrani, Oak Ridge National Lab

1:30 PM

(ICACC-S13-020-2020) Stress Analysis and Failure Behavior of SiC_f/SiC_m Textile Composite tubes

H. T. Nagaraju*[1]; J. Nance[2]; B. Sankar[1]; G. Subhash[1]; R. Haftka[1]

1. University of Florida, Mechanical and Aerospace Engineering, USA
2. University of Florida, Material Science Engineering, USA

Silicon carbide-fiber/silicon carbide-matrix (SiC_f/SiC_m) textile ceramic composite tube is a potential candidate for nuclear fuel cladding material due to its mechanical properties such as high modulus and strength. The cladding material must withstand complex stress states induced during operation of a nuclear reactor. It is imperative to understand its failure behavior and mechanical response under these loads. In our research, we conducted finite element (FE) analysis of SiC_f/SiC_m tubes by considering a representative volume element (RVE) of the structure. Due to its curved geometry and complicated braid architecture, it is difficult to create an FE mesh of a textile composite tube. We have utilized an open source software, TeXGen®, to generate FE mesh of a flat RVE. The nodal coordinates of that flat RVE are then transformed to create a curved RVE. The resulting mesh is imported to ABAQUS™ for stress analysis. Appropriate load and boundary conditions are imposed on the curved RVE under plane strain conditions. The approach has been verified by comparing the FE results of the curved RVE with analytical solutions for homogeneous isotropic and homogenous orthotropic materials. The stresses calculated through the simulations are then used to generate failure envelopes for the composite tube.

1:50 PM

(ICACC-S13-021-2020) Densification of ZrC for Fully Ceramic Microencapsulated fuels

C. Ang*[1]; L. Snead[2]; A. Gordon[1]; S. Judd[3]; K. M. Benensky[4]; Y. Katoh[1]

1. University of Tennessee, Nuclear Engineering, USA
2. Stony Brook University, Materials and Chemical Engineering, USA
3. Blue Origin, LLC, USA
4. Analytical Mechanics Associates, Inc., USA

ZrC is proposed as an improved matrix for Fully Ceramic Microencapsulated (FCM) fuels, due to limits in the current SiC matrix in hot hydrogen environments at 2000-2500K. However, its densification at conditions compatible with abiding TRISO particles is a significant challenge. The typical methods of ZrC consolidation by excessive temperature and high pressure were examined and demonstrated to be unfavorable, and were unable to exceed the intermediate stage of sintering. Instead, a modest approach toward the identification of additives to accelerate grain boundary diffusion was conducted. WC and other carbides with variable oxidation states appear to be suitable dopants to improve diffusion; densities of approaching ~95% were achievable at relatively low temperatures of 1900°C. Microscopy and grain size analysis show improved grain growth, while XRD shows a lattice shift in the ZrC phase, representative of dopants in the ZrC ionic-covalent lattice. The results are promising indicators of additives able to assist in proceeding beyond the intermediate stage of sintering in ZrC.

2:10 PM

(ICACC-S13-022-2020) Oxidation testing and microstructural analysis of AGR matrix material

T. J. Gerczak*[1]; C. Contescu[1]; J. Hunn[1]; Y. Lee[1]; R. Mee[2]

1. Oak Ridge National Laboratory, USA
2. University of Tennessee, USA

Graphite and carbonized resin matrix surround and protect tristructural-isotropic (TRISO) coated particle fuel in the fuel form of high temperature gas-cooled reactors (HTGRs). Accident scenarios in HTGRs include possible moisture ingressat high temperatures. In addition, chronic oxidation occurs due to trace moisture levels in the coolant during normal operating conditions. The response of the matrix to oxidants must be known to understand fuel performance in normal, off-normal, and low-probability accident scenarios. Matrix oxidation experiments were conducted on unfueled matrix blanks fabricated from AGR-5/6/7 base material. The oxidation experiments were conducted in various moisture environments and temperatures to obtain oxidation kinetics (P_{H2O} 3–600 Pa, 800–1200°C) and to explore accident performance (P_{H2O} 10–50 kPa, 1200–1500°C). The oxidation behavior indicated similar performance of matrix with that of nuclear grade graphite. Microstructural analysis was performed on the matrix blanks and compared with the matrix structure in fueled AGR-5/6/7 compacts. The analysis indicated nonrepresentative texture and fissures in matrix blanks, which should be considered when interpreting material properties obtained from unfueled matrix samples. This work was supported by the U.S. Department of Energy, Office of Nuclear Energy in support of the Advanced Gas Reactor Fuel Qualification and Development Program.

2:30 PM

(ICACC-S13-023-2020) Microstructural inhomogeneity in sintered Fully Ceramic Microencapsulated fuels

C. Ang*[1]; E. Deters[1]; L. Snead[2]; D. Sprouster[2]; Y. Katoh[1]

1. University of Tennessee, Nuclear Engineering, USA
2. State University of New York, Stony Brook, USA

Fully Ceramic Microencapsulated (FCM) fuels using random-2D arrangements of TRISO particles appear to be a promising engineering approach to preventing rupture via sintering. However, the major challenge is that matrix densification is constrained by the presence of non-shrinking particles and inhomogenous temperatures during sintering. Therefore, the properties of the matrix may also be inhomogenous. The hypothesis is examined by crystallite size, grain size and densification of the well-known NITE SiC materials as a function of compaction pressure. The data indicates an increased crystallite and grain size is the primary effect of lower compaction pressure. The results correlate to observations of axial and radial microstructures adjacent to the embedded fuel particles. Above the particle plane, consolidation occurs with more efficient compaction, indicated by more refined microstructures. Coarser microstructures are observed in the interparticle matrix where compaction is less efficient. The data is crucial in determination of differential mechanical and thermal properties in the axial and radial directions relative to the particle fuel plane.

3:10 PM

(ICACC-S13-024-2020) Pressureless Sintering of Fully Ceramic Microencapsulated Fuels

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1. University of Seoul, Dept. of Materials Science & Engineering, Republic of Korea
2. KEPCO NF, Republic of Korea

This study suggests a new quaternary additive composition based on AlN–Y_2O_3–Sc_2O_3–MgO to achieve successful densification of SiC without applied pressure at a temperature as low as 1850 °C. The typical sintered density, flexural strength, fracture toughness, and hardness of the SiC ceramics sintered at 1850 °C without applied

pressure were 98.3%, 510 MPa, 6.9 MPa $m^{1/2}$, and 24.7 GPa, respectively. Fully ceramic microencapsulated (FCM) pellets containing 37 vol% TRISO particles could be successfully sintered at 1850 °C without applied pressure. Forming pressure was important for the successful densification of FCM via pressureless sintering. The residual porosities of the SiC matrix in FCM pellets were 5.8% and 1.6% when the cold isostatic pressing pressure was 135 MPa and 204 MPa, respectively. TRISO particles were not damaged after forming under an isostatic pressure of 204 MPa and subsequent sintering at 1850 °C for 2 h in an argon atmosphere. The thermal conductivities of the SiC matrix sintered with 3.87 wt% AlN–Y_2O_3– Sc_2O_3–MgO and the FCM pellet containing 37 vol% TRISO particles were 67.3 $Wm^{-1}K^{-1}$ and 44.4 $Wm^{-1}K^{-1}$ at 25 °C, respectively.

3:30 PM

(ICACC-S13-025-2020) Non-Destructive Microstructural Analysis of Fully Ceramic Microencapsulated Fuels

D. Sprouster*[1]; C. Ang[2]; B. Ahmadi[3]; J. Favata[3]; S. Shahbazmohamadi[3]; L. Snead[1]; J. Trelewicz[1]

1. Stony Brook University, Department of Materials Science and Chemical Engineering, USA
2. University of Tennessee, Nuclear Engineering, USA
3. University of Connecticut, USA

In the present work, we describe the non-destructive characterization of zero-rupture surrogate fully ceramic microencapsulated (FCM) fuel structures using X-ray based computed tomography. We highlight our efforts to quantitatively capture both matrix and surrogate tristructural isotropic (TRISO) fuel particle morphologies to generate distributions of particle size, distances between the different particle planes, and particle plane information. Our procedures also capture the microstructural defects introduced during processing specifically identifying particles that deviate from their intended plane in the ordered FCM configuration and the number of particles in contact within a given plane. High-resolution computed tomography analysis reveals large cracks around the periphery of contacting particles as well as microcrack formation within the adjacent matrix material. This information is necessary to design structures with desired packing configurations and to model the structural performance during operation for realistic compact morphologies.

Joining Technologies for Reactor Components

Room: Coquina Salon H

Session Chair: Takashi Nozawa, National Institutes for Quantum and Radiological Science and Technology

3:50 PM

(ICACC-S13-026-2020) Joining of Advanced Ceramic Structures for Nuclear Reactors

D. King*[1]; M. Doran[1]; A. Sathish[1]; J. Jarman[2]; J. Watts[2]; W. Fahrenholtz[2]; G. Hilmas[2]

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

Large and complex ceramic, or fiber-reinforced composite, structures are difficult to fabricate as single components; therefore, joining solutions are needed to transform simple shapes, amenable to typical ceramic forming techniques, into functioning structures. Currently, many ceramic composite applications focus on SiC_f/SiC composites; however, it is known that SiC dissociates, rather than melts, under standard temperature and pressure conditions. Fortunately, SiC can be combined with other constituent ceramics, such as ZrB_2, and melted through eutectic melting reactions, during arc welding. When ZrB_2 is fabricated with pure ^{11}B isotope, a SiC-$Zr^{11}B_2$ can be envisioned as a potential next gen reactor material. This talk will focus on accomplishing the goal of fabricating advanced ceramic structures for next generation nuclear reactors via arc welding technologies. The selection of SiC-ZrB_2 ceramics, base material fabrication, welding methods, and the corrosion performance of SiC-ZrB_2 ceramics in air and molten salts, at temperatures up to 1100°C, will be discussed.

4:10 PM

(ICACC-S13-027-2020) Mechanical Properties of Fusion Welds in the SiC-ZrB_2-ZrC System

J. Jarman*[1]; J. Watts[1]; G. Hilmas[1]; W. Fahrenholtz[1]; D. King[2]

1. Missouri University of Science & Technology, Department of Material Science and Engineering, USA
2. UES, Inc., USA

New techniques that would allow the joining of fiber reinforced ceramic composites are currently desired in the nuclear industry. Fusion welding has been shown to join ceramic composites with sufficient electrical conductivity, such as boride-based ceramics. Additional research is needed to determine the elevated temperature mechanical strength of produced welds in boride-based systems. In the current study, mechanical properties of both parent material and welded samples were tested at room and elevated temperatures in the SiC-ZrB_2-ZrC system. Commercial powders of SiC, ZrB_2 and ZrC were batched in various compositions to study the effect of composition on strength while maximizing the SiC content that was welded. Batches were attrition milled, dried and hot pressed to produce 55mm x 55mm x 4mm coupons. Several hot pressed billets were directly machined to ASTM standard test bars with the flexure surface polished to 0.25 um while others were first cut in half and welded back together using gas tungsten arc welding in an inert argon atmosphere, then machined and polished. Mechanical properties including Young's modulus, Vickers hardness, fracture toughness, and four-point bend strength were measured at room temperature while strength and fracture toughness were also measured as a function of temperature. These results will be discussed along with the weldability of ceramics in the SiC-ZrB_2-ZrC system.

4:30 PM

(ICACC-S13-028-2020) Pressure-less joining of SiC/SiC for LWR

M. Ferraris*[1]; V. Casalegno[1]; S. De La Pierre[1]; A. De Zanet[1]; K. Van Loo[2]; C. Lorrette[3]

1. Politecnico di Torino, DISAT, Italy
2. KULeuven, Netherlands
3. CEA, France

Several materials have been proposed for joining of SiC/SiC components for light water reactors. Among them, some glass-ceramics gave promising results as pressure-less joining materials for slurry-based joining technologies suitable for a nuclear environment. For some of them, microstructure and interfaces with SiC have been investigated before and after neutron, and ion irradiation. However, an additional key issue for joined and coated SiC-based materials in LWRs coolant environments is their hydrothermal corrosion. The present work reports on several pressure-less joining options for SiC-based components: some refractory metals and glass-ceramics have been used to join SiC/SiC composites, then tested in autoclave at 330 °C, in PWR water conditions containing 1000ppm B (H3BO3) and 2 ppm Li (LiOH)), under 15 MPa pressure for 14 days, as a pre-screening. Some mechanical test results on first joined composite end-plugs will also be discussed in relation to their density and microstructures. Acknowledgement Part of the research leading to these results has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 740415 Project "IL TROVATORE - Innovative Cladding Materials for Advanced Accident-Tolerant Energy Systems".

4:50 PM

(ICACC-S13-029-2020) Development of Joining Process Technology for CVI/CVD-SiC/SiC Core Materials

S. Suyama*[1]; M. Ukai[1]; M. Akimoto[1]; T. Nishimura[1]; S. Tajima[1]

1. Toshiba Energy Systems & Solutions Corporation, Japan

SiC/SiC composites are attractive engineering materials due to their excellent performance in areas such as thermal stability, high wear resistance, high corrosion resistance, high thermal conductivity, low thermal expansion and low density. In particular, the CVI-SiC/SiC composite has been investigated for use in nuclear reactor cores because of its inherent low activation, low neutron absorption cross-section and stability under irradiation due to its high purity and crystalline SiC matrix. After the nuclear power plant accident in Fukushima, it started to be developed in many research programs as a component of accident-tolerant fuels (ATFs). Toshiba has been developing process technologies for thin-walled and elongated CVI-SiC/SiC-based tubes and for the end plugs of the tubes. The components are required to have good hermetic seal and resist hydrothermal corrosion in light water reactor (LWR) environments. Therefore, the tube material is being developed using a CVD-SiC coated CVI-SiC/SiC composite (CVI/CVD-SiC/SiC). The joining process for end plugs of tubes also requires both properties. This study describes the joining process technology for end plugs of CVI/CVD-SiC/SiC tubes. Toshiba has applied the brazing process by local heating in the area of the end plug, and designed its structure for the end plug of the CVI/CVD-SiC/SiC tube. A joined specimen has shown good hermetic seal.

S14: Crystalline Materials for Electrical, Optical and Medical Applications

Optical Material II

Room: Halifax A/B

Session Chairs: Kenji Toda, Niigata University; Takayuki Yanagida, Nara Institute of Science and Technology

9:00 AM

(ICACC-S14-009-2020) Scintillation properties of fluoride neutron scintillators at elevated temperatures (Invited)

N. Kawaguchi*[1]; T. Kato[1]; T. Yanagida[1]

1. Nara Institute of Science and Technology, Japan

The nuclear logging is a method for gathering information about the layers of rock drilled through. The γ-ray and neutron scintillation detectors which operate at high temperatures (approximately 200 °C) are used for such applications. The Tl-doped NaI single crystal and the Ce-doped lithium silicate glass (Li-glass) are current standard materials for γ-ray and neutron detection at high temperatures, respectively. Both materials show good temperature responses, but the light output of Li-glass is significantly lower than that of Tl-doped NaI single crystal. Although temperature dependence of conventional γ-ray scintillators have been intensively investigated, only few neutron scintillators have been investigated. Thus, it is attractive to investigate performances of other neutron scintillators at elevated temperatures. In the past few years, we have studied scintillation properties of various fluoride-based non-hygroscopic materials for thermal neutron detection (e.g., LiF:W, LiF/CaF_2:Eu eutectic, $LiCaAlF_6$:Ce and $LiCaAlF_6$:Eu). Among these materials, $LiCaAlF_6$:Eu shows the highest light yield (typically ~20,000 photons/MeV). In this study, we have investigated scintillation properties of selected fluoride neutron scintillators at elevated temperatures.

9:30 AM

(ICACC-S14-010-2020) Enhancing the energy resolution of KSr_2I_5:Eu scintillator by compositional engineering (Invited)

L. M. Stand*[2]; M. Zhuravleva[2]; M. Koschan[2]; E. Lukosi[1]; C. Melcher[2]

1. University of Tennessee, USA
2. University of Tennessee, Scintillation Materials Research Center, USA

Arguably the most difficult challenge in scintillator development is reducing the energy resolution to less than 2% at 662 keV, which is required for nuclear nonproliferation applications in order to distinguish gamma-ray signatures of illicit radioactive sources. The work presented here outlines our efforts in this direction, with the best results thus far including light yield of 120,000 ph/MeV and energy resolution of 2.2%. Using the Bridgman technique, we grew single crystals of $KSr_2Br_xI_{5-x}$:Eu and $KSr_{2-x}Ba_xI_5$:Eu, and used a combination of codoping and optimization of both anion and cation ratios in order to obtain the highest achievable light yield and energy resolution out of each mixed composition. To suppress the ^{40}K background, K was successfully replaced by Cs and Tl, giving formation to new $Cs_{1-x}Tl_xSr_2I_5$:Eu scintillators.

10:20 AM

(ICACC-S14-011-2020) Air-stable metal-halide single crystal scintillator $Cs_3Cu_2I_5$: Intrinsic and with TI doping

D. Yuan*[1]

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

Concerning the requirements of low dose utilization and high image quality for single-photon emission computed tomography (SPECT, a nuclear medicine imaging technique), the demanded scintillator must have a high gamma-ray detection efficiency, high light yield with good energy resolution. So far, the most popular scintillator for SPECT is NaI:Tl, which has a high light yield but poor energy resolution. Here, $Cs_3Cu_2I_5$ is found to be a scintillator candidate that can be grown into bulk single crystals using the vertical Bridgman technique. Its intrinsic crystal emits scintillation under Gamma-ray radiation, with a self-trapped exciton (STE) mechanism. After introducing 1% Tl^+ into Cs^+ sites as an activator, we see the main Tl^+ emission together with a small spectral overlap from STE centers, and the light yield is improved by 60% up to 26,000 Photons/MeV (higher if we count the PMT quantum efficiency). It has an energy resolution of 4.5% at 662 keV, and much better light yield non-proportionality (less than 3%) against that of NaI:Tl. With a density of 4.53 g/cm^3 and effective Z value of 52.40, $Cs_3Cu_2I_5$:Tl shows higher stopping power and shorter attenuation length comparing with that of NaI:Tl. The higher energy resolution is another advantage compared with that of NaI: Tl, although a lower light yield that could be improved by increasing Tl concentration in the future.

10:40 AM

(ICACC-S14-012-2020) Flexible ceramics coating on metal or plastics substrate prepared by photo assisted metal organic deposition (Invited)

T. Tsuchiya*[1]; Y. Uzawa[1]; T. Nakajima[1]; I. Yamaguchi[1]; J. Nomoto[1]

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

Metal oxides are expected to be key materials which are used for a new component or devices by controlling metal composition, a crystal structure, orientation or multilayer of the film, a carrier, a spin, etc. However, in most case, the thin film processing temperature is more than 500°C, it is difficult to develop the oxide film on plastic or metal substrates. For these purposes, we have developed the photo-induced chemical solution process(PICSP). By using

the PICSP process, flexible white luminescent phosphor and the long-lasting phosphor thin films(LED lightning applications) were prepared on glass and PET substrates at room temperature. The luminescent thin film shows higher luminescence compared with commercially available one. Additionally, we developed flexible thin film resistor (RuO_2) on polyimide substrate by using a photo reaction of hybrid solution (PRHS) process. The sheet resistance of the flexible RuO_2 film was 0.5~100Ω. Moreover, for the metal coating application, $La_{1-x}Sr_xMnO_3$ film was successfully obtained on the Ni(W) metal tape without oxidation of Ni(W). In this presentation, we demonstrate a preparation of flexible oxide films on plastics and metal substrates by photo-induced chemical solution process at low temperature in air, and their prominent unusual properties of the obtained film.

11:10 AM

(ICACC-S14-013-2020) Properties of metal halide perovskites and their applications in optoelectronic devices

X. Zhang*[1]

1. Southern University of Science and Technology, China

The recent great progress and success in metal halide perovskite optoelectronic devices has verified its designation as "all-powerful material". Its attraction resides not only in the promising characteristics in the application of highly efficient photovoltaic devices for energy harvesting, but also in various kinds of optoelectronic applications, such as LED, lasing, photodetectors, x-ray diffraction detection and plasmonics. The perovskite nanocrystal behaves like quantum dots (QDs), which presents high efficiency quantum yield, tunable bandgap, and narrow-band emission, which is recognized as one of next promising emitting materials. Moreover, the piezoelectric properties of perovskites make it prominent in nanogenerator and self-driving devices.

11:30 AM

(ICACC-S14-014-2020) Relationship between elastic modulus and luminescent properties of phosphors (Invited)

H. Masai*[1]

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

It is conventionally recognized that elastic modulus is affected by the average bond strength and packing density of the matrix. Not only the thermal and mechanical properties but also the durability against high energy light source have some relationships to the elastic modulus. Since formation of defect in materials also correlates with the property, it is worthwhile to examine a relationship between elastic modulus and luminescent properties by various excitation sources. Here, the luminescent properties will be discussed based on various measurement methods concerning the elastic properties. The prompt and storage luminescence depending on the chemical composition and structure will be reported mainly for amorphous materials. The free volumes of materials are also used as a structural parameter for quantification of the structure, in addition to the inelastic light scattering data. Defect-related luminescence will be shown by a combination with ESR results.

Optical Material III

Room: Halifax A/B

Session Chairs: Victoria Blair, US Army Research Laboratory; Dongsheng Yuan, National Institute for Materials Science (NIMS)

1:30 PM

(ICACC-S14-015-2020) Synthesis and photoluminescence of Eu^{2+}-activated silicate phosphors designed by crystal-site engineering (Invited)

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

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

In general, it would be difficult for the selective tuning of a desired emission in phosphors because there is the size mismatch of ionic radius between the luminescent ion and the ion at the specific site. However, if we take into consideration the fact that luminescent ions will enter a less appropriate site by doping in a large concentration, we can expect an observation of the selective emission from the luminescent ions at the less appropriate site. Therefore, the crystal-site engineering is suitable to design new phosphors. Recently, we discovered new red-emitting $Ca_3ZrSi_2O_9$(CZSO):Eu^{2+} phosphors excited by near-UV light region. CZSO with low Eu^{2+} concentrations (Eu^{2+}: 1 mol%) exhibited a green emission at 530 nm. In contrast, with increasing Eu^{2+} concentration to 10 mol%, CZSO:Eu^{2+} phosphors exhibited red emission at 650 nm. We presume for CZSO:Eu^{2+} phosphors with high Eu^{2+} concentration that the green emission from Eu^{2+} occupying the large Ca sites disappeared owing to the concentration quenching of Eu^{2+} and the reabsorption of the green emission by Eu^{2+} occupying the large Ca sites. Thus, the red emission of CZSO:Eu^{2+} phosphors should be attributed to the Eu^{2+} occupying the small Ca sites. Furthermore, we also investigated $Ca_3MgSi_2O_8$:Eu^{2+} and $CaAl_2Si_2O_8$:Eu^{2+} in term of the crystal-site engineering, the results of which will be given at the day of our presentation.

2:00 PM

(ICACC-S14-016-2020) Single phase color tunable phosphors for solid-state lighting applications (Invited)

J. McKittrick*[1]; J. Ha[1]; Y. Kim[3]; E. Novitskaya[1]; Z. Wang[1]; O. Graeve[1]; S. Ong[2]; W. Im[3]

1. University of California, San Diego, USA
2. University of California, San Diego, Department of NanoEngineering, USA
3. Chonnam National University, Republic of Korea

To improve the optical properties of phosphor-converted white-light-emitting diodes, one approach is to utilize near-UV (380-420 nm) LEDs with a mixture of red, green, and blue phosphors. However, in this system, the efficiency of blue emission is poor due to the strong re-absorption of blue light by the red and green phosphors. Single-phase phosphors are considered as a possible solution to avoid this re-absorption issue. $Sr_{2-x}Eu_xLiAlO_4$ (green-emitting) and $Sr_{2-y}Ce_yLiAlO_4$ (blue-emitting) phosphors were prepared by the combustion synthesis method. The emission intensities at 150°C of these compositions were 90% and 88% of the room temperature values, respectively, showing good thermal quenching resistance. Eu^{2+} and Ce^{3+} co-activated Sr_2LiAlO_4 ($Sr_{2-x-y}Eu_xCe_yLiAlO_4$) were prepared to investigate the properties of color tunable single-phase phosphors. The emission color changed from blue, to cool-white, to green, depending on x and y. When y was constant and x increased, the emission intensity of Ce^{3+} decreased and that of Eu^{2+} increased, indicating that there was an energy transfer from Ce^{3+} to Eu^{2+}. With x, y = 0.005, 0.001, the maximum value of the quantum efficiency was 55%, an increase of 40% over those of the singly activated powders. This work was supported by the National Science Foundation, Grant DMR-1411192.

2:30 PM

(ICACC-S14-017-2020) Synthesis of perovskite quantum dot materials using novel water-assisted solid-state reaction method (Invited)

K. Toda*[1]

1. Niigata University, Japan

Perovskite quantum dot (QD) materials were synthesized using a novel water-assisted solid-state reaction (WASSR) method. This novel soft chemical synthesis method is very simple and can synthesize QD materials just by storing or mixing raw materials added a small amount (typically 10wt%) of water in a reactor at low temperature below 500 K. Our proposed low cost and low-temperature synthesis technique are promising for an industrial mass production of QD materials.

3:20 PM

(ICACC-S14-018-2020) Magneto-Optical Materials for Infrared Isolators (Invited)

R. Sharma*[1]; C. Goncalves[2]; R. M. Gaume[1]; K. Richardson[1]

1. University of Central Florida, CREOL, USA
2. CREOL, OCL - GPCL, USA

Faraday rotators provide important isolation functions in optical systems, particularly for laser feedback suppression. Whereas a variety of magneto-optic materials have been developed for visible and near-infrared applications, only a few materials have been characterized for mid- and longwave infrared applications. Candidate optical isolator materials must possess key physical properties, including a large Verdet constant, low absorption, wide transmission and near room temperature operation. This talk summarizes the attributes of candidate materials for integrated magneto-optical devices, reviews state-of-the-art magneto-optic (MO) materials suitable for Faraday isolators, and explores some promising compounds for mid- to long wave infrared applications.

3:50 PM

(ICACC-S14-019-2020) Optical performance of transition metal doped crystalline composites

B. Setera*[1]; D. Sachs[1]; C. Su[2]; B. Arnold[1]; F. Choa[1]; L. Singh[3]; K. Mandal[3]; C. Su[4]; N. B. Singh[1]

1. University of Maryland Baltimore County, Chemistry and Biochemistry and Computer Science and Electrical Engineering, USA
2. University of Maryland Baltimore County, Chemistry and Biochemistry, USA
3. Indian Institute of Technology(BHU), Chemistry, India
4. NASA Marshall Space Flight Center, USA

Approach of transition metal doping in ceramic and non-ceramic matrix to achieve multifunctional materials is more important than ever due to increasing uncertainty of the availability and high cost of the rare-earth elements. A detailed study on the crystal quality absorption, emission, morphological and characteristics of transition metal (TM) doped crystalline urea and binary and ternary selenides have been investigated to understand their potential application as room-temperature tunable mid-infrared lasers and g-ray detectors. Their performance is compared with single Tm-doped ZnSe crystals. Bulk quality, point defects and crystallinity was evaluated by optical transparency, scanning electron microscopy (SEM) and X-ray diffraction pattern. Effect of TM on the optical performance of material was observed to be significantly different in both host materials. In addition, these composites have potential for rad detection. The g-ray performance was determined for the ceramic thin film as well as ceramic – organic composite. The radiation exposure and sensitivity was determined by measuring current –voltage characteristics of irradiated and non-irradiated composites.

4:10 PM

(ICACC-S14-020-2020) Magnetic Field-Assisted Finishing of Fused Silica Laser Optics

J. T. Long*[1]; D. Poljak[1]; Y. Funamoto[2]; D. Shima[2]; H. Marui[2]; T. Kamimura[2]; H. Yamaguchi[3]

1. University of Florida, Materials Science and Engineering, USA
2. Osaka Institute of Technology, Engineering, Japan
3. University of Florida, Mechanical and Aerospace Engineering, USA

Polished fused silica often has subsurface damage and a layer of contamination. The damage and contamination can be present at depths of up to 100 μm and 100 nm, respectively, and have proven challenging to remove. The presence of contamination on the surface of polished fused silica also contributes to a significant reduction in the laser-induced-damage threshold (LIDT). Magnetic field-assisted finishing (MAF) using a synthetic polishing cloth has been previously applied to remove the subsurface damage and the contamination layer from fused silica, and the process feasibility was demonstrated by an improved surface LIDT. However, the polishing characteristics are strongly dependent on the polishing pad conditions. This paper proposes the removal of the fused silica contamination layer using MAF with an iron-particle brush as a polishing tool, which, unlike the synthetic polishing cloth used previously, does not degrade during polishing and is reusable. This will help maintain consistent material removal regardless of polishing time. Polishing experiments using MAF with an iron-particle brush will demonstrate the polishing characteristics on the surface of fused silica and the prevention of iron particle contamination.

4:30 PM

(ICACC-S14-021-2020) Towards high-throughput R & D of solid state lasers by crystal engineering and informatics (Invited)

T. Matsuura*[1]; H. Koinuma[1]

1. SCT INC, Japan

A R&D proposal from our venture company is chosen as a fundamental research within a big Japanese Governmental competitive funding scheme, where Government is purposing to fund novel and challenging fundamental researches that are potentially changing/creating a field of new science. In our proposed project, we are organizing a strong research consortium with several universities, research institutes and companies. The purpose of the research is to create the innovative research protocol for finding new smart materials, where combinatorial laser MBE technology and intelligent materials informatics are to be hybridized. We are challenging for three types of laser materials: high power laser from rare-earth-oxide single crystals, quantum cascade lasers (QCL) using new semiconductors of ZnO and electrically-pumped lasers using perovskite halides. Through this project we aim at making a paradigm shift of materials R&D by design and creation of a new smart model of combinatorial laser MBE (CLMBE) with intelligent functions such as autonomous operation/security and big data management/application. This will be a game changer with very-high-throughput materials-research. The size of the funding is multimillion dollars every fiscal year for initially 3 fiscal year until 2021, and potentially furthering for 2 years. We are open for fruitful discussion and potential collaboration.

S15: 4th International Symposium on Additive Manufacturing and 3-D Printing Technologies

Stereolithography I

Room: Coquina Salon B
Session Chair: Jens Bauer, University of California, Irvine

8:30 AM

(ICACC-S15-010-2020) Additive Manufacturing of Polymer-Derived Ceramic Composites (Invited)

K. A. Porter[1]; M. R. O'Masta[1]; P. P. Bui[1]; E. Stonkevitch[1]; Z. C. Eckel[1]; T. Schaedler*[1]

1. HRL Laboratories, USA

Reinforcement of ceramics with a second ceramic phase is a well-established method to create a composite with strength and toughness beyond either constituent material. However, 3D printing approaches that rely on surface interactions for sintering are limited in the amount of reinforcement that can be incorporated. Here we discuss the addition of ceramic reinforcement media to silicon-based, pre-ceramic resins that can be printed on commercial stereolithography printers and subsequently converted into a ceramic matrix composite (CMC) by pyrolysis. We will discuss the use of various reinforcement materials, form factors (e.g. particles and whiskers) and volume fractions in relation to performance and compatibility with the printing method. The CMCs are over an order magnitude stronger and four times tougher than the base polymer-derived ceramic. In addition, we investigate the high temperature (>1000C) properties of the 3D printed CMCs.

9:00 AM

(ICACC-S15-011-2020) New applications of hybrid multi-materials and smart design

C. Chaput*[1]; R. Gaignon[1]

1. 3DCERAM SINTO INC, USA

3DCERAM brings together unique expertise in the field of 3D ceramic printing to develop a unique 3D printing process, based on laser stereolithography technology. 3D printing lets users push back production limits. Ceramics are no exception to the rule. Thanks to the unique process, 3DCERAM can produce ceramic parts with porosity structured in 3 dimensions. Applied to the biomedical market, the process allows the realization of eyeballs, bone substitutes or made-to-measure cranial implants in ceramics showing exceptional performance. To open up this technology to a wider spread of professionals 3DCeram proposes multi-material solutions. The new Ceramaker Hybrid is able to print several materials at the same time and can manufacture smart design parts. After printing, the part is debinded and sintered and by this way the resin deposit in place of internal cannels was burned. After sintering we can obtain pure alumina parts with internal channels inside. With 3DCERAM, 3Dprinting is just not repeating what we can do with other production technology but is going a step forward.

9:20 AM

(ICACC-S15-012-2020) Mechanical and microstructural properties of ceramics produced by lithographic additive manufacturing

M. Schwentenwein*[1]; T. Lube[2]; R. Danzer[2]; J. Homa[1]

1. Lithoz GmbH, Austria
2. Montanuniversitaet Leoben, Austria

In recent years additive manufacturing (AM) techniques started to become a relevant fabrication method in the field of high-performance ceramics. The increasing importance of these components also requires the availability of testing and characterization protocols that ensure the quality and homogeneity of parts made by AM and to allow for a proper understanding of the relevant structure-property relationships. One of the most important aspects is the strength and isotropy of additive manufactured components. In this contribution the mechanical and microstructural properties of specimens from additive manufactured alumina, zirconia and silicon nitride components fabricated by lithography-based ceramic manufacturing (LCM) are presented. The influence of different testing orientations with respect to the building direction was investigated. It is shown that proper choice of the materials system as well as the printing and post-processing parameters are important to realize a homogeneous and isotropic microstructure and thus, ceramic components with isotropic material properties and no artifacts from the layer-by-layer build-up. In terms of strength, the obtained values for the AM parts are already at eye-level with those made using conventional fabrication techniques such as isostatic pressing or injection molding.

9:40 AM

(ICACC-S15-013-2020) Fabrication of Ceramic Objects with Fluctuated Patterns by Ultraviolet Laser Stereolithography

S. Kirihara*[1]

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

Naturally fluctuated patterns were systematically processed by stereolithographic additive manufacturing for material and energy fluids modulations in living environments. Inverse proportions of amplitudes to exponential frequencies in fluctuation formulas can be derived from Fourier series expansions of naturally wavy profiles. Fluctuated surfaces on solid models were graphed through inverse Fourier transformations. Geometrically modulated artifacts composed of alumina were reproduced by computer aided- design, manufacture and evaluation. Ceramic particles were dispersed into photosensitive resin at 50 vol. % to create paste material. An ultraviolet laser beam was scanned on the paste material spread by a knife edge, and a two-dimensional cross section was formed by the resin dewaxing and powder sintering. Through automatically layer laminations, a three-dimensional object with fluctuated patterns could be fabricated. Water or gaseous flows and electromagnetic wave propagations on the formed fluctuated components were simulated theoretically and measured experimentally.

Stereolithography II

Room: Coquina Salon B
Session Chair: Tobias Schaedler, HRL Laboratories

10:20 AM

(ICACC-S15-014-2020) Two-Photon-Polymerized Nanoarchitected Materials and Metamaterials (Invited)

J. Bauer*[1]; C. Crook[1]; A. Guell Izard[1]; Z. C. Eckel[2]; T. Schaedler[2]; L. Valdevit[1]

1. University of California, Irvine, USA
2. HRL Laboratories, USA

One- and two-dimensional nanoscale objects, such as nanowires and thin films, are known to hold exceptional physical properties. Yet, their properties are intrinsically coupled to their small size and their solitary nature and can hardly be accessed in actual materials of practical volume. Nanoarchitectures like 3D networks constructed from nanowires or thin films have the potential to overcome such limitations. Two-photon polymerization direct laser writing (TPP-DLW) is the most versatile technology for fabricating nanoarchitected materials and metamaterials and rapidly progresses towards higher throughput. However, TPP-DLW is today still largely empirical, without systematic knowledge on the dependency of material properties and process parameters and mostly limited to polymers. Here, we systematically characterize the mechanical properties of TPP-DLW-printed polymeric and polymer-derived ceramic materials and metamaterials. We explore the interplay of material systems including acrylic polymers, glassy carbon and silicon oxycarbide,

with size-effects and different topological designs; and show unique characteristics, like targeted tailorability of strength and stiffness via TPP-DLW process parameter selection, non-brittle deformability of ceramic materials and architectures, and mechanical performance at the theoretical limits of strength and stiffness.

10:50 AM

(ICACC-S15-015-2020) Design for AM and thermal treatment: The influence of topology on SLA 3D-printing, debinding and sintering of Alumina periodic architectures

O. Santoliquido*[1]; G. Bianchi[1]; R. König[1]; M. Spaggiari[1]; A. Ortona[1]

1. SUPSI, MEMTi, Switzerland

Complex ceramic components can be produced nowadays by the stereolithography technique. However, this method has a strong limitation in the production of massive components, called bulk, having solid walls thicker than approx. 10 mm. This is due to the high content of organic binder in the green bodies, which during the thermal debinding leads to the formation of defects such as cracks and delaminations. The obstacle can be overcome by introducing the so-called "design for AM approach", and in particular by substituting the bulk volume with a porous architecture. This also allows handling the component weight and its behavior when subjected to an external load (*i.e.* mechanical properties). In the present study, different tubular lattices were 3D-printed starting from an Alumina-based photosensitive suspension. Through a DOE approach different combinations between the characteristic parameters of the architectures were tested. For each elementary cell type (cube, rotated cube, octet or tetrakaidecahedron) the porosity range was varied in terms of walls thickness and struts diameter. The behavior during the sintering phase (*i.e.* deformations) was studied through morphological inspections, while the mechanical properties (*i.e.* stresses and deformations) were assessed through a FEM analysis by applying a force on the external surface.

11:10 AM

(ICACC-S15-016-2020) Establishment of the Degree of Dispersion Effect in Stereolithography

M. K. Alazzawi*[1]; B. Beyoglu[1]; R. A. Haber[1]

1. Rutgers University, Materials Science and Engineering, USA

Stereolithography processes result in an undesirable anisotropic shrinkage, the causes of which are unresolved. The degree of dispersion of stereolithography suspensions may impact the depth of cure and the anisotropic shrinkage of formed objects. This research aims to study whether a higher degree of dispersion can result in lower anisotropic shrinkage and higher depth of cure. In this study, the degree of dispersion of suspensions was altered by applying mechanical shear stress. Two different mixers were applied with various mechanical shear stresses, and their parameters were investigated. The degree of dispersion was assessed using a developed method combining fineness of grind and cohesive energy density measurements. The suspensions were printed using a stereolithography apparatus. The shrinkage and depth of cure were characterized. These findings will be discussed.

11:30 AM

(ICACC-S15-017-2020) Additive Manufacturing of Thin Shell Mold via A Support-Free Suspension-Enclosing Projection-Stereolithography Process

X. Song*[1]; L. He[1]

1. University of Iowa, Industrial and Systems Engineering, USA

Stereolithography has attracted a lot of interest in fabricating complex ceramic objects, including thin shell molds for shell-mold casting. However, when building mold structures with complex overhangs, this process requires creating support structures underneath, which consequently lead to many issues in the resulting components, such as poor surface quality, high risk of damage, etc. In this article, a new Suspension-Enclosing Projection-Stereolithography (SEPS) process is presented to enable the support-free fabrication of ceramic structures with complex overhangs. In this process, a high solid-loading ceramic slurry is used as the feedstock material. The support-free fabrication of an overhang is achieved by dynamically tuning the temperature-dependent yield stress of the feedstock material before and after the material is coated via a modified slot-die extruder. The rheological properties of the ceramic slurry under different temperatures are studied. This new process provides an efficient route for fabricating complex shell molds with overhanging cavities.

Application of Materials and Components

Room: Coquina Salon B

Session Chair: Soshu Kirihara, Osaka University

1:30 PM

(ICACC-S15-018-2020) New designs and applications of porous ceramic components: The unmissable advantage of additive manufacturing (Invited)

A. Ortona*[1]

1. SUPSI, MEMTi, Switzerland

Ceramic components have been used by mankind as indispensable tools for thousands of years. In some recent application, their properties were so unique that, they become the one and only materials' choice. On the other side ceramic components do have drawbacks: on top there are their mechanical behaviour and processing. the Hybrid Materials Laboratory presents several innovative porous ceramic components which were developed in different fields: automotive, aerospace, combustion, heat exchange and storage, filtration, catalysis and concentrated solar energy. This components' outstanding performances could be obtained only by combining design and computer simulations with additive manufacturing.

2:00 PM

(ICACC-S15-019-2020) Printing ceramic porous structures for high-temperature applications

S. Sobhani*[1]; P. Muhunthan[1]; E. Boigne[1]; S. M. Allan[2]; M. Ihme[1]

1. Stanford University, Mechanical Engineering, USA
2. Lithoz America, LLC, USA

Macroporous ceramic structures enable high-temperature and corrosion resistance while maintaining a low pressure-drop, and are therefore applied to numerous fluid flow applications. In this work, a framework is proposed for designing and manufacturing tailored porous structures of alumina for application to high-temperature environments. Lithography-based Ceramic Manufacturing is implemented, enabling production of complex and customized structures on a layer-by-layer basis from Computer Aided Design (CAD) model data. First, the printed samples were tested to characterize the effects of feedstock particle diameter, layer thickness, and printing orientation on the macro-scale thermal and durability properties of LCM-alumina. SEM and EDX analysis techniques were employed to test for material degradation and chemical changes, and XCT imaging was used to test the fidelity of the printing technology in reproducing the input STL geometry. Next, the benefits of microstructure tailoring are illustrated experimentally for a porous media burner operating with a smoothly graded matrix structure.

Selective Laser Melting and Sintering I

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

2:20 PM

(ICACC-S15-020-2020) Selective Laser Sintering of Electroceramics for Radio Frequency and Microwave Applications

R. Gheisari*[1]; A. Goulas[1]; D. Engstrom[1]

1. Loughborough University, Mechanical Engineering, United Kingdom

Telecommunication, microwave electronics and antennas have transformed our lives in the past decade by enabling wireless transfer of vast amounts of data to electronic devices at home or at work. Information is needed in all areas of society and instant access can create more wealth, better health and an improved environment. Additive manufacturing (AM) of electroceramics offers a unique route for fabrication of novel 3D radio frequency (RF) and microwave communication components, embedded electronics and sensors for exploitation within the Internet of Things. A variety of different AM techniques capable of fabricating 3D ceramic parts have been developed over the past years, such as stereolithography, fused deposition modeling (FDM), selective laser sintering/melting (SLS/M), and slurry-based 3D printing [1–4]. A significant advantage of the SLS 3D printing is that it reduces fabrication time and allows producing multi-material structures (e.g. ceramic/metal) using in-situ laser sintering where the materials chemical incompatibility is an issue using conventional sintering methods. The proposed process uses formulations similar to those in conventional tape casting and screen printing. Thus, the SLS of electroceramics such as bismuth molybdate and barium titanate to be used for RF and microwave applications has been investigated in this research.

2:40 PM

(ICACC-S15-021-2020) Doped ceramic granules for shaping of complex alumina components via laser additive manufacturing

S. Pfeiffer*[1]; K. Florio[2]; M. Makowska[3]; H. Van Swygenhoven[3]; K. Wegener[2]; C. Aneziris[4]; T. Graule[1]

1. Empa, Swiss Federal Laboratories for Materials Science and Technology, High Performance Ceramics, Switzerland
2. ETH Zurich, Institute of Machine Tools and Manufacturing, Switzerland
3. PSI, Photon Science Division, Switzerland
4. TU Freiberg, Institute of Ceramic, Glass and Construction Materials, Germany

Producing high performance ceramic components with complex shapes in an accurate manner is a key challenge in ceramics processing. Most of the additive manufacturing processes produce only green bodies that still need cost-intensive post-processing and time-lasting sintering. Selective direct laser sintering (SLS) has a great potential to solve this shortcoming. The application of SLS to ceramics remains challenging when compared to polymer and metal processing. Several approaches i.e. use of different laser strategies, preheating temperatures and material systems, have been investigated to overcome these problems. However, challenges like crack formation and weak densification still need to be resolved. A better understanding in the interaction of the laser with the ceramic is also crucial to consolidate ceramic powders to dense structures with adequate material properties without a post-processing step. Based on our approach highly dense granules consisting of alumina with a good flowability were manufactured by spray drying. Coloured metal oxide nanoparticles were uniformly implemented within the granules to improve the interaction with the applied laser in the visible range. Dibasic ammonium citrate was found to be an appropriate dispersant. By laser additive manufacturing, granules were consolidated and alumina components with densities up to 97% were produced.

Selective Laser Melting and Sintering II

Room: Coquina Salon B
Session Chair: Alberto Ortona, SUPSI

3:20 PM

(ICACC-S15-022-2020) Additive Manufacturing of Net-Shape Non-Oxides using Photothermochemically-assisted Reaction Bonding

A. B. Peters*[1]; D. Zhang[2]; M. Brupbacher[2]; A. Hernandez[1]; D. Nagle[1]; T. Mueller[1]; J. Spicer[1]

1. Johns Hopkins University, Materials Science and Engineering, USA
2. Johns Hopkins University, Applied Physics Lab, USA

Non-oxide ceramics have an exceptionally useful set of materials characteristics but cannot generally be formed using additive manufacturing (AM) due to poor thermal shock resistances and large volumetric changes that occur during processing. In this work, we demonstrate an approach for AM of non-oxides using a precursor system that isovolumetrically converts to the desired ceramic using laser-induced, gas-solid reactions. By tailoring the ratio of metal/metal-oxide precursor constituents for a given reactant atmosphere (e.g. CH_4 for carbides or NH_3 for nitrides), volume changes associated with the conversion of each component compensate such that non-oxides are formed with no net-volume change between the solid precursors and product. Specifically, model systems composed of 86/14 and 67/33 molar Cr/Cr_2O_3 were processed in CH_4 and NH_3 respectively to yield Cr_3C_2 and CrN. Microstructural analysis indicates net-shape conversion was achieved using both furnace and laser-induced processing, where reaction bonding between particles differed from conventional AM sintering and may circumvent complications associated with traditional processing methods. With proper selection of metal/metal-oxide precursors and reactant gases, this synthesis approach is broadly applicable to the formation of other carbides, nitrides and borides via selective laser reaction sintering AM.

3:40 PM

(ICACC-S15-023-2020) Additive Manufacturing of YSZ Ceramics by Laser Engineered Net Shaping

X. Yan[2]; Y. Chen[1]; F. Wang[2]; C. Kanger[2]; M. Sealy[2]; B. Cui*[2]

1. Oak Ridge National Laboratory, USA
2. University of Nebraska-Lincoln, USA

Compared to metals, laser additive manufacturing of ceramic materials is more challenging because of the intrinsic brittleness of ceramics and the high temperature gradients, which can induce significant defects and cracking. This presentation shows our novel research on the successful additive manufacturing of yttria stabilized zirconia (YSZ) ceramics by a laser engineered net shaping (LENS) process. The microstructure formation, such as phase composition, texture, and cracks, has been carefully characterized by neutron diffraction and electron microscopy, which are correlated with the LENS conditions such as the laser power. Phase transformation from monoclinic to tetragonal/cubic occurred during the LENS of YSZ ceramics. The crack density inside the manufactured parts was reduced at a higher laser power.

4:00 PM

(ICACC-S15-024-2020) Influence of Process Parameters on the Microstructure and Surface Properties of Laser Additive Manufactured Ti-6Al-4V Composite

Y. Du[1]; S. Fatoba*[1]; H. Feng[1]

1. Kent State University, USA

The effects of processing parameters, namely laser intensity, powder feed rate and scanning speed of Laser Metal Deposition (LMD) process on the microstructure, metallurgical evolution, porosity generated in the coating, the geometrical property of the coating and the sizes of the grains in the coating were investigated.

The processing parameters were applied in combinations in order to find the optimized settings of the process that least affects the metallurgical properties of the Ti-6Al-4V alloy substrate cladded with reinforced aluminum based powder. The temperature gradient and the rate of solidification of reinforcing the Ti-6Al-4V substrate with the aluminum based power were also focused on in terms of how they were influenced by the laser intensity and the scanning speed used in the building process. The inherent material properties were dependent on the process input parameters. The characterized performances considered in the investigation was influenced significantly by the laser processing intensity. The results of the investigation showed that the density had increased in proportion to the increase of the processing laser power coupled with the reduction of the speed of the laser scan. The increased temperature field led to changes in geometry of the coatings as a results of more absorbed laser energy.

4:15 PM

(ICACC-S15-025-2020) Microstructural Enhancement and Performance of Additive Manufactured Ti-6Al-4V Composite Coatings

Y. Du[1]; S. Fatoba*[1]; H. Feng[1]

1. Kent State University, USA

The investigation focused on studying how the reinforcement powders and titanium alloy substrate were influenced by the volumetric energy absorbed. The processing parameters investigated were the laser intensity and the scanning speed. Laser surface modification technique has unique benefits and properties compared to other conventional techniques. These process factors directly affect the microstructure and properties of printed materials. The results revealed dense microstructure in the fabricated coatings in terms of the microstructural evolution, the sizes of the different grains, the structure of the phases formed and the orientation. The modified surface layer of the additively manufactured coating had an improved and a fine microstructure. Optimizing the direct laser metal deposition (DLMD) processing conditions resulted in a crack-free surface layer but still promoted a few population of gas defects. Morphological observations along the side of the surface showed the prominence of the adhesive powder, flow path of the melt pool and the areas which overlapped, attributed to the increase of the laser scanning speed. When the laser scanning speed was increased, it attributed to the grain width reduction of the prior beta and a transformation in shape of the martensitic primary alpha into fine needle-like structures.

4:30 PM

(ICACC-S15-026-2020) Effects of relative humidity at partially elevated temperature on 3D printed ordinary Portland cement (OPC) concrete

A. Ur Rehman*[1]; V. M. Sglavo[1]

1. University of Trento, Department of Industrial Engineering, Italy

The high relative humidity has positive and negative influence at ambient and high (above 300) temperatures respectively on both the hydration reaction, and the mechanical properties development of concrete. However, the effects of relative humidity at partially-elevated temperatures are unknown. This work aims to elucidate the influence of relative humidity (hundred percent) on mechanical properties of 3D printed OPC concrete. In this paper, MOR and density of concretes cured for different periods (24, 48, 72, and 96 hours) and exposed to different temperatures (90°C and 115°C) with relative humidity of hundred percent were obtained. The effects of the duration of curing and temperature on the strengths of concrete were investigated. Experimental results indicate that at relative humidity of hundred percent, after exposure to partially elevated temperatures, early-age concrete that has not been cured for a certain period cannot gain strength.

4:45 PM

(ICACC-S15-027-2020) Influence of laser parameters and material properties in selective laser sintering and melting (SLS/M) of MgO-Al_2O_3 ceramics

A. Ur Rehman*[1]; L. TingTing[1]

1. Nanjing University of Science and Technology, School of Mechanical Engineering, China

Selective laser melting and sintering (SLS/M) of MgO-Al_2O_3 ceramics has been investigated. The purpose of the work was to analyze the effects of the change in material proportions and laser parameters during SLS/M process. Several eutectic ratios of MgO: Al_2O_3 from 80: 20, 40: 60, 25: 75, and up to 10:90 ceramics wt% were printed for this purpose. Several specimens on various laser powers ranging from 70W to 90W were fabricated. The change in eutectic phases during the process was also evaluated. It was found that the surface quality of SLS/M printed samples improves with the increase in Al_2O_3 quantity. The increase in laser power can result in sintering, partial melting to complete melting in the printed article. The fascinating finding during the phase analysis found that although the eutectic phases are also changing during the SLS/M process, the rapid melting and solidification during the process does not allow enough time to reach the completion of the intermediate phases during the SLS/M. Moreover, qualitative and quantitative phase analysis showed that there were some phases present that were achievable at a much higher temperature (in the specific eutectic ratio), then the temperature reached during the SLS/M process noted by infrared (IR) camera. These findings could also be valid for other eutectic phase ceramics in SLS/M.

S17: Advanced Ceramic Materials and Processing for Photonics and Energy

Advanced and Nanostructured Materials for Photonics, Electronics and Sensing III

Room: Tomoka C

Session Chair: Federico Polo, Ca' Foscari University of Venice

8:30 AM

(ICACC-S17-010-2020) 2D layered materials: A promising family for hydrogen production through water splitting (Invited)

T. A. Shifa*[1]

1. Luleå University of Technology, Department of Engineering Science and Mathematics, Sweden

Owing to its high energy density and non-polluting characteristics, molecular hydrogen through water splitting (WS) has been regarded as one of the most promising green fuels for powering the globe. In such quest, layered materials are acknowledged for their fascinating performances in catalyzing WS. Here, I present the experimental explorations based on two families of layered materials: transition metal dichalcogenides (TMDs) and transition metal phosphorus trichalcogenides (MPX_3) towards WS via electrocatalysis and photocatalysis. Of the many approaches to improve the intrinsic activity of TMD's catalysis, doping and hybrid material formation are believed to play a significant role in optimizing the free energy of hydrogen adsorption and desorption. Hence, a well-matched rGO/WS_2 hybrid, phase pure $Co_xW_{(1-x)}S_2$ and $WS_{2(1-x)}P_{2x}$ electrocatalysts have been controllably synthesized for catalyzing WS reactions. In photocatalysis, theoretical studies reveal that auspicious activity in WS can be realized from the newly emerged layered $MnPX_3$ materials. However, experimental efforts have so far been challenged with the synthesis bottleneck. We employed a preheating strategy in chemical vapor deposition technique to be able to grow these materials. The obtained $MnPX_3$ nanosheets demonstrate a promising activity in sacrificial agent-free photocatalytic water splitting under simulated solar light.

9:00 AM

(ICACC-S17-011-2020) Multifunctional 2-D oxides: Key roles of defects and nm-scale synthesis (Invited)

S. T. Misture*[1]

1. Alfred University, MSE, USA

Vanadate, titanate, niobate and MnO_2 2-D nanosheet assemblies have been studied in detail to define the effects of atomistic defects and nanostructure on charge transport, charge storage and catalytic properties. We find a direct link between charged defects and the optical, chemical, photochemical and electrochemical function. We focus our studies on 2-D nanosheets obtained by exfoliation or hydrothermal synthesis, and on defects on the metal sublattice; that is, we introduce aliovalent substituents or reduce or oxidize some of the metal cations using suspension pH or gas-phase reduction. We further consider the nanostructures that result from controlled re-assembly of 2-D oxide nanosheets with and without extensive re-stacking. The results demonstrate improvements in charge storage and charge transfer by as large as a factor of 10 are defined by the defects, for example, and that controlling the extent of disorder in nanostructures is feasible for tuning the material response.

9:30 AM

(ICACC-S17-012-2020) Novel calcium ion conducting solid with NASICON-type structure (Invited)

N. Imanaka*[1]

1. Osaka University, Applied Chesistry, Japan

Divalent calcium ion conducting solid electrolyte with a three dimensional NASICON-type structure, $(Ca_xHf_{1-x})_{4/(4-2x)}Nb(PO_4)_3$, was successfully synthesized by introducing Ca^{2+} cations into the $HfNb(PO_4)_3$ solid, since three kinds of high valent cation of Hf^{4+}, Nb^{5+}, and P^{5+} are expected to effectively reduce electrostatic interaction toward Ca^{2+} in the structure. The compositional dependence of the lattice volume of the NASICON-type phase was investigated. As a result, by introducing Ca^{2+} ions into the Hf^{4+} site in the structure, the lattice volume of the NASICON-type phase monotonously increased with the increase of x up to 0.05, owing to the replacement of the Hf^{4+} (ionic radius: 0.085 nm) site with the larger Ca^{2+} (ionic radius: 0.114 nm) ion. For the solids with $x \geq 0.05$, the lattice volumes were kept almost constant. These results clearly suggest that the solid solubility limit of the single phase of NASICON-type $(Ca_xHf_{1-x})_{4/(4-2x)}Nb(PO_4)_3$ is approximately x = 0.05. The conductivity monotonously enhanced with the Ca^{2+} content until the solid solution limit of x = 0.05. The conductivity of the $(Ca_{0.05}Hf_{0.95})_{4/3.9}Nb(PO_4)_3$ solid was ca. two orders of magnitude higher than that of previously reported NASICON-type $Ca_{0.5}Zr_2(PO_4)_3$ solid.

Advanced and Nanostructured Materials for Photonics, Electronics and Sensing IV

Room: Tomoka C
Session Chair: David Kisailus, University of California, Riverside

10:10 AM

(ICACC-S17-013-2020) Luminescent Organic Solids with Unusual Exciton Multiplicity (Invited)

E. Hamzehpoor[1]; C. Liu[1]; D. Perepichka*[1]

1. McGill University, Chemistry, Canada

High quantum yield luminescent organic solids are required for a number of optoelectronic applications, including organic light-emitting diodes (OLEDs) and transistors (OLETs), lasers, etc. The absolute majority of organic materials can only emit efficiently from their singlet excited states. Involving high-multiplicity states (such as triplets) in exciton dynamics has several established and potential benefits; it is required to overcome 25% theoretical efficiency in OLEDs and might enable various magneto-optical devices. We will report our recent finding of (i) purely organic (C, H, N, O only) room temperature phosphors and (ii) stable free radical fluorophores with record-breaking efficiency of triplet and singlet emission, respectively. The lecture will discuss the molecular and supramolecular mechanisms in modulating the emission from high-spin excited states in organic solids.

10:40 AM

(ICACC-S17-014-2020) A Closer Look at Electron-Phonon Landascapes in Organic Crystals (Invited)

E. Orgiu*[1]

1. Institut National de la Recherche Scientifique (INRS), EMT Centre, Canada

Charge transport in organic semiconductors is notoriously extremely sensitive to the presence of disorder, both intrinsic and extrinsic, especially for n-type materials. Intrinsic dynamic disorder stems from large thermal fluctuations both in intermolecular transfer integrals and (molecular) site energies in weakly interacting van der Waals solids and sources transient localization of the charge carriers. The molecular vibrations that drive transient localization typically operate at low-frequency (< a-few-hundred cm^{-1}), which renders it difficult to assess them experimentally. Hitherto, this has prevented the identification of clear molecular design rules to control and reduce dynamic disorder. In addition, the disorder can also be extrinsic, being controlled by the gate insulator dielectric properties. In my talk I will discuss a comprehensive study of charge transport in two closely related n-type molecular organic semiconductors using a combination of temperature-dependent inelastic neutron scattering and photoelectron spectroscopy corroborated by electrical measurements, theory and simulations. We provide unambiguous evidence that ad hoc molecular design enables to free the electron charge carriers from both intrinsic and extrinsic disorder to ultimately reach band-like electron transport.

11:10 AM

(ICACC-S17-015-2020) Molecular Approaches to Create Efficient Shortwave Infrared Emitters (Invited)

J. R. Caram*[1]

1. University of California, Los Angeles, Chemistry, USA

The shortwave infrared (SWIR) is the spectral window for electromagnetic radiation with wavelengths from 1-2 microns. It occupies a special place for applications, as it is lower energy than most molecular and semiconducting bandgaps yet higher energy than most molecular vibrations and earth-based blackbody radiation sources. For this reason, this window is utilized in background free biological and scenery imaging, LIDAR and telecom applications. There is a need for molecular and nanoscale moieties which absorb and emit in this spectral window. In this presentation, I demonstrate how one can systematically improve a molecular chromophore system to achieve efficient SWIR absorption and emission, and describe a roadmap for improving dye photophysics in this window.

11:40 AM

(ICACC-S17-016-2020) Fine-tuning of photoluminescence and electrochemiluminescence in bifunctional organic dyes (Invited)

F. Polo*[1]; F. Rizzo[2]; G. Valenti[3]

1. Ca' Foscari University of Venice, Molecular Sciences and Nanosystems, Italy
2. National Research Council of Italy, Institute of Molecular Science and Technologies, Italy
3. University of Bologna, Department of Chemistry "G. Ciamician", Italy

In electrochemiluminescence (ECL), electrochemical activation of suitable molecules or ions generates species that undergo sufficiently exergonic electron-transfer reactions to form excited state species (emitters) capable of causing luminescence. There is an increasing demand for the synthesis, characterization and application of new ECL materials, such as organic dyes, metal complexes,

and nanoparticles that could allow for fine-tuning of the emission wavelength. This holds a tremendous potential for future applications of multicolor ECL, as for example in organinc light-emitting diodes and electrochemical cells, and in multiplexed detection of bioanalytes. Organic compounds could offer a more general answer to this challenge particularly because of their chemical versatility and, therefore, the possibility of modulating electronic and photophysical properties precisely. A new class of bifunctional organic dyes, consisting oftwo triphenylamines linked by a fluorene or spirobifluorene bridge, has been recently considered as suitable candidate for ECL. The phenylamine moieties were chemically modified at the para position by electron-withdrawing or electron-donating substituents. This allowed fine-tuning of the photoluminescence and ECL emission from blue to green. Solid state ECL generated by a monolayer of spirobifluorene derivative, covalently bound onto ITO, will be also shown.

Advanced and Nanostructured Materials for Photonics, Electronics and Sensing V

Room: Tomoka C

Session Chair: Alberto Vomiero, Lulea University of Technology

1:30 PM

(ICACC-S17-017-2020) Hightly efficient optical humidity sensors (Invited)

R. Nechache*[1]

1. Institut National de la Recherche Scientifique, Energy, Material and Telecommunications, Canada

Humidity sensors have gained importance in recent years because of the need to monitor and control environmental relative humidity in various industrial processes particularly in various chemical processes. Research has been carried out on various trans-domain systems to explore the possibility of utilizing them as sensors. To enhance the performance of sensors, we use the generation of surface plasmon resonance (SPR) phenomenon at the interface between the metal and the dielectric in the fiber. However, these metallic coatings mainly composed of silver or gold films do not contain systematically nanostructures (NSs) which are a key parameter promoting the SPR generation. SPR has its own advantages in the ability to control and thus optimize the sensitivity or limit of detection values of sensor through the change of the sizes and shapes of coated NSs. We investigate the SPR properties of obtained NSs and the influence of their surface chemistry on the optical and electrical properties of the bent fiber and their performance for humidity sensing. Various metal-based NSs have been successful synthesized by chemical methods however and pulsed laser ablation in liquid (PLAL). Here, we will present the optimisation of the nanoparticles PLAL synthesis and the tuning of their morphology and optical properties. The influence of their surface chemistry on performance of optical humidity sensor will be also discussed.

2:00 PM

(ICACC-S17-018-2020) Hybrid organic-inorganic layers grown on metal-transition oxides as multifunctional materials for sensing and energy storage (Invited)

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1. University of Catania, Dipartimento di Scienze Chimiche, Italy

Surface modifications of metal-transition oxides with organic or organic-inorganic coatings is the focus of much current research due to the large variety of their applications, ranging from sensing and environmental control to energy storage. The combination of organic and inorganic components in hybrid materials improves their chemico-physical properties guaranteeing multi-functionality and versatility. In this context, the present contribution reports on two novel and easy routes for the synthesis of these materials. The first route is based on the covalent anchoring on nanostructured oxides (such as ZnO and the multiferroic $BiFeO_3$) of molecular monolayers acting as versatile functional surfaces. The second route involves the direct growth of Metal Organic Framework (MOF) layers on iron-based oxides for the development of innovative systems such as batteries and sensors. In particular, for this second route, we chose the Fe-based MIL-101 and MIL-88 structures made up of tri-nuclear iron terephthalate complexes as well as Zeolitic Imidazolate Framework (ZIF)–based structures which are constituted by metal centers (such as Zn or Co) and 2-methylimidazole as their organic linker. Multifunctional properties (such as gas adsorption and electrochemical and multiferroic features) of the proposed materials were also investigated.

2:30 PM

(ICACC-S17-019-2020) Multifunctional Nanomaterials for Structural, Energy and Water Related Applications

D. Kisailus*[1]

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There is a significant need for new materials to overcome environmental challenges. Nanostructures consisting of metal oxides offer potential solutions to many of these problems. However, their poor electrical and ionic conductivity still greatly hinder their practical implementation into energy storage and conversion (e.g., fuel cells, Li-ion batteries) or water purification applications. Carbon-based or modified nanomaterials have proven to enhance the performance and reduce the cost of many of these applications. Here, based on our investigation of how biomineralizing organisms assemble and mineralize, we have developed a general approach for the production of one-dimensional (1D) conductive and porous graphitic carbon fibers embedded with metal/metal oxide nanostructures. We interpret the mechanisms of nanoparticle and graphitic formation using a combination of microscopy and spectroscopy in order to exert control over the resulting nanostructures. By modifying synthesis conditions, we have produced self-supporting porous, high surface area carbon nanofiber-metal/metal oxide nanoparticle based membranes with controlled crystallite size, phase, and porosity. In addition, we describe recent developments in structural, photocatalytic and energy based performance of these and other metal oxide/nitride based nanostructures.

Multifunctional Materials I

Room: Tomoka C

Session Chair: Fiorenzo Vetrone, Institut National de la Recherche Scientifique, Université du Québec

3:20 PM

(ICACC-S17-020-2020) Lanthanide-based Materials and Molecules for Next-Generation Optical Probes (Invited)

E. Hemmer*[1]

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Based on their outstanding optical and magnetic properties, lanthanide-based compounds have been suggested for a wide range of applications including the fields of biomedicine, optoelectronics, and solar energy conversion. For instance, lanthanide-based materials are capable to emit visible and near-infrared (NIR) light under NIR excitation, which is sought after when aiming for biomedical or photonic applications. Lanthanide-based fluorides ($MLnF_4$, M = alkali metal, Ln = lanthanides and Yttrium) are potent materials to foster upconversion and NIR emission; yet, challenges in their synthesis remain. Moreover, the establishment of structure-property relationships is crucial for the understanding of fundamental physico-chemical processes in such materials and is needed for the

design of next-generation bioprobes and energy converters. Besides, lanthanide-based molecules providing a suitable ligand scaffold to the lanthanide ions are known as bright emitters. This makes them strong candidates for amalgamation with lanthanide-based nanomaterials into advanced multi-wavelength sensitive hybrid systems or as luminescent nanothermometers. This presentation will shine a light on the versatile landscape of lanthanide-based materials and molecules focusing on materials synthesis using a microwave-assisted strategy and lanthanide-specific optical features.

3:50 PM

(ICACC-S17-021-2020) Additive manufacturing using hybrid materials to create microdevices for drug delivery applications (Invited)

R. Narayan*[1]

1. NC State University, USA

Additive manufacturing approaches may be used to develop complex tools for medical treatment. We are using two photon polymerization to create microneedle arrays for transdermal drug delivery, tissue engineering scaffolds, and other medical devices. Unlike conventional lithography-based fabrication methods, two photon polymerization is an additive manufacturing approach that involves spatial and temporal overlap of photons to initiate chemical reactions that lead to photopolymerization in a localized volume. A medical device with an arbitrary geometry is created by polymerizing the photosensitive material along the laser trace, which is translated in three dimensions using a micropositioning instrument. Several classes of inexpensive inorganic-organic hybrid materials, polymers, and other photosensitive materials have been processed using two photon polymerization. Two photon polymerization can be set up in a conventional environment; no cleanroom or other specialized facilities are required. In comparison with conventional multiple-step fabrication processes, two photon polymerization is a rapid and straightforward process. In this presentation, two photon polymerization of photosensitive materials, materials characterization, in vitro testing, and medically-relevant characterization of two photon polymerization-fabricated medical devices for drug delivery will be presented.

4:20 PM

(ICACC-S17-022-2020) "Multichannel" Water Remediation by Transition Metal Oxides (Invited)

M. Epifani*[1]

1. CNR-IMM, Italy

Transition metal oxides are emerging as promising materials for water remediation. Ferrates are known for the adsorption of organic contaminants and other toxic species, Fe(II) or Fe(III) species are active in Fenton processes. Mn(III/IV) oxides are another well known system for the abatement of contaminants. It is then worth exploring this field of materials for finding suitable compositions for water remediation. In this work unexpected adsorption and photodegradation properties from MoO_2 and V_2O_5 prepared by solvothermal synthesis will be reviewed. The contaminant removal (methylene blue, MB, as sample dye) by powerful adsorption in the dark, possibly followed by photodegradation step, gives rise to a "multichannel" action by the two oxides. In the case of MoO_2, it was found that 40% more MB than benchmark TiO_2 was adsorbed. Switching on the solar simulator, with an irradiance of 12 mW/cm^2, resulted in total removal of MB after total exposure time of 240 min. Hence the photodegradation took place under visible light. The starting MB concentration was 1.5×10^{-4} M. A 1.5×10^{-5} M MB concentration was used in the tests with V_2O_5, which was present as few layers deposited onto TiO_2 and SnO_2 nanocrystals. After 15 min only in the dark, 80% of the initial MB concentration had been depleted from the solution. These results stimulated further study of transition metal oxides for water remediation applications.

4:50 PM

(ICACC-S17-023-2020) Carbon Dots - Towards Multifunctional Materials (Invited)

R. Naccache*[1]

1. Concordia University, Chemistry and Biochemistry, Canada

Carbon dots have garnered significant interest as fluorescent materials with a vast potential in sensing and imaging applications, in optoelectronics, as well as energy conversion. Their ultra-compact size, low cytotoxicity, low photo-bleaching/blinking, tunable photoluminescence, combined with simple, environmentally-friendly and low-cost synthesis, makes them ideal candidates for study. We synthesize carbon dots via bottom-up synthesis methods, with simple organic precursors (e.g. citric acid) as the carbon source. We passivate the surface of our carbon dots to achieve high fluorescence quantum yields. Moreover, our work focuses on trying to elucidate the fluorescence mechanisms in carbon dots, which remain a subject of debate. Finally, we exploit their optical properties in order to design multifunctional materials as chiral, pH or temperature sensors, as well as fluorescent probes that are geared towards heavy metal detection in solution.

Poster Session A

Room: Ocean Center Arena

5:00 PM

(ICACC-S2-P001-2020) Particle Atomic Layer Deposition of Yttrium Oxide for Hydrolysis Protection and Sintering of Aluminum Nitride

R. J. O'Toole*[1]; C. Hill[1]; P. Buur[1]; C. Bartel[1]; C. J. Gump[2]; C. Musgrave[1]; A. W. Weimer[1]

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2. ALD NanoSolutions, Inc., USA

Aluminum nitride (AlN) has a high thermal conductivity, making it an attractive material for high power LED heat sinks. AlN particles hydrolyze in the presence of water to form impurities that reduce its thermal conductivity, so they must be processed in organic solvents which increases cost. Typically, AlN parts are densified by adding a sintering aid to enable liquid phase sintering and scavenge impurities to enhance thermal conductivity. Sintering aids are usually added by mechanical mixing, where primary AlN particles are mixed with sintering aid particles which can lead to microstructural inhomogeneities. In this work, particle atomic layer deposition (ALD) was used to conformally coat AlN particles with a thin film of yttrium oxide (Y_2O_3) to protect the AlN particles from hydrolysis and to enable liquid phase sintering. Investigation of the hydrolysis behavior showed that the Y_2O_3 film hindered hydrolysis of the AlN particles. The addition of Y_2O_3 by particle ALD increased the final relative density by ~30% relative to uncoated AlN after sintering at 1800°C for 50 min. Scanning electron microscopy was used to determine the microstructural homogeneity of coated and uncoated samples, and the densification behavior and microstructure of AlN parts with Y_2O_3 added by mechanical mixing were also analyzed to determine the importance initial sintering aid location.

(ICACC-S2-P003-2020) Morpho-structural evaluation on 4÷12 mol% Y_2O_3 doped Zirconia ceramics and their composites with Al_2O_3 prepared via classic and Spark Plasma Sintering

O. R. Vasile*[1]; A. V. Surdu[1]; A. C. Birca[1]; R. Trusca[1]; M. I. Vasile[2]; E. Tanasa[1]; B. S. Vasile[2]

1. University Politehnica from Bucharest, Faculty of Applied Chemistry and Materials Science, National Research Center for Micro and Nanomaterials, Romania
2. University Politehnica from Bucharest, Romania

For many years now zirconia presents a great interest in various areas, starting from biomedical ones like application as an implant, electrolyte for solid oxide fuel cells, aerospace technologies as thermal barrier coatings, etc. The present work is focused in the evaluation of the morphology and structure of yttria doped zirconia ceramics and their composites with alumina. The amount of yttria used in in the range of 4 mol. % to 12 mol. % and the alumina used for composite is 5 wt.%. The sintering procedure used was classic sintering at 1600 C for 3 hours and Spark Plasma Sintering (SPS) at temperature of densification from 1250 C to 1300 C for 5 minutes. The starting powders were synthetized via sol-gel and polyol route. All the obtained ceramics were characterized via high resolution SEM coupled with EDX analysis, XRD followed by Riedveld refinement and RAMAN Spectroscopy. The morphology of the samples has drastic changes from 15-20 μm grains, polyhedral for classic sintering samples to 80-90 nm grains almost spherical for SPS sintered ceramics. The XRD analysis reveals that the phases for the same ceramics but processed via polyol or sol-gel method leads to the formation of different phases and percentage of yttria stabilised zirconia.

(ICACC-S2-P004-2020) Effectiveness of the process of modification of SiOC-based materials with phosphate ions assisted with co-dopants

M. Gaweda*[1]; P. Jelen[1]; M. T. Sitarz[1]

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

SiOC (silicon oxycarbide or black glasses) based materials might be obtained by the ceramization of the polymeric precursors such as polisesquioxanes. It gives a wide range of possible forms of the prepared materials: coatings, films, bulk, foams, etc. Moreover, if the precursors are self-synthesized e.g. via the sol-gel method, the amount of the introduced carbon ions bonded into the structure as well as of the free carbon phase might be controlled. It also enables modification with another cations and anions to alter the properties of the final material. Modifications with aluminium, magnesium, boron, calcium or nitrogen ions are well described in the literature. Here, as the dopant, phosphate ions were chosen. While organophosphorus compounds are poorly reactive, co-modification with aluminium and boron ions was proposed. The aim of this exertion was to crate stable $AlPO_4$ and BPO_4 units able to incorporate in the silica matrix. Spectroscopic studies (FTIR, Raman, XPS, EDS) enable to answer how the dopants are implemented into the polymeric precursors as well as into the glass network and if the desired structural units were obtained. Also, the effectiveness of the modification process depending on the type of the organophosphorus compound and the presence of the co-modifier was evaluated.

(ICACC-S4-P006-2020) Ballistic Performance of Yttrium Aluminum Garnet (YAG) transparent ceramic armor against 12.7mm Armor Piercing Incendiary

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This paper focuses on the anti-penetration performance of Yttrium Aluminum Garnet (YAG) transparent ceramic armor. The experiments were conducted using 12.7mm Armor Piercing Incendiary at a striking velocity of 488 m/s. The transparent ceramic armor consists of a hard-transparent ceramic layer with glass interlayers, polyurethane bonding and polycarbonate backplane. The deformation process of the transparent armors and the projectile were recorded by high-speed photographs and flash-radiographs. The energy absorptions of transparent armors were analyzed by the deflection of backplane. The penetration resistances were analyzed by the different layer designs. A numerical simulation has been built and computed by AUTODYN, whose results mainly meet the experimental results. The protective performance of striking, interlayer, bonding and backplane materials were analyzed respectively. Further analysis on the anti-penetration performance is given in terms of the residual mass of bullet core. The results show that the thickness of transparent ceramic has a great effect on the crushing of the projectile. Moreover, the anti-penetration ability of the transparent ceramic armor can be changed by altering the laminated structure of polyurethane-polycarbonate backplane. This conclusion provides a strong reference for transparent ceramic armor design.

(ICACC-S4-P007-2020) Densification of Boron Suboxide (B_6O) by Spark Plasma Sintering

M. A. Arroyo-Green*[1]; R. A. Riera[1]; S. Bavdekar[1]; G. Subhash[1]

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

Boron suboxide (B_6O) is a promising new material for armor applications as it possesses a higher hardness and compressive strength than boron carbide (B_4C) with a comparable density (2.54 g/cm^3). However, current attempts to fabricate this material have not resulted in a fully dense material. In this study, Spark Plasma Sintering (SPS) is utilized to optimize the processing parameters with an aim to achieve a density greater than 98% of its theoretical value. The effects of various sintering parameters – ramp rate, maximum temperature, hold time, pressure, and volume fraction of sintering aid (Al_2O_3, Y_2O_3, etc.) – on the resulting density of B_6O were investigated using a design of experiments (DOE) approach. Ultrasonic and quasistatic microVickers indentation experiments were performed on the fabricated samples to obtain their elastic moduli and hardness.

(ICACC-S4-P008-2020) Alternative Method for Preparing Silicon Doped Boron Carbide

K. Christian*[1]; C. Hwang[1]; Q. Yang[1]; R. A. Haber[1]

1. Rutgers University, Dept. of Materials Science and Engineering, USA

Silicon doping is often used to reduce stress-induced amorphization in boron carbide. Up to date, reaction hot pressing has been used to make silicon doped boron carbide. However, both liquid phase sintering and a long dwell time before reaching homogeneity result in large grain sizes, which degrades the mechanical properties. To combat these problems, a three-step approach was devised. Commercial silicon, amorphous boron, and boron carbide powders were melted into ingots using an arc melter, crushed down into controlled particle sizes through a milling process, and then sintered. Because the powder was pre-reacted and the particle size is finer, the diffusion distance was reduced, leading to a faster sintering time. This work was carried out to validate the feasibility of the three-step process as an alternative method to producing silicon doped boron carbide and to demonstrate the ability to control chemistry and microstructure in the bulk by adjusting the melt composition and sintering protocols.

(ICACC-S4-P009-2020) Processing and Characterizing Al-doped Boron Carbide Bulk Ceramic

Q. Yang*[1]; E. Gronske[1]; C. Hwang[1]; R. A. Haber[1]

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Boron carbide ceramics undergo stress-induced amorphization during high-pressure events which leads to the loss of shear strength and fragmentation. Experimentally, microalloying boron carbide with foreign atoms such as Al, Si, and B has been speculated to

substantiate the suppression of amorphization. Recently, Si-doped and B-doped boron carbide bulk ceramics have been fabricated and have shown the propensity to suppress amorphization. However, Al-doped boron carbide bulk ceramic has never been produced and its ability to reduce amorphization has not been verified. For the first time, we fabricated Al-doped boron carbide bulk ceramics. Their mechanical properties and the ability to suppress stress-induced amorphization were elucidated.

(ICACC-S4-P010-2020) Studying Amorphization in Rhombohedral Boron-Based Materials

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Amorphization in boron carbide (B4C) and boron-rich boron carbide (B-rich BC) have been previously studied in a Diamond Anvil Cell (DAC). The threshold pressure to induce amorphization in these samples have been determined, and the extent of amorphization has been quantified. Now, silicon-doped boron carbide (SI-BC) and boron suboxide (B6O) will be studied in a DAC to observe, evaluate, and quantify any amorphous-like behavior present in these materials. It is believed that these materials will amorphize in manner similar to that of boron carbide, so these experiments will give further insight into that phenomena. This study will show how the new chain structure in Si-BC mitigates amorphization, and B6O will give insight into a rhombohedral boride material amorphizing without having a chain structure, such as boron carbide has. Experiments will be self-consistent with previous B4C and B-rich BC DAC experiments, which will be reviewed, and were done in-situ in a Renishaw Raman Microspectroscopy system, using a 532 nm laser source.

(ICACC-S4-P011-2020) On the Densification of Ceramics with Bi-Modal Size Hard Inclusions

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1. CCDC Army Research Laboratory, USA

During the sintering of a ceramic particulate body containing randomly distributed hard inclusions, densification can become severely constrained when the volume fraction of inclusions exceeds a critical or threshold value. Above this value, the inclusions form a "rigid" percolated network or cluster whose size spans the dimensions of the densifying body resulting in arrested densification. In this study, aspects of percolation theory are applied to developing models for the densification of a ceramic particulate body containing bi-modal size hard spherical inclusions. In particular, efforts are focused on obtaining estimates for the critical volume fraction of hard inclusions corresponding to the "rigidity" percolation threshold as a function of initial density of the ceramic particulate body, as well as the size and volume ratios of the hard spheres. Both deterministic and probabilistic models were developed based on simple geometric ideas and the LAMMPS GRANULAR package, respectively. As expected, the models predict the percolation threshold for the bi-modal size inclusions to be higher than mono-size inclusions which is agreement with experimental results. Details of the models and further results are discussed.

(ICACC-S4-P012-2020) Effects of Slurry Composition on Silicon Carbide Spray Dried Granules

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2. US Army Research Laboratory, SEAP/CQL Program, USA

The effects of slurry composition and spray drying parameters on the morphology and structure of silicon carbide granules were examined in this study in an effort to expand the design space for particulate-toughened ceramic microstructures. Aqueous-based slurry compositions were used to determine the effect of different organic additives on granule morphology. Four different aqueous suspensions with 40 wt% silicon carbide and 3 wt% binder were prepared. The binders included polyethylene glycol (PEG), polyethylenimine (PEI), poly(acrylic) acid (PAA), and polyvinyl alcohol (PVA). Spray-drying parameters for the BUCHI B290 correspond to a drying temperature of 175 °C, gas flow rate of 473 L/min, slurry feed rate of 5 mL/min, and a nozzle opening of 1.4 μm. The spray-dried granules were examined using a Phenom XL scanning electron microscope. Particle size analysis was performed on each sample using Image J. The average granule size fell within a tight range (8 to 15 μm) for all slurry compositions. In terms of desired granule morphology, PEI and PAA yielded less favorable results than PEG and PVA. Experimental procedures and results will be discussed further.

(ICACC-S4-P013-2020) Quantitative Characterization of Arc-Melted Si-doped Boron-Rich Ceramics

H. Wolf*[1]; Q. Yang[2]; K. D. Behler[3]; J. LaSalvia[4]; R. A. Haber[2]; M. Harmer[1]; C. Marvel[5]

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A combination of arc-melting, attrition milling, and conventional sintering is currently being pursued to manufacture dense, B-rich Si-doped boron carbide for commercial scale armor ceramics. However, the fundamental thermodynamics and microstructures of arc-melted Si-doped B-rich boron carbides are mostly unknown. This work aimed to synthesize and accurately analyze a variety of arc-melted bulk samples to connect processing to microstructure and assess the potential of stable silicon-doped boron carbide. In total four bulk specimens were fabricated (~$B_{4.3}C$, ~$B_{6.5}C$, ~$B_{6.5}C$ w/ 2.5 at% Si, and ~$B_{6.5}C$ w/ 4 at%), encased in commercial-grade boron carbide to minimize Si evaporation, and annealed to evaluate homogenization thermodynamics and kinetics. A new z-factor calibrated SEM-EDS analysis was also conducted to measure the bulk compositions, B/C ratio, and Si concentration profiles. z-factor microanalysis, traditionally a transmission electron microscopy technique, was used to calibrate the new SEM-EDS analysis. Overall, it was observed that the Si-doped as-melted specimens contained a variety of Si-rich second phases and that boron doping reduced any microstructural evolution kinetics. Experiments in progress will determine the optimal annealing temperatures and times to produce the best Si-doped boron carbide armor ceramics.

(ICACC-S4-P014-2020) Growth of high purity zone-refined boron carbide single crystals by laser diode floating zone method

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2. Johns Hopkins University, Department of Mechanical Engineering, USA
3. Johns Hopkins University, Department of Chemistry, USA
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5. Morgan State University, Department of Electrical and Computer Engineering, USA

We report on the growth and characterization of 4mm diameter x50mm long boron carbide (B_4C) single crystals using a laser diode floating zone (LD-FZ) method. Electron micro-structure analysis shows the presence of a significant number of twinning-boundaries along the growth direction ($<001>_h$) oriented in the $<1210>_h$ plane. Faster growth rates tended to reduce the number of twins seen. Zone refinement of these crystals led to a significant reduction of trace impurities at the expense of increased twinning. Powder x-ray diffraction confirms that the bulk is rhombohedral B_4C, as shown by the micro-structure. Berkovich nano-indentation of the key $<001>_h$ plane showed 41 ± 1 GPa hardness, with a Young's modulus of 520 ± 14 GPa, like the other planes measured in the literature.

(ICACC-S4-P130-2020) Thermodynamically consistent modeling of ceramic fracture with piezoelectric effects

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Brittle fracturing is the characteristic feature of ceramic armor. Different widely known approaches have been suggested for modeling this feature. One of the most intriguing phenomenon in brittle fracture is the loss of axial symmetry of the damage pattern; this pattern accompanies penetration of an axisymmetric projectile through isotropic initially uniform brittle plate. This loss of symmetry can be explained and modelled on the basis of the theory presented in and references therein. The theory was coined as the Phenomenological Mechanochemistry of Damage (PMD) and was implemented in the SIERRA software. In several applications of ceramic armor the piezoelectric forces can reach can reach high magnitude. In this talk, we demonstrate how to modify the master system of PMD taking such effects into account. Our presentation is based on the recent report.

(ICACC-S5-P015-2020) Investigation of substituted Bioactive glass/Hydroxyapatite Composite system on mechanical and biological performance

S. Yadav*[1]

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Zirconia substituted 1393 type bioactive glass and hydroxyapatite were synthesized by sol-gel and co-precipitation method respectively. Composites were prepared with different ratio of hydroxyapatite from 20wt% to 80wt% in substituted bioglass system and sintered at two different temperatures (600°C and 1000°C) to study the effect on phase, bioactivity, cell proliferation and mechanical strength. Increase in sintering temperature change the amorphous nature of bioglass into crystalline which confirmed by XRD. In terms of bioactivity, composites show dissolution and apatite formation behavior during immersion in SBF. Increase in density and compressive strength was observed with increasing sintering temperature due to incorporation of glass structure with hydroxyapatite. Cell proliferation was measured by MTT assay over MG63 cell lines and results indicate that composites sintered at 1000°C have better cell growth rate than composites sintered at 600°C. Results indicate that increasing in sintering temperature provides more crystallinity which improves the density, compressive strength and cell proliferation. Therefore, this study demonstrated that sintering in composite affect bioactivity, cell growth and mechanical properties which makes it suitable biomaterials for biomedical application.

(ICACC-S5-P016-2020) Characterization and analysis of poly (vinyl alcohol) / attapulgite composite hydrogels in drug delivery systems

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2. Federal Institute of Rio Grande do Norte, Brazil
3. Federal University of Rio Grande do Norte, Physics, Brazil

The development of new, more efficient and cheaper materials has been the focus of several studies. Drug containing hydrogels may be potential materials applied to controlled drug release. Controlled-release films that exhibited near-optimal behavior would be placed in the tissue and, upon swelling, the drug would be released within the therapeutic range during the treatment time. In this work composite hydrogels in the form of polyvinyl alcohol (PVA) film were developed using treated attapulgite as a crosslinking agent. The composite hydrogels were obtained in solution by varying the amount of attapulgite (2% -5%) in the polymeric matrix to study its influence on crystallinity, swelling properties, and drug release. The prepared materials were characterized by DRX, FRX, BET, TG, swelling degree and mechanical resistance. The results show that the attapulgite/drug composite films have better properties among the studied films, such as high mechanical resistance and adequate swelling degree. Keywords: hydrogel, attapulgite, composite hydrogels, release systems.

(ICACC-S7-P017-2020) Efficient oxygen reduction electrodes with carbon nanomaterials for proton exchange membrane fuel cell

D. Lee*[1]; H. Kim[1]

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Proton exchange membrane fuel cells (PEMFCs) have attracted considerable attention for power sources of zero emission vehicles as they are compact, lightweight, clean, and have a high power density. The oxygen reduction reaction (ORR) at the cathode of a PEMFC plays a key role in determining the performance of a fuel cell, and durable ORR catalysts able to endure over 5,000 hours of operation are essential for the commercial adoption of fuel cells. This study developed electrochemically efficient and durable oxygen reduction reaction (ORR) catalysts for proton exchange membrane fuel cells (PEMFCs). Pt catalysts were directly synthesized on graphene oxide (GO) and reduced graphene oxide (rGO), and Pt/GO-r catalysts was prepared by heat treatment of Pt/GO catalysts. The characteristics of the synthesized catalysts were analyzed with transmission electron microscopy, X-ray diffraction, thermogravimetric analysis, X-ray photoelectron spectrometer, cyclic voltammetry and Raman spectroscopy analysis. The results confirmed that the reduced graphene oxide was superior comparing with the CB support in oxygen reduction activity and long-term durability, and the concentration of oxygen in the graphene support is a key factor to enhance the oxygen reduction properties and long-term durability of Pt/rGO catalyst.

(ICACC-S7-P018-2020) STEM observations of domain structures related piezoelectric properties in PZT thin films

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2. Kobe University, Mechanical Engineering, Japan

$Pb(Zr,Ti)O_3$ (PZT), which has excellent piezoelectric properties, is expected as MEMS applications such as sensors, actuators and energy harvesters. PZT exhibits high dielectric and piezoelectric properties in the composition around the morphotropic phase boundary (MPB) which is at the interface between tetragonal(P4mm) and rhombohedral (R3m) phases. Although it is known that PZT thin films have different piezoelectric properties from bulk samples, it is important to study the relationship between crystal structure and piezoelectric properties even in thin films for the development of piezoelectric application. In order to further improve piezoelectric properties, we fabricated the PZT thin film using rf-magnetron sputtering, and by performing in-situ X-ray diffraction (XRD) measurement using synchrotron radiation XRD, we have systematically investigated the correlation between crystal structure and piezoelectric properties. However, the microstructure and domain structure of the PZT thin film have not been sufficiently clarified. Therefore, we fabricated a thin sample for cross-sectional observation from the PZT thin film by Focused Ion-Beam, and carried out the microstructure observation by Scanning Transmission Electron Microscopy (STEM). In the presentation, we report the domain structure and the electric field responsiveness of the PZT thin films.

(ICACC-S7-P019-2020) Development of oxygen evolution catalysts based on Ni,Fe,Co oxyhydroxides by electrodeposition

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Electrolysis of water is a simple and effective method to produce hydrogen and oxygen. When coupled with renewable energy sources, it has great potential for clean production of hydrogen. In the overall reaction, the hydrogen evolution reaction is a relatively fast reaction, with the overall limiting reaction being the oxygen evolution reaction, therefore appropriate catalysts for the oxygen evolution are intensively sought. In this work, we have prepared catalysts on nickel foil and foam substrates by a versatile electrodeposition method employing dissolved metal salts containing Ni, Fe and Co. Electrocatalysts were screened for their electrochemical activity and were characterized microstructurally. Results show that the electrodeposition offers the potential to produce materials with desired stoichiometry and high electrocatalytic activity (with an overpotential of ~200 mV at 10 mA cm-2, in aqueous 1M KOH) with good stability.

(ICACC-S7-P020-2020) Heterometallic Alkoxides as Single Source Precursors for the Generation of $InFeO_3$

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Due to the great interest of ternary oxides regarding their catalytic, magnetic and electro optical properties, the focus is on the generation of those materials. Previously, the formation of ternary indium ferrites was achieved using multi source precursors, which often suffer from the formation of side products, such as iron and indium oxide. In contrast, metal alkoxides as single source precursors provide the accessibility of phase pure ceramics, since they carry the required atomistic ratio for materials formation inside their molecular structure. Consequently, the generation of metastable phases is controlled by the preformed metal oxygen bonds, ligand design, metal ratio and oxidation states. In this regard, we report the synthesis of heterometallic indium iron alkoxides with various indium iron ratios. The structural constitutions were proofed by X-ray investigations and thermal gravimetrical measurements were performed. In addition to that, $InFeO_3$ was generated by thermal decomposition of the precursors.

(ICACC-S7-P021-2020) Electrospinning of 1D nanofibers of Potassium Sodium Niobate for energy harvesting

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Electrospinning is a simple, scalable and most importantly, a process deriving minimal energy input. It offers capabilities for making fibers from polymer solutions and melts, with diameters ranging from the nano to micro-scale. The fibres can be directly electrospun on desired substrates or can be peeled off from the collector plate to obtain self-supported nanomeshes. Nano- and microfibers have found applications in fields such as energy harvesting, filtration, optical and chemical sensors, electrode materials and biological scaffolds. The material in its nanofiber morphology presents inherent benefits like high surface to volume ratio, high pore volume, and high mechanical flexibility. These benefits greatly enhance the potential of the nanofibers as an effective energy harvester from sources of vibration. In this work, we present energy harvesting by electrospun nanofibers of Potassium Sodium Niobate (KNN) for low power device applications, specifically for wireless sensor networks in cars.

(ICACC-S7-P022-2020) Tailored Magnetic Nanocomposites with Antibacterial and Adsorption Activity for the Remediation of Drinking Water

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The remediation of contaminated water is one of the major challenges today. Different micropollutants present in drinking water from many sources such as agriculture as well as industrial and municipal waste water may cause harm to the human body. Therefore, we present cost-effective, polyol process-synthesized, highly magnetic nanoparticles (MNPs) subsequently coated with a polymer to provide carboxy functions. In order to enhance the density of COOH groups for the adsorption of the heavy metal cation Ni^{2+} and the organic model pollutant methylene blue (MB) L-cysteine (L-cys) was attached via carbodiimide coupling. With the help of thiol-ene click chemistry, free thiol groups of L-cys were coupled to an antibacterial imidazole derivative. The tailored MNPs were micrographically characterized. Subsequently, photometric adsorption measurements for MB and Ni^{2+} as well as antibacterial testings against gram-negative (E. coli) and gram-positive (B. subtilis) bacteria were performed. After the adsorption measurements the MNPs were collected by an external magnetic field, cleared from the adsorbate and reused again. Furthermore, these smart nanocomposites can be extended for the adsorption of other heavy metal cations and organic pollutants.

(ICACC-S7-P023-2020) Production of Ni/ $Ce_{1-x}Zr_xO_{2-\delta}$ doped with Nb, Ti materials in supercritical alcohol media for catalytic syngas production

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Dry reforming of methane or ethanol is a perspective way to transform two greenhouse gases as well as a model reactions for biofuels transformations. Ni-containing catalysts are attractive in these reactions due to their high activity and low price. Mixed $Ce_{1-x}Zr_xO_{2-\delta}$ oxides are promising catalysts supports due to their high oxygen mobility and reactivity. In our work $Ce_{1-x}Zr_xO_{2-\delta}$ doped with Nb, Ti were synthesized using supercritical fluid technology. The influence of the contact time, the type of supercritical fluid, the temperature and pressure on the degree of crystallization, the size of the crystallites, the specific surface and the morphology of the particles were investigated. The supporting of the active metal - nickel (2 to 10 wt.%) onto the oxides was done in two ways: the deposition of nickel in a onepot supercritical synthesis in parallel with the synthesis of a mixed oxide and insipient wetness impregnation of oxide support with nickel nitrate solution. The obtained catalysts were characterized by physical-chemical methods and tested in dry reforming of methane and ethanol reactions. The work was supported by the Russian Science Foundation, Project 18-73-10167.

(ICACC-S7-P024-2020) Quantification method for modified nanoparticles using the ESA effect

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Surface functionalization of nanoparticles became a promising field for various applications methods e.g. in cancer treatments for the usage of mesoporous silica (SiO_2) nanoparticles or as coating material with self- cleaning properties. In this regard, the quantification of modified nanoparticles with different functional groups or ligands is crucial for possible medical applications and for the control over surface properties and chemical structure. In this work, a novel quantification method of freely accessible hydroxide

groups located on the particle surface of SiO_2, hematite (α-Fe_2O_3) and spinel ($MgAl_2O_4$) nanoparticles, utilizing the electronic sonic amplitude (ESA) effect supported by simultaneous conductivity measurements, is presented. As a result, the zeta potential of charged particles located in an alternating electric field is calculated from the resulting sound wave, opening insights into the surface chemistry of nanoparticles.

(ICACC-S8-P025-2020) Effects of SiC Particle Size and Concentration on the Thermal Properties of Reaction Bonded SiC Composites

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Reaction bonded silicon carbide (RBSC) composites are fabricated by the reactive infiltration of molten Si into preforms of SiC plus carbon, flexibly designed for different applications. RBSC materials have favorable properties including high hardness, high thermal conductivity, low thermal expansion and high stiffness for many applications in the wear, nuclear, armor, LCD glass, semiconductor capital equipment, chip manufacturing, solar, oil and gas and refractory industries. This study attempts to further optimize or tailor the thermal properties of RBSC, by controlling the SiC particle size and the final concentration of the SiC content. SiC powders with different sizes (12 μm, 30 μm, 50 μm and 200 μm) were selected as the starting raw materials. The final SiC concentrations of the samples are controlled by the amount of initial carbon before molten Si infiltration. This study correlates the starting SiC powder size and final SiC concentration with the microstructure and thermal properties (CTE and thermal conductivity) of the RBSC composites.

(ICACC-S8-P026-2020) Comparative investigation of grinding characteristics between single system and binary systems in uniaxial compression tests

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Grinding is a key process which determines the quality of final products in mining and ceramic industries. To achieve high efficient grinding process, it is necessary to control grinding process based on grinding characteristics of raw materials. Grinding mechanism of a single component has been well investigated, while grinding mechanism of multiple components has hardly investigated. Therefore, this study evaluated the grinding characteristics of binary components using experimental and numerical approaches. In this research, silica sand and limestone were used because these materials had different physical properties. To investigate grinding characteristics of binary components, uniaxial compression tests of a single component and binary components were conducted. Focusing on the particle size distribution (PSD), experimental results showed that grindability of binary components was significantly different from that of a single component, especially when a ratio of the mixture of silica sand and limestone varied. To evaluate different grinding characteristics, the discrete element simulation with the breakage model was also performed. Time variation of PSD during uniaxial compaction was calculated. From the above, the grinding characteristics of binary components were evaluated by experiments and simulations.

(ICACC-S8-P027-2020) Densification of Samaria-Doped Ceria by AC Electric Field-Assisted Pressureless Sintering

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Ceria-based solid solutions have been considered as promising electrolytes for intermediate temperature (600–800°C) solid oxide fuel cells (IT-SOFCs) due to their excellent oxygen-ion conductivity compared to yttria stabilized zirconia (YSZ). One of the main concerns on these solid electrolytes is related to their relatively low sinterability. High sintering temperatures, mostly above 1500 °C, are required to attain good densification. At high temperatures a reduction of Ce^{4+} to Ce^{3+} can occur, with consequent increase in the electronic conductivity. However, the use of unconventional sintering methods such as electric field-assisted (flash) sintering is one of the strategies for lowering the sintering temperature and optimizing properties. In this study CeO_2:20 mol% Sm_2O_3 green ceramic pellets were sintered conventionally at 1500°C/2 h and flash sintered at 800°C, 1000°C and 1200°C by applying a 200 $V.cm^{-1}$ electric field. The main results show that the final shrinkage level is nearly independent of the temperature when the electric field is applied and slightly better than that of the 1500°C sintered pellet, and the bulk conductivity of the sample flash sintered at 1200°C is similar to that of the sample sintered at 1500°C. The availability of a pathway for the electric current pulse derived from the applied electric field is proposed as the reason for the achieved shrinkages.

(ICACC-S8-P028-2020) Boron and ß-Silicon carbide powders: Specialized development for advanced applications

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A unique combination of low specific gravity and high hardness makes B_4C attractive for personal armor. However, since difficult to form and to sinter, hot pressing technology was dominating, but with limited freedom for geometries. New requirements and increasing demand for complex shapes became a challenge and led to more intensive development for alternatives, like pressureless sintering offering better shaping possibilities. Thus, finer starting materials are required; particularly higher sintering activity is now a key property. Next generation turbines are based on CMCs operating at higher temperatures and contribute less weight than nickel-base superalloys. CMCs of SiC/SiC can take the heat and cut component weight by half. Its production process requires SiC powders, preferably of ß-SiC type. Today's powder manufacturers face a multitude of challenges to satisfy their customers. Höganäs, a renowned producer of high class non-oxide ceramic powders, has taken that opportunity introducing a set of micron and sub-micron class B_4C and ß-SiC powders. The grades vary by fineness and purity due to a combination of various milling and purification techniques available through a 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 those powders.

(ICACC-S8-P029-2020) Refractory Metal (RM) – Wrap: A pressure-less joining technique for ceramics

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A new process called "RM-Wrap" (RM= Refractory Metals, i.e. Mo, Nb, Ta, Zr) has been developed as a pressure-less and tailorable technique to join ceramics such as SiC, alumina and mullite ($3Al_2O_3.2SiO_2$). In the RM-Wrap joining technique the refractory metal foil is used as a wrap containing one or more silicon foils; optimized joining treatment consisted of heating to 1450 °C with heating rate of 1000 °C / hour followed by a dwell time of 5 minutes in Argon flow. The resulting joining materials are in-situ formed composites made of refractory metal disilicide particles (MoSi2, NbSi2, TaSi2 or ZrSi2) embedded in a silicon rich matrix. The joint morphology and elemental composition of the joining material have been investigated in detail using XRD, FESEM and EDS: the joints are uniform, continuous and crack free. The joint strength of RM-Wrap joined

SiC was measured at room temperature using three different mechanical tests: (i) single lap (SL), (ii) single lap off-set (SLO) and (iii) torsion on hourglass shaped samples (THG). Furthermore, oxidation tests were carried out on SiC joints at 1100 °C, 6 hours in air; after the tests, the joint morphology showed no changes and the samples remained firmly joined.

(ICACC-S8-P030-2020) Energy – fractal nature and thermo dynamic in $BaTiO_3$ -ceramics doped by Y additives

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The correspondence between the fractals and the Nature, is the same from thermodynamically point of view. These structures have been inspired from Nature and Euclidian geometry. Fractal material analysis and self-similarity, makes them suitable for processing in disorder to the order applications. Relation within the matter we treat as a thermodynamical system; functions as temperature, free energy, enthalpy and entropy are important to understand the processes. The great scientific interest is to control chaotical processes and structures and the chaos and order border. World's need for energy, imposed the technological challenges whole spectra. We discuss the fractals analysis in energetic questions and diverse technologies. We performed the experiments on $BaTiO_3$ synthetized with Y additives. The intergrain layers are medium where we establish the new approach for electronic properties integrations by complex fractal correction with grains and pores surfaces influences, and particles Brownian motion. We correlated fractals with temperature and opened frontiers for intergrain properties integrations.

(ICACC-S8-P032-2020) Synthesis of Cs_2SiO_3 and $Cs_2Si_4O_9$ Glasses

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Cesium released in Fukushima Daiichi severe accident is an important major that should be concerned. Recently, releasing form of Cs is supposed to react to structure materials inside the nuclear reactor, in other words, the chemical interaction between Cs with SiO_2 in either SUS or in concrete may take place. These reactions produce the cesium silicate compounds which may be migrated inside the materials in the nuclear reactor. In the literature, the phases in the Cs-Si-O systems are already calculated, but the experimental data is still not enough to validation this system, the chemical compounds between Cs and Si have still not clear yet. In this research, Cs_2SiO_3 and $Cs_2Si_4O_9$ are synthesized from Cs_2CO_3 and SiO_2 by solid-state reaction or by sol-gel method. The synthesized compounds will be analyzed by XRD including Rietveld analysis as a purity test. Both Raman and FT-IR will be applied to detect the vibrational characteristics of chemicals functional groups. The crystallographic data and diffraction patterns will be obtained by TEM. The stability of the product material to moisture is studied by TG-DTA. The phase transition of compounds will be studied at the temperature range below 1000°C.

(ICACC-S11-P033-2020) Partial amorphization and phase control of Cobalt nickel sulfide for an efficient oxygen evolution reaction

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Introduction of a thin amorphous layer on a pristine surface can boost the oxygen evolution reaction (OER) activity of transition metal sulfides (TMSs). We introduce the novel strategy to improve the OER properties by developing cobalt nickel sulfide (CoxNi1-xS2, CNS) with a high density of crystalline and amorphous phase boundaries. Electrochemical activation (ECA) can partially amorphize hollow CNS nanoparticles derived from surface-selective sulfidation. The ECA-treated CNS (ECA-CNS) electrocatalyst, which is comprised of CNS nanodots separated by thin amorphous layers, shows high densities of crystalline and amorphous phase boundaries. This catalyst shows superior OER catalytic performance with a current density of 10 mA cm-2 at a small overpotential of 290 mV, a low Tafel slope of 46 mV dec-1, a high mass activity of 217 A g-1, a high turnover frequency of 0.21 s-1 at an overpotential of 340 mV, and excellent long-term stability in alkaline media.

(ICACC-S11-P034-2020) Preparation and Properties of Natural Rubber with Organic-inorganic Nanomatrix Structure

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The outstanding mechanical properties of soft materials i.e. natural rubber are partly due to the organic–inorganic nanomatrix structure because numerous organic microparticles are dispersed in a small amount of an inorganic nanomatrix composed of inorganic nanoparticles and organic macromolecules. Here we form an organic–inorganic nanomatrix using graft-copolymerization of a vinyl monomer with an inorganic oxide precursor (vinyl triethoxy silane) onto natural rubber particles with an average diameter of 1 μm dispersed in water. The inorganic oxide precursor is converted into inorganic oxide nanoparticles through hydrolysis and condensation, forming chemical linkages between natural rubber microparticles and inorganic oxide nanoparticles. Transmission electron microscopy indicates that the organic–inorganic nanomatrix is densely filled with inorganic oxide nanoparticles and the natural rubber microparticles are dispersed in the nanomatrix. This nanomatrix composite realizes both energetic elasticity and entropic elasticity of a soft material, opening a novel field of building block chemistry with respect to a pair of organic microparticles and inorganic nanoparticles.

(ICACC-S11-P036-2020) Synthesis and thermoelectric properties of $CrSi_2$ dispersed with fine secondary phase

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$CrSi_2$ is a thermoelectric material that has high electrical conductivity and Seebeck coefficient, but it has relatively high thermal conductivity. Reduction of its thermal conductivity is necessary to improve thermoelectric performance. In this study, we tried to synthesize $CrSi_2$ with precipitated metallic secondary phase (CrSi) to reduce thermal conductivity without decrease in its electrical conductivity. Polycrystalline samples of $CrSi_{1.96}$ were prepared by sintering. The as-sintered samples were heat treated to obtain supersaturated solid solution, followed by subsequent heat treatment at lower temperature to precipitate CrSi. CrSi phase was almost disappeared and supersaturated solid solution was obtained by the first heat treatment. After precipitation process, the sample contained secondary phase which was smaller than that of the as-sintered $CrSi_{1.96}$. However, the precipitated CrSi was not small enough to

decrease thermal diffusivity. On the other hand, electrical conductivity of the CrSi precipitated $CrSi_{1.96}$ increase up to 86.2×10^3 S/m. Finally, CrSi precipitation increased Power Factor by the increase. The secondary phase, CrSi, precipitated in $CrSi_2$ matrix successfully enhanced the electrical conductivity without increase of thermal diffusivity. Further improvement of the thermoelectric performance of $CrSi_2$ is expected by controlling temperature and time of the heat treatments.

(ICACC-S11-P037-2020) Dental ridge augmentation by Ezechbone® implantation using a minimally invasive vestibular tunnel approach

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Conventional procedure of dentoalveolar bone grafting involves creating a rectangular-shaped flap through one crest incision accompanied with two vertical releasing incisions to expose the bony defect, followed by the placement of bone graft into the defect. To avoid migration of the graft, a barrier membrane or bone screws are often used to fix the implant before the primary closure with tension-free suture. Not only costly and time-consuming, the crest incision made right on top of the bony defect in this conventional procedure largely increases dehiscence and infection risks. With a different approach, the minimally invasive VTA method makes a minimal incision line on vestibule about 5 mm distal to the defect. A blunt dissection is made with curette to elevate soft tissue to form a subperiosteal tunnel under the gingiva. Ezechbone® Cement and Ezechbone® Granule are then delivered into the bone defect through the tinny tunnel using a specially-designed instrument without the need of cutting periosteum. This minimally invasive procedure would not need any membrane or bone screws and can cut short the surgery time from about 2 hours for the conventional flap procedure to 20-30 minutes which means very dear to an oral surgeon. This research was partly sponsored by Southern Taiwan Science Park Bureau Smart Biotech Medical Cluster under contract # CY-05-08-38-107.

(ICACC-S11-P038-2020) A fast-healing Ca/P/S-based bone substitute for orthopedic applications

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The implantation device used for this study is a synthetic, inorganic, highly porous, granular, 100% resorbable and fast-healing Ca/P/S-based bone-substituting material developed by a National Cheng-Kung University/Joy Medical Devices joint research project. The final product (Ezechbone® Granule) is proprietarily manufactured by JMD, an ISO 13485/GMP-certified facility in Kaohsiung, Taiwan. The safety and efficacy of the product has been confirmed by a series of chemical/physical characterization and biocompatibility tests. Animal models for implantation include SD rat femur body, New Zealand white rabbit femur condyle and mandible, Lanyu pig mandible, and osteoporotic goat spine. Histopathologic examination indicates that the implant is intimately integrated with surrounding bone tissues. The majority of the implant is readily resorbed and replaced by new bone generally in a one-to-one resorption manner as early as 4W post-implantation. The early-stage new bone formation was found much faster in Ezechbone® Granule group than in autologous group. Capsule formation, inflammation, macrophages, necrosis fibrosis or other unwanted tissue responses are substantially absent. Clinical case reports of this presentation focus on a variety of spinal and orthopedic applications. This research was partly sponsored by Southern Taiwan Science Park Bureau Smart Biotech Medical Cluster under contract # CY-05-08-38-107.

(ICACC-S12-P039-2020) Surface Chemistry of Ti_2CT_x

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The properties of the MXens are determined by the surface structure based on different surface groups, mostly a mix of O-, OH- and F-groups, that result from the chemical exfoliation process. However, a thorough understanding of the (surface) structure depending on the exfoliation chemistry is still missing but greatly needed in order to drive this research area further. We used Ti_2CT_x as a model system based on the MAX phase precursor Ti_2AlC that was synthesized by a solid-state microwave synthesis process. We varied the surface functionalization of the resulting MXene during the exfoliation process or by post-synthesis treatments, e.g. by varying the concentration of the hydrofluoric acid, temperature and reaction time. In addition, we attempted the attachment of molecules on the surface of the MXenes. The resulting Ti_2CT_x samples were characterized by XRD, SEM, XPS and NMR and zeta-potential measurements. Preliminary ^1H, ^{13}C and ^{19}F-NMR results show that etching with differently concentrated HF, e.g. 10% and 40% leads to in significant differences in the surface groups and resulting NMR signals. This shows that NMR studies on MXens are a powerful tool and will help to better understand and control the relationship between the surface groups, the synthesis conditions and the materials properties.

(ICACC-S12-P040-2020) Undergraduate Research: Tribology and Wettability of MAX/MAB Phases and Their Composites

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MAX and MAB phases have emerged as novel nanolayered ternary compounds for multifunctional applications. These solids have shown promise as potential materials for tribology applications. In this undergraduate research poster, we will document and review some of the recent progress which has been made in this field. In addition, we will perform wettability and tribology experiments by using different fluids. It is expected that these results will shed light on the tribological behavior of these materials in oil deficient conditions.

(ICACC-S13-P041-2020) Development of SiC core material for LWR

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Toshiba Energy Systems & Solutions Corporation (Toshiba ESS) has been developing core material of Silicon Carbide (SiC) composite made with CVI/CVD method as a candidate of accident tolerant fuel (ATF), in cooperation with Japanese Industries and national universities since 2008. SiC composite to be applied to core material should meet the performance requirement, such as mechanical strength, airtightness or corrosion resistance. Thus Toshiba ESS has proceeded the development through phase 1 (from FY2012 to FY2015) and phase 2 (from FY2016 to FY2019) and succeeded in building manufacturing process of SiC composite which meets the performance and function requirement for practical use in Light Water Reactors (LWR). Currently feasibility studies for mass production after FY2020 are being proceeded. The principal results of the various examinations and the current issues about our research and development shall be presented.

(ICACC-S14-P042-2020) Floating zone growth of Be, Mg, Ca, Sr and Ba doped Ga_2O_3 crystals for scintillator uses

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Scintillators are a kind of fluorescent materials, and they have a function to convert invisible ionizing radiation into visible photons. They are utilized in fields of security, medical imaging, well-logging, and some other applications. Ga_2O_3 is known for the semiconductor material, and optical and scintillation properties of undoped Ga_2O_3 single crystal were reported. Moreover, our group evaluated scintillation properties of Ga_2O_3 doped with ns^2 ions and discovered that Sn-doped Ga_2O_3 showed the highest scintillation light yield among them. In this study, we synthesized the Ga_2O_3 single crystals nominally doped with 1.0% alkali metal elements (Be, Mg, Ca, Sr and Ba) by the floating zone (FZ) method. Then, we evaluated optical and scintillation properties.

(ICACC-S14-P043-2020) Growth and scintillation properties of neodymium activated $YAlO_3$

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Scintillators are one of the phosphors, and they can convert ionizing radiations to UV-NIR photons immediately. The spectrum of the application is wide, including medical imaging, security, well-logging, environmental monitoring and high energy physics. Recently, the trend of R&D of new scintillators is insulator materials doped with Ce which shows efficient luminescence due to 5d-4f transition of Ce^{3+}. On the other hand, some rare earth emission centers can show 4f-4f transition, and some of them can be applicable for the scintillator. In the case of Nd-doped YAG, it exhibited relatively high scintillation light yield of ~ 10000 ph/MeV under ^{137}Cs gamma-ray excitation in the pulse height spectrum. Technically, scintillation decay with several micro seconds is acceptable for practical uses. In order to expand studies of Nd-doped scintillators, in this study, we synthesis and evaluate Nd-doped $YAlO_3$ (YAP) crystals.

(ICACC-S14-P044-2020) Analysis of CNWs grown on sputtered metal oxide and nitride

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In this paper, we characterize the growth of carbon nanowalls (CNWs) on silicon dioxide (SiO_2) and zinc oxide (ZnO), which are used as piezoelectric ceramics, and titanium nitride (TiN), which has excellent abrasion resistance and heat resistance. All metal oxide and nitride were deposited on a p-type Si wafer (100) using radio frequency magnetron sputtering system with 4-inch target of them. CNWs were grown on them using microwave plasma enhanced chemical vapor deposition (MPECVD) with a mixture of methane (CH_4) gas and hydrogen (H_2) gas (temperature: 700°C, microwave power: 1300 W, 10 minutes). In order to observe surface characteristics, field emission scanning electron microscopy (FESEM) was used and energy dispersive X-ray spectroscopy (EDS) was used to confirm the elements of CNWs before and after growth on metal oxide and nitride. To compare the adhesion between them and CNWs, samples were placed in acetone and cleaned using ultrasonic cleaning for a period of time. We also investigated structural characteristics using X-ray diffraction (XRD) and Raman spectroscopy. Electrical properties were confirmed by Hall measurement, and the samples were also examined before and after ultrasonic cleaning.

(ICACC-S14-P045-2020) High light-yield self-activated scintillators based on thallium alkali-metal halides

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The development of novel scintillators with high light yields is of considerable interest. In addition, scintillators that have high effective atomic numbers are required to achieve high detection efficiency with respect to gamma rays or X-rays. In this study, we developed novel scintillators based on thallium (Z=81) alkali-metal halides with high effective atomic numbers. Such compounds exhibited similar emission characteristics for both UV and X-ray excitations with respect to their wavelengths and decay behaviors. As for the scintillation properties, some compounds exhibited high light yields exceeding 40,000 photons/MeV. In addition, we observed scintillation decay with two components whose decay time constants were on the order of several tens and several hundreds of nanoseconds.

(ICACC-S14-P046-2020) Excitation density effects on scintillation properties of Li-based scintillators for neutron detection

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The effects of linear energy transfer (LET), or excitation density, on scintillation properties are important, especially in the discrimination of the detection pulse shape for the neutron detection of events based on the (n,a) reactions of ^6Li or ^{10}B. As the response of photomultiplier tubes is generally much faster than the time scale of the occurrence of scintillation, the detection pulse shape generally resembles the scintillation temporal profile. In recent years, our group has studied scintillation temporal profiles using pulsed ion beams to reveal the underlying physics of the effects of LET on scintillation. In this study, we analyzed the scintillation temporal profiles of Li-based scintillators for the detection of neutrons. LET dependences were explained in terms of two competitions: one is the competition between the energy transfer to the luminescent centers and the quenching in the host due to the interaction between excited states, and the other is the competition among multiple excited states for energy transfer to a nearby luminescent center that leads to energy transfer failure for some of the excited states. In the latter case, subsequent competition occurred among the residual excited states for energy transfer to the Ce^{3+} ions located far from the original position of the excited states; this was followed by quenching.

(ICACC-S14-P047-2020) Relationship between defect formation by X-ray irradiation and thermally stimulated luminescence of binary zinc phosphate glasses

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We examined the relationship between defect formation in the UV region by X-ray irradiation and its luminescence properties of binary zinc phosphate glasses. The emergence of absorption bands in the UV region linearly increased on increasing the irradiation dose. For up to 10 Gy irradiation of X-ray from a tungsten source, the generated absorption bands disappeared after annealing at 350 °C. Moreover, the thermally stimulated luminescence (TSL) intensity

linearly increased on increasing the irradiation dose. There is a linear relationship between the peak area of TSL and that of the generated absorption bands. In contrast, the absorption, i.e., defect, generated by Cu-Ka 1000 Gy irradiation survived after annealing at 350 °C. The generated defects served as emission centers of photoluminescence (PL), which was confirmed by comparison between the optical absorption and PL excitation spectra.

(ICACC-S14-P048-2020) Luminescence of Sn^{2+} center in ZnO-P_2O_5-B_2O_3 glasses

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Photoluminescence (PL) of Sn^{2+} in oxide glasses is affected by the local coordination field because of the electrons in the outermost shell. Because not only the emission energy but also the transparency vary depending on the local coordination, it is worth examining the local distribution of Sn^{2+} cations in oxide glasses, which have a tendency toward phase separation, especially in an ionic (phosphate)–covalent (borate) binary system. Here, we report the structural changes in zinc borophosphate glass and the PL properties of Sn^{2+} in oxide glasses. The building blocks of the main glass network vary depending on the chemical composition. We found that the Sn^{2+} species in B_2O_3-rich glasses differ from those in P_2O_5-rich glasses, as observed in the optical absorption, PL peaks, and PL decay constants. ^{119}Sn Mössbauer spectra indicate that isomer shifts of Sn^{2+} also affect the local coordination change depending on the chemical composition. According to these results for the Sn^{2+} center, we conclude that Sn^{2+} centers are homogeneously dispersed in the borophosphate network without localization around the phosphate region.

(ICACC-S14-P049-2020) Radioluminescence Properties of Pr-doped Li_2O-GeO_2 Glasses

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The scintillator, which emits light under radiation irradiation, plays an important role in radiation measurement applications. Single crystal scintillators are mainly used for such applications, and only a few glass scintillators are used. Glass scintillators were intensively studied around 1960. Scintillation characteristics of silicate glass, borosilicate glass, phosphate glass were investigated. It was found that its performance depends on the type of glass former and silicate glass exhibited excellent performance among them. The Ce-doped lithium silicate glass, that emits light with the excitation energy from the nuclear reaction of ^{6}Li and neutron, has been put to practical use as a neutron scintillator. This material is still used as a standard neutron scintillator. However, glass scintillators are not widely used for γ-ray detection. Because glass materials have excellent formability and durability, we are interested in the research and development of novel glass scintillators. In this study, we focused on GeO_2 as glass former for scintillators. We have investigated scintillation characteristics of GeO_2 containing glasses, and then we have found that Ce-doped $25Li_2O$-$75GeO_2$ glassed shows no significant radioluminescence. In contrast, Pr-doped $25Li_2O$-$75GeO_2$ glasses show radioluminescence. Their decay times under X-ray excitation are acceptable values for scintillators.

(ICACC-S14-P050-2020) Radioluminescence and Dosimetric Properties of Sn-doped ZnO-P_2O_5-SiO_2 Glasses

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Inorganic phosphors including scintillators and dosimetric materials are used for radiation measurements for industrial, medical, and security applications. Over the past few decades, many scintillators and dosimetric materials have been developed. Most of them are single crystals, crystalline powders, or ceramics. In contrast, there are a few products made of glass materials. Glass scintillators are used for thermal neutron detection, but they are not widely used for X-ray and γ-ray detection. Among three types of dosimetric materials such as thermoluminescence (TL), optically stimulated luminescence (OSL) and radiophotoluminescence (RPL) materials, the practical uses of glass dosimetric materials are only in RPL materials. We are interested in glass materials for such applications because they are potentially cost-effective and have excellent formability and durability. Since H. Masai and his group reported that the Sn-doped ZnO-P_2O_5 glasses show the high quantum yields, scintillation and dosimetric properties of Sn-doped glasses are particularly in our interest. Our group have studied scintillation and dosimetric properties of the Sn-doped Li_2O-B_2O_3-SiO_2, SrO-B_2O_3, and $Zn_3(PO_4)_2$-$NaPO_3$ glasses, and we found that their properties depend on compositions of host glasses. In this study, scintillation and dosimetric properties of Sn-doped ZnO-P_2O_5-SiO_2 glasses have been investigated.

(ICACC-S15-P051-2020) Direct ink writing of kaolinite clay using lime, fly ash, and talc as additives

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Kaolinite clay based-ceramics with several inexpensive ceramic powders lime, fly ash, and talc, were fabricated with the direct ink writing technique. These type of clay is abundant worldwide and commonly used in the fabrication of traditional ceramics. In order to tests the samples in compression tests, cylindrical specimens were printed with water to clay ratios between 0.68 and 0.72, with 3.0, 5.0, and 7.0wt% of additives content. Scanning electron microscopy, X-ray fluorescence, and X-ray diffraction tests were also conducted. Results showed that specimens with fly ash as an additive and 0.70 water to clay ratio showed the best performance in terms of workability, mechanical properties, and surface finishing.

(ICACC-S15-P052-2020) 3D printing ceramics using stereolithography

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Additive manufacturing is an ever-growing field with new techniques being developed as well as a wide assortment of materials becoming accessible. With this has come the ability to fabricate ceramics with various 3D printing techniques. In this study, a commercial, off-the-shelf silica loaded resin will be explored using a low-cost commercial stereolithography printer. Through this, a good baseline will be created that others developing their own resin can use as a comparison because to our knowledge, none exists. Two sets of samples – square plates and honeycomb cellular structure, were printed. Measurements were taken to quantify the printing resolution limit of this technique. The green bodies were heat treated to different maximum temperatures, and the effect of temperatures on the microstructure and mechanical properties of the honeycomb cellular components were studied.

(ICACC-S15-P053-2020) Influence of Meta-kaolin on Properties of 3D Printable Cementitious Mixture for Application in Additive Manufacturing

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There is a growing interest in investigating additive manufacturing processes using cement-based materials in the construction industry. However, the approach to developing a viable cementitious mixture for 3D printing relies heavily on repeated trials, until suitable mixture proportions are determined for a given set of materials. The focus of the present study is to examine the behavior of cementitious

mixtures prepared with Portland cement in combination with meta-kaolin (MK) and chemical admixtures such as superplasticizer, viscosity modifying agent, set retarder and additives such as polypropylene fibers. In this parametric study, the influence of meta-kaolin on the rheological behavior of mortars was investigated and the results showed that the addition of meta-kaolin enhances the mixture viscosity, aids in shape retention and reduces bleeding in the mixtures, however setting time was shortened. Rheological properties of these mixtures (i.e. yield stress and plastic viscosity) were measured and correlated with the performance measures such as extrudability, buildability and open time. The results from this investigation showed that MK can beneficially be used to provide a good 3D printable mix, and the proportions of the materials in mixture can be tailored to meet desired rheological and performance measures for viable 3D print operations.

(ICACC-S15-P054-2020) Extrusion-based 3D Printing of Yttria-stabilized Zirconia Nanoparticles through the Presence of Single Polymer Additive

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Additive manufacturing can enable the fabrication of hierarchical, geometrical, and thin-walled ceramic objects. Zirconia has high thermal stability, good resistance to corrosion and chemicals and found use in several applications, such as dental and surgical implants, and composite cell scaffolds, that necessitate biocompatibility. To produce high density, good accuracy, and low shrinkage zirconia-based structures, we have studied the colloidal stability of aqueous suspensions of 3% mol yttria-stabilized zirconia nanoparticles (ZNPs) in the presence of a single additive. This additive, a grafted poly(carboxy ether)(PCE)-based copolymer, was specifically tailored for ZNPs to harness electrostatic stabilization and steric hindrance to optimize the loading of ZNPs and the use of this suspension in extrusion-based 3D printing. We synthesized a matrix of PCE-based copolymers with different comonomer ratios and side chain density to determine the optimum copolymer for a stable dispersion. We have carried out rheological characterization to realize an ink formulation with the highest purity.

(ICACC-S15-P130-2020) Mitigating distortion in binder jet 3D printed ceramics

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Binder jet 3D printing is a solid-state additive manufacturing technique that constructs near net-shaped components layer-by-layer in four stages: powder deposition, spreading, binding, and sintering. Sintering enhances the mechanical properties of the printed component, often at the cost of shape distortion due to material creep. The aim of this work is to understand how the introduction of nanocrystalline ceramic necks, derived from a liquid precursor treatment, affect creep, distortion, and densification. Scanning electron microscopy revealed microstructural differences in treated and untreated samples. Dilatometry experiments revealed that the treatment results in a reduction in creep in the components. The creep strain rate was comparable in both specimens suggesting that although the treatment mitigates distortion, the mechanism for creep is preserved.

(ICACC-S17-P055-2020) Structural-Luminescent Characterization of Eu^{3+}-Activated Spinel-Type $MgGa_2O_4$: Mn^{2+} Phosphors

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This work is aimed to shed light on the mechanism of emission in Eu-doped $MgGa_2O_4$: Mn^{2+} spinel phosphor in correlation with the preferential chemical surrounding of Eu^{3+} activators. This ceramics were prepared via high-temperature solid-state reaction and characterized using XRD with Rietveld refinements, TEM and EDX, positron annihilation lifetime spectroscopy and optical-luminescence spectroscopy. Morphology investigations showed grains of irregular shape with a homogeneous distribution of Eu^{3+}. The inability of Eu penetrates into ceramic grains was confirmed by positron annihilation lifetime spectroscopy. The effect of Eu^{3+} doping results in enhanced positron trapping rate due to occupancy of vacancy-type defects at ceramic grains. This effect is counter-balanced by the reduced decaying of bound positron-electron states in intergranular free-volume spaces. The characteristic bands related to Mn^{2+} and Eu^{3+} ions have been found in optical absorption and excitation spectra. There is an efficient energy transfer from host to Eu^{3+} ions. Photoluminescence emission spectra exhibit matrix luminescence as well as emission band of Mn^{2+} ions and narrow lines of Eu^{3+} in blue, green and red spectral region, respectively. The intensity ratio of Eu^{3+} emission lines indicates the high asymmetry around Eu^{3+} ions.

(ICACC-S17-P058-2020) Characterization of $Bi_{1-x}Eu_xFeO_3$ ceramics prepared by by spark plasma sintering

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$BiFeO_3$ ceramics are difficult to prepare due to Bi_2O_3 volatilisation and kinetic reasons. The focus of the study was the investigation of unconventional spark plasma sintering method on the electrical and magnetic behaviour of Eu substituted $BiFeO_3$ ceramics. Eu substituted $BiFeO_3$ fine-grained ceramics were by SPS at 625°C under 50 MPa applied pressure. XRD results show that at a substitution degree of 10% a polymorphic transition from rhombohedral to orthorhombic polymorph appears. FE-SEM investigations indicated that the incorporation of Eu^{3+} solute on Bi sites determines a strong decrease of the average grain size from 925 nm to 173-217 nm. The ceramics exhibit low values of relative permittivity when increasing the Eu^{3+} content. The structural transition has a beneficial effect on the dielectric losses which decrease with about one order of magnitude. Magnetic hysteresis loops collected on the ceramics showed an antiferromagnetic behavior or a weak ferromagnetic behavior depending on the substitution rate.

(ICACC-S17-P059-2020) Optical properties of Eu substituted $BiFeO_3$ powders and ceramics prepared by sol-gel

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$BiFeO_3$ powders exhibit a number of interesting functional properties, however, it presents difficulties when it comes to processing in terms of thermodynamic instability during heat-treatments and reaction kinetics. The focus of the study was to investigate the influence of Eu substitution in $BiFeO_3$ (0, 5, 10, 15 and 20% substitution) powders on optical properties. Eu substituted $BiFeO_3$ powders were synthetized by sol-precipitation method from nitrates precursors. The powders were thermal treated at 600°C/1h for crystallization. The XRD and Raman results on powders show that Eu substitution induces a structural phase transition from

rhomboedral (R3c) to orthorhombic (Pnma) polymorph. The average particle size ranged between 155 and 40 nm, decreasing with the increase of Eu^{3+} content. The most pronounced ferromagnetic behavior was observed for $Bi_{0.95}Eu_{0.05}FeO_3$ composition, which exhibited a saturation magnetization of 1.65 emu/g and a coercitive field of 100 Oe. The luminescence emission increases with the increase of the Eu^{3+} content, but the quenching of the fluorescence specific to europium ions seems to be induced by a masking effect of $BiFeO_3$, as in other rare-earth doped bismuth ferrite systems.

(ICACC-GYIF-P060-2020) Effects of Morphology on the Compressive Mechanical Properties of Ice-templated Sintered Metal Oxides with Directional Porosity

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Conventionally processed sintered porous metal oxides with open-cell architecture such as lithium titanate (LTO) are utilized as electrode material in batteries for energy storage. Significant gain in battery performance can be achieved by introducing directional porosity in electrodes which will lower pore tortuosity and improve lithium ion transportation. While the majority of processing methods generate porous materials with random pore orientation, ice-templating enables the synthesis of directionally porous ceramics with improved mechanical properties. However, progress in utilizing this technique toward the synthesis of sintered porous metal oxides for energy related applications has been limited. One interesting aspect in ice-templating is that at a targeted level of porosity, pore morphologies can be drastically varied while preserving pore directionality by changing the processing variables and additives. In this presentation, we will discuss the specific effects of different processing variables and additives on pore morphology in ice-templated sintered LTO ceramics, and the influence of the resulting pore morphologies on compressive mechanical properties and failure characteristics. The results will significantly contribute to understand the processing-microstructure-property relationships in ice-templated sintered LTO materials.

(ICACC-GYIF-P061-2020) Processing and Mechanical Characterization of Ice-templated Alumina-Epoxy Composites

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In the ice-templating technique, an aqueous ceramic suspension is solidified under the influence of unidirectional temperature gradient. During the solidification process, phase separation of solvent crystals and ceramic particles results in a unique morphology containing alternate layers of ice and ceramic. Freeze drying and sintering result in a ceramic foam that contains alternate layers of ceramic lamella walls and pores oriented along the growth direction of ice crystals. By infiltrating the ceramic foams with a polymer phase, ice-templated ceramic-polymer composites are developed. In this work, we will present the on-going work on the processing and mechanical characterization of ice-templated alumina-epoxy composites. The work will show the effects of composition and freezing kinetics (used during unidirectional solidification) on the compressive strength, variability of strength, and overall compressive mechanical response of ice-templated alumina-epoxy composites. Microstructural analysis of the ice-templated ceramic alumina foams, ice-templated alumina-epoxy composites, and deformed composite materials will enable better understanding of the mechanical properties and failure characteristics in relation to processing aspects of the composites.

(ICACC-GYIF-P131-2020) Physico-chemical and biological characterization of phosphate bio-glass implant-type coatings fabricated by radio-frequency magnetron sputtering

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Currently, there is a search for innovative implant-coating designs, acting as smart multi-functional biointerfaces capable to tackle the most demanding healthcare requirements: e.g. controlled release of therapeutic ions, degradation speed matching bone growth rate, antimicrobial efficiency. Phosphate bio-glasses (PBGs) can, when appropriately designed, meet these requirements and stimulate the biological responses at a molecular level. In this work, radio-frequency magnetron sputtered (RF-MS) is used as tool to tailor PBGs structure starting from a single parent composition (mol%): 50-P_2O_5, 35-CaO, 10-Na_2O and 5-Fe_2O_3. Resorbable PBG coatings have been synthesized by RF-MS under different conditions, with the aim to engineer the network glass connectivity – the key control parameter of PBG degradation speed. The (ellipsometry, AFM, EDS, FTIR) results indicated that by simply modifying the inert (argon) sputtering pressure one can incrementally modify (and thus design) the morphology, composition and structure of the films. The possibility to fine tune the PBG films physico-chemical features, and thereby their biological response (as shown by the in vitro degradation and cytocompatibility assays), could constitute an important technological step in their future development as a new generation of highly performant implant coatings.

(ICACC-GYIF-P132-2020) Ternary and quaternary modified silica with transition metal for ethanol transformation

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1,3-Butadiene (BD) is used for the production of polymers, such as Styrene-Butadiene Rubber (SBR), leading the market of vehicles tires, which account for more than 50% of the available butadiene, globally and recently gained renewed interest in finding new synthesis methods starting from ethanol. Different types of catalysts have been recently evaluated in the ethanol transformation into BD and a well-studied class of materials is the MgO/SiO_2 system [1]. In this study Mn-M@silica functionalized hollow spheres were used in ethanol transformation. Mn-M (M= Zn, Fe, Ni, Cu, Co) core is found inside the SiO_2 shell, and are prepared by hydrothermal carbonation (HTC) using glucose. The shell was also modified with ZrO_2 oxide in order to increase the acidity of the catalyst. The catalysts were characterized using several techniques: X-Ray diffraction, TPD-NH_3, Raman, FTIR, XP Spectroscopy, SEM-EDX. The XRD results evidenced the presence of metallic species inside the core after the hydrothermal carbonation, which are further oxidized when silica or SiO_2-ZrO_2 is added to form the shell. SEM analyses confirm the formation of spheres and depending on the preparation method, 300 nm spheres were obtained. The materials prepared showed good activity for the ethanol transformation at a space velocity of 1.1 g EtOH/g catalyst per h.

(ICACC-GYIF-P135-2020) Undergraduate Research: Design of Novel Hydrogel based Functional Materials

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Recently, National Academy of Engineering (NAE) has come up with 14 Grand Challenges. Different schools are developing Global Challenge Scholar's Program (GCSP) to incorporate some of the key elements of grand challenge in the educational program. Some of the important elements of the GCSP program are: "(a) A creative learning experience connected to the Grand Challenges such as research or design projects, (b) Authentic experiential learning with clients and

mentors that includes interdisciplinary experience in fields such as public policy, business, law, medicine, ethics, and communications, (c) Entrepreneurship and innovation, (d) Global and cross-cultural perspectives, and (e) Development of social consciousness through service-learning "[1]. In this poster, we will present the recent progress in undergraduate (UG) research as an integral component of GCSP in the grand challenge area of biomedical based innovations. More particularly, the team will focus on different types of hydrogels. The mechanical property and microstructure of these hydrogels will be documented. It will be compared with other hydrogels.

(ICACC-WW-P067-2020) Wear mechanism of spark plasma sintered MWCNTs reinforced zirconia composites under dry sliding conditions

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Advanced ceramics composites have shown interesting tribological performances in a wide range of applications during the last decades. In the present work, the tribological properties of ZrO_2 – 8 mol.% Y_2O_3 (8YSZ) containing (0 wt. %, 1 wt. %, 5 wt. % and 10 wt. %) of MWCNTs were investigated. The friction and wear rates were evaluated using the ball-on disk technique at dry sliding conditions. The sliding speed was set up from low (V_1=0.036m/s) to high (V_2=0.11m/s) values for comparative evaluation of the speed rate to the tribological properties of the studied composites. The microstructural examination after fractographic test revealed two types of fracture mode (trans-granular fracture and inter-granular fracture) depending on MWCNT content. The morphology of the wear tracks was visualized using Scanning Electron Microscopy. The factors responsible for achieving the highest wear rate in the monolithic material at low speed rate and significantly lower wear in the composites with MWCNTs have been determined. Finally, energy dispersive X-ray spectroscopy measurements were applied inside and outside the wear track to investigate the chemical composition of tribolayers accompanying the specific tribological properties obtained in our composites.

(ICACC-WW-P062-2020) Wetting Properties Of Molten CaO-MgO-Al_2O_3 Silicates On Model Environmental Barrier Coating Materials

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The use of SiC/SiC ceramic-matrix composites will allow higher operating temperatures and lighter parts, improving the efficiency of both jet and land-based power generation turbine engines. In such environments, SiC oxidizes, and the resulting silica surface reacts with water vapor and volatizes. Environmental barrier coatings (EBCs) are designed to prevent these reactions. However, the coating interaction with ingested airborne silicates (CMAS) can lead to undesirable reactions or grain boundary infiltration, resulting in failure that leaves the turbine part unprotected. This study aims to determine the effects of temperature and surface crystallographic orientation on CMAS wetting behavior using single crystals with different surface termination planes of a model EBC material, yttrium aluminate perovskite (YAP). The effect of surface orientation on these reactions will guide future grain boundary engineering to limit CMAS infiltration. We found that the CMAS contact angle on YAP varied with temperature and surface orientation, where CMAS/YAP contact angles are greater than 90° at 1200°C. By 1265°C, all three tested YAP orientations transitioned to wetting behavior, which could be due to a known chemical reaction product (Y-Ca-Si apatite). The effect of the surface termination plane on the CMAS/YAP interaction and its ramifications for CMAS-mitigation will be discussed.

(ICACC-WW-P063-2020) Bioceramics in the $Ca_2P_2O_7$ – $Mg_2P_2O_7$ system with a tailored architecture, fabricated via stereolithography, for personalised bone-tissue engineering

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Reconstructive surgery and orthopedics need a new generation material for bone replacement and grafting with a personalized architecture. This study aim is to develop resorbable bioceramics in the $Ca_2P_2O_7$ – $Mg_2P_2O_7$ system with a tailored pore space architecture created by the method of stable layer-by-layer 3D printing from a light-cured suspension for personalized bone-tissue engineering. Substitution of calcium phosphates ceramic with alternative cations, such as magnesium, coupled with condensed phosphate ions play a significant role in the bone remodeling process, affecting the early-stage of bone regeneration through stimulating osteogenic differentiation, prohibiting osteoclastic activity, and transforming into mechanically enhanced hydroxyapatite bone tissues. Osteoconductivity of the implants and sufficient strength are also achieved by obtaining a macroporous material with a specific architecture (gyroid and Kelvin), in which up to 80% of the total volume is connected pores. For these bioceramics, the composition of suspension for DLP-printing is revealed, rheological, mechanical and toxicological tests are carried out and in vivo study is done. RFBR partially supported this study under Grant No. 19-38-90274\19, 18-29-11079.

(ICACC-WW-P064-2020) Effect of Multi-Element Doping on Cubic Phase Stabilization of $Li_7La_3Zr_2O_{12}$ by Using Waste Materials

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Renewable energy sources require energy storage systems. However, there are some problems about liquid electrolytes used in the storage systems, such as battery explosions. Solid state electrolytes offer a solution for this problem. The more recently developed and more promising option is $Li_7La_3Zr_2O_{12}$. There are a few studies based on the effect of Fe doping. The only Fe source used so far has been Fe_2O_3. Different Fe containing waste material can be used, for example, (NiCoMn) arising from nickel metal production. For this purpose, the present study is aimed to use nickel waste material's potential to achieve cubic phase of LLZO through multi-valent doping to increase ionic conductivity. 2-10 wt. % nickel waste material incorporated LLZO powders were synthesized by solid state reaction method. A low temperature cubic phase stabilization after calcination, improvement on ionic conductivity, controlled grain size were observed in comparison to pure Fe doping.

(ICACC-WW-P065-2020) Particle-Specific Design of a Grafted Copolymer Enables 3D Printing of Highly-Loaded Ceramic Inks

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There is a consensus, both from commercial suppliers and academia, on the need for precise calibration of interparticle forces — which determine the "printability" of the suspensions— to design ceramic inks for additive manufacturing. The current calibration of these

forces relies on electrostatic repulsion and requires changing the pH of the medium, adding salt, and utilizing oppositely-charged polyelectrolyte species in addition to other binders, defoamers, and organic solvents. However, i) they cannot provide the scalability and cost-effectiveness of water, ii) concurrent optimization of minimum 2 additives complicates the ink formulation and the precise dimensional control of the final object/feature due to high volume, iii) usually requires binder removal steps after printing. To design a single additive that offers stability and viscosity-control, we utilized a grafted random copolymer that harnessed electrostatic repulsion and steric hindrance. We changed the charge, ionization capacity, and structure of poly(ethylene glycol) (PEG) grafted random copolymer of acrylic acid (AA) and 2-acrylamido-2-methylpropane sulfonic acid (AMPS), to reach theoretical particle loadings as calculated by Krieger-Dougherty equation. This particle-specific design of an additive enabled the realization of alumina inks with > 80 wt. % particle loading with < 1,5 wt.% use of a single additive.

(ICACC-WW-P066-2020) Preparation of UV- curable $MgAl_2O_4$ slurry for stereolithography- based additive manufacturing

P. Zubrzycka*[1]; M. Borlaf[1]; M. Radecka[2]; T. Graule[1]

1. Empa, Laboratory for High Performance Ceramics, Switzerland
2. AGH University of Science and Technology, Poland

Additive manufacturing enables to produce parts with different geometry and high complexity on the base of CAD model using the same equipment and manufacturing procedure for any shape. Forming ceramic parts by stereolithography- based additive manufacturing consist of following steps: slurry preparation, 3D printing involving photopolymerization process, debinding and sintering. Slurry preparation plays a crucial role for the further sintering process and the final microstructure of the printed parts. Therefore, UV- curable slurry should fulfill following requirements: high solid load, nearly agglomerate- free powder distribution, low viscosity, shear- thinning behavior, time- stability. This work presents the study on the influence of dispersant structure on the abovementioned properties of the slurry. To characterize the raw material following measurements were performed: SEM, TGA- DSC, dilatometry, BET, XRD. To optimize the slurry, rheological properties and particle size distribution were examined. Finally, the printed parts were debinded and sintered, and they were characterized by density measurement and optical microscopy.

Wednesday, January 29, 2020

4th Pacific Rim Engineering Ceramics Summit

Challenges and Opportunities for Ceramic Technologies II

Room: Coquina Salon E

Session Chairs: Manabu Fukushima, National Institute of Advanced Industrial Science and Technology (AIST); Zoltan Lences, Institute of Inorganoc Chemistry, Slovak Academy of Sciences

8:30 AM

(ICACC-PACRIM-013-2020) Current Trends and Future Directions of Ceramic Membrane Technology 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 presentation, we focused the current trends and future direction of ceramic membrane technology 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.

9:00 AM

(ICACC-PACRIM-014-2020) Stereolithographic Additive Manufacturing of Ceramic Components for Energy Storage (Invited)

S. Kirihara*[1]

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

In stereolithographic additive manufacturing, 2-D cross sections were created through photo polymerization by UV laser drawing on spread resin paste including ceramic nanoparticles, and 3-D models were sterically printed by layer lamination. In this investigation, solid electrolyte dendrites of yttria-stabilized zirconia (YSZ) with spatially ordered pores were processed for fuel cell miniaturization. Subsequently, ceramic sheets of lithium-lanthanum-titanate (LLT) with micro emboss patterns were developed for all-solid batteries. 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 3-D printing, nanometer sized ceramic particles were dispersed in to acrylic liquid resins at 50 % in volume fraction. The resin paste was spread on a glass substrate at 100 μm in layer thickness by a mechanically moved knife edge. An ultraviolet laser beam of 355 nm in wavelength was adjusted at 50 μm in variable diameter and scanned on the spread resin surface. Composite precursors were dewaxed and sintered in the air atmosphere to obtain the fine ceramic microstructures.

9:30 AM

(ICACC-PACRIM-015-2020) Understanding the Stochastic Variability in Ice-Templated Ceramic through Micro-Mechanical Modeling (Invited)

O. Kravchenko*[1]; S. Sattar[2]; S. Kravchenko[3]

1. Old Dominion University, Department of Mechanical and Aerospace Engineering, USA
2. Old Dominion University, USA
3. Purdue University, USA

To understand the variability in macroscopic mechanical behavior of ice-templated porous materials, the homogenized strength and stiffness must be expressed as a function of local material descriptors, e.g. microscopic morphology and relative density. Therefore, the computational multi-scale framework is needed to study the behavior of ice-templated porous material structures. The presentation introduces the micromechanical finite element modeling that was used to study the effects of density and morphology on the compressive behavior of ice-templated alumina. The proposed FEA framework utilized the representative volume approach with hexagonal lamellar wall orientation in the plane normal to the ice-crystal growth direction and periodic boundary conditions, while the fracture behavior was modelled using the smeared cracking. The modeling showed that the mechanical behavior of ice-templated alumina on microscale with close to unidirectional pore orientation

is governed by brittle fracture rather than by buckling of lamellar walls. The validated computational model was used to explore the bulk behavior as a function of the relative density and lamellar wall thickness. The future research challenges require greater understanding of stochastic nature of the morphology that can be observed on the microscale and how it affects the macroscopic response.

10:10 AM

(ICACC-PACRIM-016-2020) Development of High Performance SiC-Based Ceramics and Composites (Invited)

K. Yoshida*[1]

1. Tokyo Institute of Technology, Laboratory for Advanced Nuclear Energy, Institute of Innovative Research, Japan

Silicon carbide (SiC) ceramics has been considered as a key material for structural applications such as aerospace industries, nuclear and fusion applications and high-temperature gas turbine at high temperatures. Many researchers have been studying SiC-based ceramics with excellent properties. Our group have been developing high performance SiC-based ceramics and composites (monolithic SiC ceramics, porous SiC ceramics and SiC fiber-reinforced SiC matrix composites). Porous SiC ceramics have received great attention in environmental and energy fields, and our group have proposed the surface functionalization of porous ceramics based on in-situ grain growth. SiC fiber-reinforced SiC matrix composites have been expected to be applied for high temperature components in jet engines. We have developed novel fabrication process of interphases for high performance SiC_f/SiC composites. Furthermore we have been studying monolithic SiC ceramics with excellent properties. In this symposium, our research activities on high performance SiC-based ceramics and composites will be introduced and discussed.

10:40 AM

(ICACC-PACRIM-017-2020) Additive Manufacturing of Ceramics using Preceramic Polymers (Invited)

G. Franchin[1]; H. Elsayed[1]; P. Colombo*[1]; K. Huang[1]

1. University of Padova, Industrial Engineering, Italy

Preceramic polymers are precursors for ceramic phases of different composition. They convert into nano-structured ceramic materials in the system Si(X)OCN (with X = Al, Ti, Zr, etc.), also called PDCs or Polymer-Derived-Ceramics, by high temperature pyrolysis. This talk will discuss the fabrication of porous structures starting from pure preceramic polymers (e.g. silicone resins) or silicone resins plus reactive fillers to produce advanced silicate ceramic phases, including bioceramics and Ceramic Matrix Composites, suitable for different potential applications. Different types of additive 3D manufacturing techniques were employed, including: a) direct printing using a fused deposition printer; b) direct printing using a paste extrusion printer (Direct Ink writing); c) indirect printing using a powder bed-based printer (in collaboration with researchers from BAM, Berlin, Germany); d) indirect printing using a stereolithographic printer; e) indirect printing with sub-micron resolution using 2 Photon Polymerization fabrication. Advantages and disadvantages of the different processing techniques employed, in relation to the use of preceramic polymers, will be discussed, and examples of produced and characterized porous structures for potential use in different applications will be presented.

11:10 AM

(ICACC-PACRIM-018-2020) Structural Analysis of $BaTiO_{3-x}(OH)_x$ tetragonal nanorods fabricated by hydrothermal precipitation method (Invited)

M. Inada*[1]

1. Kyushu University, Center of Advanced Instrumental Analaysis, Japan

It is known that cubic barium titanate ($BaTiO_3$) particles easily form by hydrothermal method. Generally, the tetragonal $BaTiO_3$ is prepared by calcination or dry heat treatment at over 1000°C, resulting in significant particle growth. In the previous study, we fabricated tetragonal $BaTiO_3$ nanorods by hydrothermal treatment of hydroxide precursor with 10vol% ethylene glycol (EG) as solvent (Ceram. Inter., 41 (2015) 5581). The crystallization of cubic $BaTiO_3$ via dissolution-reprecipitation of precursor could be suppressed by the addition of EG, resulting in the formation of tetragonal $BaTiO_3$ under hydrothermal treatment at 200°C. Recently, we found that the synthesized tetragonal $BaTiO_3$ includes a much amount of hydroxyl (OH) group and the OH group influences the stability of $BaTiO_3$ tetragonality. In order to clarify the effect of OH group on the tetragonality of $BaTiO_3$, the structural analysis was carried out in this study. XRD analysis indicates that the oxygen site of synthesized tetragonal $BaTiO_3$ was partially occupied by OH ion. The OH content and c/a ratio increased by EG addition at hydrothermal treatment. The tetragonality of synthesized nanorod disappeared by heat treatment, indicating that substituted hydroxyl group stabilized the tetragonal crystalline structure.

11:40 AM

(ICACC-PACRIM-019-2020) Porous SiC ceramics with excellent thermal insulation performance and high mechanical strength

R. Malik*[1]; Y. Kim[1]

1. University of Seoul, Dept. of Materials Science & Engineering, Republic of Korea

Nano-SiC porous ceramics are potential candidates for high-temperature thermal insulators for high technology and re-entry vehicles that experience surface temperature as high as 1650 °C due to atmospheric resistance. However, a major problem hindering their successful application is their low strength and high solid thermal conduction. In the present research, we have developed novel thermal insulators with secondary phase bonding which facilitated low thermal conductivity due to the creation of multiple phonon scattering interfaces between adjacent SiC particles and also provided high inter-particle bonding strength. Trimodal hierarchically (micro-/meso-/macro-) porous SiC ceramics processed form nano β-SiC, polysiloxane as starting powders using carbon as a pore former exhibited a very low thermal conductivity (0.047 $Wm^{-1}K^{-1}$, 74.1 % porous), which is an order of magnitude lower than the previously reported lowest thermal conductivity (0.14 $Wm^{-1}K^{-1}$, 76.3 % porous) for powder processed porous SiC ceramics. The porous SiC ceramic with 10 wt% polysiloxane exhibited 420 % higher specific compressive strength compared to the previously reported value for nano-SiC porous ceramics at a similar thermal conductivity (~0.15 $Wm^{-1}K^{-1}$).

Current trends: Coatings

Room: Coquina Salon E

Session Chairs: Hua-Tay Lin, Guangdong University of Technology; In-Hyuck Song, Korea Institute of Materials Science

1:30 PM

(ICACC-PACRIM-020-2020) Plasma Resistant YOF Coatings for the Silicon Wafer Processing Equipments (Invited)

S. Lee*[1]; Y. Oh[1]

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

Control of contamination particles in the semiconductor industry is a key factor in the overall process yield. In this presentation, we will discuss the possibility of oxyfluoride ceamics as plasma resistant materials for suppressing the contamination particles. Generally, when oxide ceramics are exposed to a fluorine plasma, the surface of the ceramics is transformed into a thin layer containing high amount of fluorine. Then, the concomitant sputtering of the transformed layer induced by ionized species in the plasma produces fluorine-based particles on the surface layer of the ceramics. The difference in the thermal expansion coefficient between the generated particles and the oxide substrate develops residual stresses due to the temperature change during the wafer processing, resulting

in fall-off of the particles from the ceramics surface. Therefore, it is important to reduce the difference in the coefficient of thermal expansion to suppress the contamination by fall-off of the particles. From this point of view, oxyfluoride materials having a large thermal expansion coefficient can reduce the generation of contamination particles in a semiconductor fabrication process. In this presentation, we will also discuss other benefits of the oxyfluoride mateirals as well as new coating methods of the oxyfluoride materials.

2:00 PM

(ICACC-PACRIM-021-2020) Advancing Development of Environmental Barrier Coatings Resistant to Attack by Molten Calcium-Magnesium-Aluminosilicate (CMAS) (Invited)

V. L. Wiesner[1]; J. L. Stokes[2]; N. P. Bansal[3]; G. Costa[3]; B. Kowalski[2]; M. J. Presby[2]; C. Bodenschatz[4]; B. S. Good[5]; M. Kulis[2]; B. J. Harder*[4]

1. NASA Langley Research Center, Advanced Materials and Processing Branch, USA
2. NASA Glenn Research Center, Environmental Effects and Coatings Branch, USA
3. NASA Glenn Research Center, USA
4. NASA Glenn Research Center, Environmental Effects and Coatings, USA
5. NASA Glenn Research Center, Durability and Protective Coatings Branch, USA

Ceramic matrix composites (CMCs) are a leading material system to replace metal-based parts in the hot-section of air-breathing turbine engines to improve fuel efficiency in aircraft engines. CMCs have higher temperature capabilities and lower density compared with traditional metallic structural materials. However, silicon-based CMCs are susceptible to oxidation in the harsh combustion environment encountered in turbine engines. Consequently, environmental barrier coatings (EBCs) are being developed to protect CMC components to improve durability and extend service life of CMCs. Sand, volcanic ash and other particulate debris, which are generally comprised of calcium-magnesium-aluminosilicate (CMAS) and other trace oxides, are routinely ingested by aircraft engines. At temperatures above 1200°C, CMAS particulates melt. Near target operating temperatures (~1500°C) of future CMC-based aircraft engines, molten CMAS behaves like a viscous melt that can infiltrate and chemically interact with protective coatings. These interactions can cause premature failure of the EBC system and ultimately the overall CMC engine component. Degradation of candidate EBC materials by molten CMAS will be presented with a focus on recent work, as well as methods of evaluating the complex high-temperature materials interactions, underway at NASA Glenn Research Center.

2:30 PM

(ICACC-PACRIM-022-2020) Strategies for improving oxygen shielding performance of multilayer EBCs (Invited)

S. Kitaoka*[1]; T. Matsudaira[1]; M. Wada[1]; M. Tanaka[1]; T. Ogawa[1]

1. Japan Fine Ceramics Center, Japan

Environmental barrier coatings (EBCs) can play a key role in enabling SiC fiber-reinforced SiC matrix composites to be employed as advanced hot-section components in airplane engines. EBCs must exhibit excellent oxygen/water vapor shielding characteristics and thermomechanical durability in severe combustion environments. Thus, a multilayer structure is generally adopted, with each layer having its own unique properties. A large oxygen potential gradient ($d\mu_O$) across an EBC at elevated temperature results in inward diffusion of oxide ions and outward diffusion of cations. Cation transport through an oxide layer typically induces decomposition of the oxide, and can thus cause the EBC structure to collapse. Therefore, to develop a robust EBC with excellent gas shielding performance, it is necessary to both understand and control mass transfer within the EBC. We have elucidated the mass transfer processes within the constituent oxides of EBCs by determining the oxygen permeability for wafers cut from sintered bodies serving as model EBCs. The results indicated that controlling the mobility of the ions constituting the oxides by addition of dopants, creating a difference in cation chemical potential at the interfaces of multilayered EBCs, and utilizing surface charges produced under a $d\mu_O$ provide effective approaches for improving the oxygen shielding performance of EBCs.

3:20 PM

(ICACC-PACRIM-023-2020) Chemical Vapor Deposition and Microcantilever Beam Testing of Alumina–hafnia Eutectic Composite Films (Invited)

A. Ito*[1]

1. Yokohama National University, Environment and Information Sciences, Japan

Alumina is a traditional engineering ceramic but is utilized in various industrial products, such as protective coatings for cutting tools. Hafnia is promising for high-temperature engineering ceramics used in thermal and environmental barrier coatings for turbine blades in aircraft engines because these have a higher melting point and lower thermal conductivity than zirconia. In addition, alumina–hafnia composites exhibit self-organized microstructure due to a eutectic nature of alumina–hafnia system. Although alumina–hafnia composite films can be a novel structural and functional coating, the formation of eutectic microstructure was limited to melt-solidification process. On the other hand, the mechanical properties of ceramic components in the form of coating would be different from that of bulk. However, mechanical properties of coatings are hard to evaluate excluding the influence from the underlying substrate for typical macroscopic indentation and bending tests. This talk will introduce you to our recent challenges on chemical vapor deposition (CVD) of alumina–hafnia eutectic composite films and evaluating mechanical properties of single columnar grain in CVD-hafnia films using focused ion beam milling and microcantilever beam testing.

3:50 PM

(ICACC-PACRIM-024-2020) RE-silicate multifunctional thermal and environmental barrier coatings: Current status and perspectives (Invited)

J. Wang*[1]

1. Shenyang National Laboratory for Materials Science, Institute of Metal Research, Chinese Academy of Sciences, Advanced Ceramics and Composites Division, China

SiC_f/SiC ceramic matrix composites (CMCs) are the key high temperature structural materials in future gas turbine engines. Multifunctional thermal environmental barrier coating (TEBC) is critically requested to protect CMC-components from severe high temperature oxidation and corrosion in extreme combustion environments. Current approaches basically focused on the fundamental and technological developments of RE-silicate TEBCs for balanced performances, resources and costs. Future optimal choices of RE-silicate TEBCs request the comprehensive knowledge of their property up limit in extreme conditions. Efficient screening of TEBC candidates is succeed through strategic material informatics that discloses the trend and mechanism of property degradations in extreme environments. Once we have the database and selection guideline of RE-silicate TEBCs, advancements of different powder fabrication and coating processing technologies would promote robust TEBCs for advanced SiC_f/SiC CMCs.

4:20 PM

(ICACC-PACRIM-025-2020) Development of Aluminum Nitride Piezoelectric Thin Film by Doping (Invited)

M. Uehara*[1]; S. Anggraini[1]; K. Hirata[1]; H. Yamada[1]; M. Akiyama[1]

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

Wurtzite aluminum nitride (AlN) is a piezoelectric material. It is used for application of microelectromechanical systems (MEMS) such as a sensor and bulk acoustic wave (BAW) resonator because

these materials have high hardness, good temperature stability and high mechanical quality factor. The piezoelectric property of AlN is significantly increased by Sc doping. The d_{33} piezoelectric efficient of Sc-AlN is approximately 5-times compared with that of AlN. Sc-AlN has received a great deal of attention in application to MEMS. However, Sc is expensive element for industrial use. Many groups are searching for alternative dopant. In this paper, we introduce our investigation results about the doping effect on AlN. Mg-Nb co-doping is effective. The d_{33} of $Mg_{39.3}Nb_{25.0}Al_{35.7}N$ is 22 pC/N, which is about 4-times that of AlN. The d_{33} is increased by Mg-Nb co-doping, is not increased by Mg or Nb single doping. We discuss about the influence of doping on piezoelectric properties and crystal structure of AlN.

4:50 PM

(ICACC-PACRIM-026-2020) Porous Functional Nitride Ceramics and their Applications (Invited)

F. Chen*[1]; M. Jia[1]; Q. Shen[1]; L. Zhang[1]

1. Wuhan University of Technology, State Key Lab of Advanced Technology for Materials Synthesis and Processing, China

Porous ceramics are a new type of functional ceramic and are gradually being applied to electrode substrates, catalyst supports and filter membranes. In addition to superior mechanical properties, some nitride ceramics also have unique functions such as low dielectric properties or high electrical conductivity. For these functional characteristic, porous nitride ceramics for extreme environments have been developed. In the research of lightweight high-strength porous ceramics, a new system of phosphate-bonded Si_3N_4 porous ceramics with multi-scale pore structure and its low-temperature sintering technology were proposed, and the application of the Si_3N_4 porous ceramics with low dielectric properties in the high speed permeable radome was realized. In the research of conductive porous ceramics, the gel-casting and pressureless sintering technology of TiN porous ceramics with high strength and porosity were developed. It is of great significance for reducing the cost of high-temperature energy storage batteries when using TiN porous ceramics as the membrane materials. The TiN porous ceramics conductive membrane, which achieves chemical selectivity by preferred faradaic reaction instead of by regulated ionic conduction.

9th Global Young Investigator Forum

Multi-functional materials for water catalysis

Room: Coquina Salon G

Session Chair: Daniele Benetti, Institut National de la Recherche Scientifique

8:30 AM

(ICACC-GYIF-023-2020) MOF-templated TiO_2/Quantum Dots heterojunction as photoanode for efficient Photoelectrochemical Hydrogen Generation

L. Shi*[1]; D. Benetti[1]; F. Li[1]; Q. Wei[2]; F. Rosei[1]

1. Institut National de la Recherche Scientifique, Énergie Matériaux Télécommunications, Canada
2. University of Jinan, Chemitsry, China

The separation and transport of photogenerated charges carriers are two of the most crucial factors that determine the overall efficiency of photoelectrochemical (PEC) systems. Various strategies have been exploited for achieving efficient charge separation and transfer. Among them, the design of semiconductor heterojunction has been shown to be one of the most effective approaches. Titanium dioxide (TiO_2) has been one of the most common metal oxide used for water splitting. However, the control of its phase alignment and the interface structure remains a challenge. Furthermore, TiO_2 have a limited absorption of sunlight due to its large intrinsic band gap (3.2 eV). Herein, we explore a simple metal organic framework (MOF)-derived synthesis to obtain a controlled mixed-phase (anatase and rutile) of TiO_2 nanoparticles, which retain the MOF crystal morphology. Compared with commercial TiO_2 films, the MOF-derived TiO_2 film sensitized by core-shell CdSe@CdS QDs, showed an enhanced PEC device stability of +42.1% and PEC performance of +47.6%. The enhanced performance is due to the presence of mixed rutile/anatase phases, that creates a favorable band energy alignment for the separation of the photogenerated charges. The proposed MOF-derived TiO_2 is an efficient strategy to improve the efficiency of the TiO_2-QDs heterojunction based PEC system for hydrogen generation.

8:50 AM

(ICACC-GYIF-024-2020) Ferroelectric polarization-enhanced charge separation in ferroelectric-metal oxide semiconductor hybrid for photoelectrochemical applications

M. Zhang*[1]; F. Li[1]; D. Benetti[1]; L. Shi[4]; R. Nechache[2]; X. Qi[5]; F. Rosei[3]

1. Institut National de la Recherche Scientifique, Énergie Matériaux Télécommunications, Canada
2. Ecole de technologie Superieure, Electrical Engineering, Canada
3. INRS, Canada
4. INRS, EMT, Canada
5. Northeastern University, China

Efficient charge separation for photoelectrochemical (PEC) water-splitting is still a major challenge. Here we successfully synthesized a ferroelectric-semiconductor hybrid structure device in which the photoanode is also sensitized with colloidal quantum dots (QDs). By tuning the amount of ferroelectric materials $BaTiO_3$ (BTO) incorporated in the TiO_2, it is possible to generate a considerable enhancement of photocurrents density (J_{ph}). Specifically, it was found that the optimum amount of BTO in the semiconductor, enhanced the J_{ph} of more than 40%, with the highest J_{ph} and charge-separation efficiency ($\eta_{separation}$) of 12.1 mA/cm^2 and 22.3% at 0.5 V versus the reversible hydrogen electrode (RHE) respectively. Through systematically investigation of the light absorption and band alignment, the increased J_{ph} was proved to be due to a better charge separation induced by ferroelectric polarization effect. The PEC performance can be further manipulated by controlling the polarization state. This work provides unique insights to improve the performance of PEC photoelectrodes in which the material with combined ferroelectric and semiconducting features is sensitized using colloidal "giant" QDs.

Advanced and Nanostructured Materials for Biomedical Applications

Room: Coquina Salon G

Session Chair: To be determined, ACerS

9:10 AM

(ICACC-GYIF-025-2020) Spectral tuning of the small and bright Li-based rare-earth nanoparticles

A. Skripka*[1]; T. Cheng[1]; C. Jones[2]; J. Marques-Hueso[2]; F. Vetrone[1]

1. Institut National de la Recherche Scientifique, Canada
2. Heriot-Watt University, United Kingdom

Rare-earth nanoparticles (RENPs) are a particularly attractive class of luminescent nanomaterials poised for many biomedical applications. RENPs not only possess the classical Stokes emission, but also upconvert low energy radiation, like near-infrared (NIR), to light of UV-vis wavelengths. Opposite to other multiphoton luminescence processes, upconversion can be attained with inexpensive laser sources operating at 10^{-3}-10^3W/cm^2. RENPs' responsiveness to NIR light means that they can be excited deeper within the body, since biological tissues are partially transparent in NIR region. Thus, RENPs are being studied as imaging agents, sensors, and mediators of photochemical processes. In that regard, field of materials science is constantly pushed in the direction of brightest, color tunable,

and at the same time small RENPs – in order to ensure efficiency and biocompatibility. Here, we study a library of $LiYbF_4$: RE^{3+}/$LiYF_4$RENPs, where RE^{3+}– Er^{3+}, Tm^{3+} and Ho^{3+}. Utilizing first nuclei stabilization synthesis approach, we have developed earlier, we can easily vary the concentration of dopants within these RENPs while preserving their overall size in the 20-30 nm range. By studying the upconversion spectra, lifetimes and quantum yield of individual bands, we showcase the full gamut of properties that can be fined tuned to the desired biomedical purpose.

9:30 AM

(ICACC-GYIF-026-2020) Luminescent lanthanide-doped nanomaterials as promising bioimaging probes (Invited)

E. Martinazzo Rodrigues*[1]; I. Halimi[1]; E. Hemmer[1]

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

Lanthanide-doped nanoparticles (Ln-NPs) are known to be versatile nanoplatforms for a wide range of applications from temperature sensors to luminescent or magnetic bioimaging probes. The possibility to be excited with near-infrared (NIR) light and the capability to emit in the visible and NIR spectral regions make them ideal candidates for luminescence-based bioimaging. The presence of Gd^{3+} ions adds the capacity to act as MRI contrast agent. The improvement of such performances is possible through deeper understanding of the relationships between structure (size, crystalline phase) and materials properties (luminescence, magnetic behavior). Therefore, our research aims to establish such relationships through the study of the luminescence behavior of Ln-NPs synthesized by a microwave-assisted thermal decomposition approach. Exploring the chemistry of different precursors, small-yet-bright Ln-NPs of controlled crystalline phase and size in the sub-10nm realm were easily obtained. Indeed, assessment of growth mechanisms revealed a strong dependence of the nanostructure on the type of precursor used, ultimately leading to the observed differences in structure and luminescence. Such investigations open the path towards the engineering of next-generation, more efficient Ln-NP-based bioimaging probes.

10:20 AM

(ICACC-GYIF-027-2020) Rare-earth-doped optical materials: Properties, limits and enhancement strategies (Invited)

F. Enrichi*[1]

1. Ca' Foscari University of Venice, Department of Molecular Sciences and Nanosystems, Italy

The spectroscopic properties of rare-earth-ions make them suitable for many optical applications. Their use is well established in phosphors for lighting, light amplifiers, lasers, spectral conversion layers, and optical biomarkers. However, major limitations are related to the small excitation cross section and absorption spectral bandwidth, which reduce their effective implementation and use. This presentation will be focused on the sensitizing process of rare earth ions by semiconductor or metal nanoaggregates. As examples, I will illustrate the role of silicon or silver nanoaggregates for broadband enhanced PL excitation in different rare earth doped materials, with potential applications in photovoltaics and lighting.

10:50 AM

(ICACC-GYIF-028-2020) Advanced bio/nanomaterials for point-of-care molecular detection in rapid sensory devices (Invited)

S. S. Mahshid*[1]

1. Sunnybrook Research Institute, University of Toronto, Biological Sciences Platform, Canada

Rapid point-of-care detection of various analytes in complex matrices could, in principle, impact many applications, including medical diagnostics, prognostics, and therapeutics. Over the past two decades, many molecular detection approaches have been explored among which, electrochemical biosensors have shown a lot of promises since they are known to be rapid, reagentless, and easily multiplexed. Given the fact that signal originates from the electron transfer, we engineer the sensor's surface by combining high-curvature nanostructured materials with biological recognition molecules to provide a direct analysis of clinical samples with appropriate target detection limits, and low levels of false negatives and false positives. These new biosensing devices provide multiplexing detection of proteins and small molecules at their therapeutic ranges for applications in disease diagnostics and therapeutic monitoring.

FS3: Molecular-level Processing and Chemical Engineering of Functional Materials

Simulation and Characterization of Polymer derived Ceramics

Room: Coquina Salon C
Session Chairs: Sanjay Mathur, University of Cologne; Emanuel Ionescu, Technical University Darmstadt

1:30 PM

(ICACC-FS3-001-2020) Simulations of Polymer Pyrolysis: Converting Polysiloxanes into Silicon Oxycarbide Ceramics (Invited)

P. Kroll*[1]; I. Ponomarev[1]

1. University of Texas, Arlington, USA

Polymer-derived ceramics are processed via thermal treatment of polymer precursors. Selecting the right precursor to obtain a material with desirable composition and properties is a crucial process. Open questions remain as to how precursor architecture and processing conditions affect the final material. Computational studies of this topic are scarce, not at least due to the complexity of the problem arising on different time and length scales. Here we present reactive force field (ReaxFF) simulations of the pyrolysis of polysiloxanesresulting in amorphous SiCO ceramics. We briefly illustrate the development process and establish the unprecedented accuracy of the force field, which allows simulations with a quality approaching that of quantum-chemical calculations. We show that simulations of systems extending several nanometer for several nanoseconds are currently feasible within ReaxFF. We then perform reactive molecular dynamics simulations to the pyrolysis of two distinct polysiloxanes. Results for PMHS cross-linked with DVB are in auspiciously good agreement to experiment: mass loss, density, and chemical composition of the resulting ceramic match observed data. A closer look into structure formation provides insight into the development of free carbon and its interface with the surrounding matrix. More examples pertain to the evolution of carbon structures in SiCO ceramics.

2:00 PM

(ICACC-FS3-002-2020) Porosity evolution during the pyrolytic conversion of preceramic polysilazane materials (Invited)

T. Konegger*[1]; C. Drechsel[1]; H. Peterlik[2]

1. TU Wien - Vienna University of Technology, Institute of Chemical Technologies and Analytics, Austria
2. University of Vienna, Faculty of Physics, Austria

Microporous ceramics open new potential fields for ceramic materials, e.g. in environment-related fields such as membrane-based separation. Polymer-derived ceramics such as polysilazane-derived silicon carbonitrides are highly promising in this regard, offering excellent high-temperature properties and chemical stability. Here, micro- and mesoporosity can be generated during the polymer-to-ceramic conversion process. However, for prospective applications, tailorable and well-defined pore structures under

anticipated operating conditions are crucial. This talk highlights the results of a project with the aims to elucidate mechanisms responsible for the evolution of microporosity in polysilazane-derived materials, and to identify pathways to control and tailor the resulting structures with respect to pore size as well as thermal and chemical stability. All steps in the processing chain from the precursor to the final ceramic material are critically and systematically evaluated to clarify the respective effects of precursor chemistry, cross-linking conditions, and pyrolysis conditions on the state of porosity in the final materials. Additionally, the findings are used to evaluate the general applicability of the micropore setup of SiCN(O) ceramics for prospective applications as high-temperature gas separation membrane materials.

2:30 PM

(ICACC-FS3-003-2020) Short- and Intermediate- Range Structure of Polymer Derived Ceramics: Results from Multi-Nuclear Solid-State NMR Spectroscopy (Invited)

S. Sen*[1]

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

Silicon oxycarbide (SiOC) and carbonitride (SiCN) polymer derived ceramics (PDCs) are technologically important high-temperature materials that have been studied extensively over the past decades. However, the structure-property relationships in these materials are not fully understood, primarily because the atomic structure at length scales beyond the nearest neighbors remain speculative at best. The atomic structures of SiOC and SiCN PDCs with different carbon contents are studied using multi-nuclear (^{29}Si, ^{13}C, ^{15}N), one- and two- dimensional solid state nuclear magnetic resonance (NMR) spectroscopic techniques. The results are found to provide unique and quantitative information regarding the relative concentrations of the SiC_xO_{4-x} and SiC_xN_{4-x} tetrahedral building blocks in these PDCs, their local symmetry in the bonding environment and mutual connectivity. The possible consequences of the connectivity of the coordination polyhedral in these PDCs on the packing of the resulting network and its thermodynamic stability will be discussed.

Silicon Carbide and Nitride based Polymer derived Ceramics

Room: Coquina Salon C

Session Chairs: Thomas Konegger, TU Wien - Vienna University of Technology; Sabyasachi Sen, University of California, Davis

3:20 PM

(ICACC-FS3-004-2020) SiC/C Composites from Molecular Precursors (Invited)

R. Bordia*[1]; S. Arreguin[2]; Y. Yang[2]; F. Ohuchi[2]; T. Konegger[3]

1. Clemson University, Materials Science and Engineering, USA
2. University of Washington, USA
3. TU Wien - Vienna University of Technology, Institute of Chemical Technologies and Analytics, Austria

SiC/C composites are potentially attractive for a variety of applications including structural elements in nuclear power plants, as electrodes in MHD energy conversion systems and as anodes for Li-ion batteries. We have processed these composites using molecular precursors for both SiC and C. Results will be presented to show the versatility of the PDC route, to tailor the composition, nanostructure and the properties of the resultant composites. The polytypes of the resultant SiC have been identified and a novel finding is that due to the molecular scale mixing of the carbon, the polytypes of SiC depend not only on the temperature but also on the carbon content. The electrical and surface electronic properties, as a function of carbon content and temperature, of the composites will be presented together with the mechanical properties. We gratefully acknowledge partial support from the US Department of Energy, National Energy Technology Lab under grant No. DE-FE0023142

3:50 PM

(ICACC-FS3-005-2020) A polymer blend method for designing improved silicon carbide based materials (Invited)

K. Kita*[1]; M. Fukushima[1]; H. Hyuga[1]; M. Hotta[1]

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

Polycarbosilane is one of promising precursor for making silicon carbide products with unique shapes such as fiber and membrane which can contribute to development and improving of ceramics matrix composites, gas separator, thermal barrier coating, etc. These products are usually manufactured through a process called "Precursor method" and a lot of papers have been reported how to improve these products. For improving these products, we paid attention to a polymer blend method that is blending polycarbosilane and a polymer which have good solubility with polycarbosilane. It was reported that some polymer blend methods succeeded in producing silicon carbide fibers which included unique characteristics, such as very fine fibers, fibers with very high yield, etc. In this study, mixtures of polycarbosilane and some polymers were prepared as a precursor polymer, in which transformed into silicon carbide fibers were produced by them. Finally, some silicon carbide fibers which included unique characteristics such as single hollow fibers could be obtained.

4:20 PM

(ICACC-FS3-006-2020) Synthesis of polymer-derived SiCNO ceramic aerogel and relative composites for electromagnetic absorption applications (Invited)

G. Shao*[1]

1. Zhengzhou University, School of Materials Science and Engineering, China

Electromagnetic absorption materials with "lightweight, broadband, strong absorption and thinness" are highly desired and crucial important for reducing Electromagnetic (EM) pollution and/or interference, caused by rapid development of wireless equipments, electronic devices and telecommunication, which is severely impacted the information security and health of humans. In this talk 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, with 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, and relative effective band width of 3.8 GHz, 6.6 GHz and 4.2GHz. Meanwhile, the SiC nanowire/SiCNO ceramic aerogel was synthesized by a in-situ reaction induced by microwave heating. The EM absorption property and formation mechanism was discussed.

4:50 PM

(ICACC-FS3-007-2020) Single-source-precursor synthesis and phase evolution of mesoporous VN/Si_3N_4 nanocomposites

E. Ionescu*[1]

1. Technical University Darmstadt, Materials Science, Germany

In the present work, a polymeric single-source precursor has been synthesized via chemical modification of a perhydro polysilazane (PHPS) with vanadyl acetylacetonate (i.e., $VO(acac)_2$) and converted into X-ray amorphous SiVN(O) upon ammonolysis at 1000 °C. The amorphous as-prepared SiVN(O) crystallizes at temperatures beyond 1400 °C and converts into a nanocomposite consisting of VN nanoparticles embedded in a polycrystalline β-Si_3N_4 matrix, showing a high temperature behavior similar to other Si-M-N systems (e.g., M = Hf, Ti, Fe). Moreover, mesoporous high-surface area SiVN(O) and VN/Si_3N_4 materials were prepared in the present work by using a blend of polystyrene and the $VO(acac)_2$-modified PHPS precursor. Upon adjusting the amount of polystyrene and

cross-linking conditions, mesoporous SiVN(O) with a surface area of > 500 m^2/g and a mesopores volume of 1.58 cm^3/g was obtained. Interestingly, a large fraction of the high surface area and large amount of mesoporosity from SiVN(O) was retained in the materials even after exposing it to temperatures up to 1400 °C. Thus, the resulting VN/Si_3N_4 exhibited a surface area of ca. 200 g/m^2 and a mesopores volume of ca. 1.29 cm^3/g. The obtained mesoporous Si-V-N(O)-based systems are expected to show promising catalytic performance in thermal dehydrogenation processes.

FS4: Green Technologies and Ceramic/ Carbon Reinforced Polymers

Mechanical Behavior or Ceramic/Carbon Reinforced Polymers and Composites I

Room: Halifax A/B

Session Chairs: Hiroshi Tsuda, National Institute of Advanced Industrial Science and Technology (AIST); Toshihira Irisawa, Nagoya University

1:30 PM

(ICACC-FS4-001-2020) Evaluation of Nonlinear Mechanical Behavior in Fiber Reinforced Laminated Composites (Invited)

S. Ogihara*[1]

1. Tokyo University of Science, Japan

Nonlinear mehanical behavior in Carbon Fiber Reinforced Plastic Compsite (CFRP) angle-ply laminates is experimentally investigated. Material used was Toray prepreg system, T700SC/2500, with 0.05mm thickness. The laminate configurations is $[(+-45)_{12}]_s$ which reulted in the total thickness of about 2.4mm. The specimen size was 280mm long including 50mm tab grip section for both ends and 20mm wide. Monotonic tensile test with different strain rates and stress relaxation test with incrasing strains are conducted. Loading-unloading cyclic tests were also performed. The laminates exhibited apparent nonlinearity in monotonic tensile loading and strain rate dependence, stress relaxation behavior. It was also shown that the material exhibits plasticity behavior and hysteresis behavior in loading-unloading tests. It was found that during the hysteresis, the equilibrium stress at a strain during unloading and loading seems different, which means that the equilibrium stress has hysteresis loops. Based on the observation above, it was implied that a viscoplasticity model with equilibrium stress which has hysteresis behavior is necessary.

2:00 PM

(ICACC-FS4-002-2020) Deformation Behavior of Continuous Carbon Fiber Reinforced PA6 during V-Shape Molding

S. Takemura*[1]; S. Kobayashi[2]; T. Osada[1]

1. Tokyo Metropolitan University, Mechanical Systems Engineering, Japan
2. Tokyo Metropolitan University, Mechanical Engineering, Japan

This paper investigates the deformation behavior of carbon fiber reinforced thermoplastics (CFRTP) during V-shape molding experimentally. Forming was conducted with a bending-type V-shape mold which has angle 90°. CFRTP studied was plain woven carbon fabric reinforced polyamide 6 (PA6). Pre-consolidated laminates were preheated above melting temperature of PA6 in a hot press machine, and then pressed after transferred to the V-shape mold. The effect of molding conditions, such as molding temperature, pre-heating temperature, molding load and volume fraction of fiber (Vf), were investigated. Also, in order to investigate the mechanical property of V-shape specimen, four point bending test was conducted. The test was carried out using similar testing tool to ASTM D 6415. As a result, in all conditions not including lower Vf condition, fiber breakage and misalignments were occurred. However, in the case of lower Vf, good specimens without fiber damages were obtained. This is because the interlayer slippage was occurred with increasing the amount of polymer in interlayer. The interlayer slippage, inhibited shear stress, which causes the fiber damages. For all lower Vf conditions, a final part angle smaller than mold angle, which is known as spring-in, was observed.

2:20 PM

(ICACC-FS4-003-2020) Residual Internal Pressure Strength Evaluation of Composite Pipe Subjected to Out-of-Plane Impact Loading using Finite Element Method

S. Fukumoto*[1]; S. Kobayashi[2]; T. Osada[1]

1. Tokyo Metropolitan University, Mechanical System Engineering, Japan
2. Tokyo Metropolitan University, Mechanical Engineering, Japan

The pressure vessels mounted on fuel cell vehicles are required to have high pressure resistance and to be light weight in order to increase the cruising distance. Therefore, in recent years the use of composite pressure vessels has been expanded. Carbon fiber reinforced plastics (CFRP) used in the composite pressure vessel are excellent in specific strength, specific rigidity, and fatigue resistance. On the other hand, CFRP is easily damaged by out-of-plane impact loading. It is also known that the burst pressure of the composite pressure vessel is reduced by the out-of-plane impact damage. Therefore, the internal pressure test after impact on the vessel is required as a design verification test. Furthermore, many tests on the expensive composite vessels are required to satisfy the design requirements which include to minimize the amount of the expensive materials, such as CFRP. In this research, the effect of curvature on the stress distribution of the CFRP/aluminum composite plate was investigated to establish a simple evaluation method of residual internal pressure strength after impact of the composite vessel experiments using a composite plate. Stress distribution in composite pipes and hybrid plates were calculated with finite element method. Impact tests were also conducted on the composite pipes and the hybrid plates.

2:40 PM

(ICACC-FS4-004-2020) Self-deployment force of CFRP bistable open sectional partial cylindrical beam

S. Kajihara*[1]; T. Aoki[1]

1. University of Tokyo, Aeronautics and Astronautics, Japan

Carbon Fiber Reinforced Plastic (CFRP) which has high specific strength and high specific stiffness is widely used in aerospace structures. It is well known that CFRP laminate formed into open sectional partial cylindrical shape can be designed to have bistability which means having two different stable states: coiled state and deployed state. This bistable beam can transform from the coiled state to the deployed state without applying external force and becomes beam structure that can sustain axial force in the deployed configuration. By utilizing these properties, the CFRP bistable beam is expected to be applied as the components of deployable space structures. However, the self-deployment is not well quantitatively predicted. There are few papers researching the mechanism of self-deploying behavior and the quantitative evaluation is not enough neither numerically or experimentally. In this study, numerical analysis and experiment were performed and the self-deployment force which can be directly calculated from static equilibrium was investigated. In addition, analytical model was made and a series of the transitional configuration from the coiled shape to the deployed shape was simulated. As a result, it was suggested that the self-deployment force can accurately be derived from the strain energy in the coiled state.

Mechanical Behavior or Ceramic/Carbon Reinforced Polymers and Composites II

Room: Halifax A/B
Session Chairs: Henry Colorado, Universidad de Antioquia; Satoshi Kobayashi, Tokyo Metropolitan University

3:20 PM

(ICACC-FS4-005-2020) Effect of Crystal State on Static Bending Poperties of Carbon Fiber Reinforced Polyamide 6 (Invited)

T. Sakai*[1]; R. Fukushima[1]; N. B. Shamsudim[1]; K. Kageyama[1]

1. Saitama University, Japan

CFRTP (Carbon fiber reinforced thermoplastics) are consisted with the carbon fiber and thermoplastics such as PP, PA6, PI, and PC and so on. Mechanical properties of CFRTP should be depended on the matrix resin, therefore, if the mechanical properties of matrix resin has changed, CFRTP's properties will be also changed. In this study, carbon fiber reinforced polyamide 6 was used for evaluate the effect of change in the crystal state. The materials were heat treated for crystallization at crystallization temperature, and the crystallinity was controlled by heat treatment time. As the result of static bending tests, bending strength was increased with the increase in the crystallinity. For bending modulus, the value was increased at the first heat treatment for 1 hour, however, the modulus with longer heat treatment was decreased with the increase in the heat treatment. To understand the reason of these bending properties changed by the crystal state, DSC analysis was carried out. As the results of DSC analysis, the peak shape of melting point was changed with heat treatment for 1 hour and 30 hour. The double peak was observed with over 1 hour heat treatment, and over 30 hours heat treatment made the melting temperature more lower. This is because the bending modulus of CFRTP was dominated by the peak shape and peak area of the melting point.

3:50 PM

(ICACC-FS4-006-2020) Influence of Ceramic Architecture and Loading Orientation on Compressive Response of Ice-templated Hierarchical Ceramic-polymer Composites (Invited)

D. Ghosh*[1]; S. Akurati[1]; J. Marin[1]

1. Old Dominion University, Mechanical and Aerospace Engineering, USA

Ice-templated composites exhibit hierarchical architecture which resemble to the architecture observed in nacre and the significant interest is due to the synergy of strength and damage-tolerance that can be achieved in engineering materials. Ice-templating is an environment-friendly processing technique in which phase separation of solvent crystals and ceramic particles followed by freeze drying and sintering result in ceramic foams with directional porosity. Due to low pore tortuosity, polymer infiltration of ice-templated ceramic foams enables to develop novel hierarchical ceramic-polymer (strong-tough) composites. However, little attention is given on strength and deformation behavior under uniaxial compression. For compressive loading orientation parallel to ceramic and polymer layers, it is expected that the material response will be influenced by the brittle ceramic phase. Whereas, for compressive loading orientation perpendicular to the layers, ductile polymer phase will have significant influence. In this presentation, we will discuss the effects of composition (ceramic to polymer ratio), architecture of ice-templated sintered ceramic foam, and loading orientation on uniaxial compressive strength and deformation behavior to shed light on the structure-property relationships in ice-templated ceramic-polymer composites.

4:20 PM

(ICACC-FS4-007-2020) Tensile Behavior of Polymeric Composites Reinforced by Jute Fabric for Piping Repair

M. O. Moreira[1]; K. Silva[1]; R. G. Almeida[1]; F. P. Lopes*[1]; E. A. Carvalho[1]; C. F. Vieira[2]

1. State University of Northern Rio de Janeiro, Advanced Materials Laboratory, Brazil
2. State University of the North Fluminense, Advanced Materials Laboratory, Brazil

Composite repairs are becoming widely used as an alternative in the energy industries, instead of repairs welded, full-encirclement sleeves for oil and gas transmission/transportation pipelines. The primary advantage of this repair type is that the execution is considered a cold work, making it possible to perform in classified zones for potential explosions. However, nowadays polymeric composite repairs are not fabricated using natural fibers, which are economical and sustainable. The present paper is concerned with the analysis of jute fabric reinforcing epoxy resin in composites repair systems for metallic piping with localized corrosion damage that impair the serviceability. The main motivation for the study presented in this paper, it is the sustainable rehabilitation of corroded pipelines in offshore platforms. According to ASTM D3039, tensile tests were performed to analyze the mechanical improvement of jute fabric into polymeric composites compared to pure epoxy resin. Scanning Electron Microscopy were carried out to reveal the fracture surface and the interaction between the jute fabric and the epoxy resin. The results demonstrated the strong potential benefits of using this new eco-friendly composite as a piping repair.

4:40 PM

(ICACC-FS4-008-2020) Strain/displacement distribution measurement technique using grid patterns and its application to CFRP (Invited)

H. Tsuda*[1]; S. Ri[1]; Q. Wang[1]; P. Xia[1]

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

Strain gauges and extensometers have been commonly employed to measure strain and displacement. Distribution measurement of strain or displacement from these conventional sensors, however, is difficult because they are single-point measurement sensors. A full-field measurement utilizing images allows to obtain such a distributional information. The full-field measurement method can be classified into two types: digital image correlation method using speckle patterns and Moiré method using grid patterns. The digital image correlation method tends to be applied to measurement for relatively small-scale structures or materials because painting of speckle pattern to large area is technically difficult. On the other hand, the Moiré method has the advantage that this can be applied to any scale of structures and materials. This study introduces the strain/displacement measurement technology based on the Moiré method as well as its application to CFRP: microscale strain distribution of CFRP under 3-point bending, evaluation of a stress-strain curve under tensile loading of CFRP, and displacement distribution measurement of a filament-wound CFRP pressure vessel under verification test.

5:10 PM

(ICACC-FS4-009-2020) Evaluation of Interfacial and Mechanical Properties of Various Thermally-Recycled Carbon Fibers/ Recycled PET Composites

J. Park*[1]; Y. Baek[1]; P. Shin[1]; J. Kim[1]; L. K. DeVries[2]

1. Gyeongsang National University, Materials Eng. & Convergence Technology, Republic of Korea
2. The University of Utah, Mechanical Engineering, USA

The mechanical and interfacial properties were evaluated for carbon fiber reinforced composites (CFRC) manufactured using thermally recycled waste carbon fiber and recycled polyethylene terephthalate

(PET). The mechanical properties of the recycled fiber were determined and compared to those of neat fibers using the single-fiber tensile test. The surfaces of the recycled and neat carbon fiber were examined and compared using FE-SEM and dynamic contact angle measurements. A goal of the study was to determine the applicability of industrial use of recycled CF and/or recycled PET in CFRC. Mechanical properties were measured using short beam and tensile tests. These properties were observed to be correlated with crystallinity. The interfacial properties between the recycled carbon fibers and recycled PET were evaluated using the microdroplet test. At low temperature residual resin remained on the recycled CFs surface resulting relatively the low interfacial properties. At excessively high temperatures, oxidation occurred, on the CFs surface, which also resulting in relatively poor low mechanical properties. The optimal treatment condition was 500 oC, where the surface was relatively clean and the reduction in mechanical properties was minimized.

S1: Mechanical Behavior and Performance of Ceramics & Composites

Fracture Mechanics and Failure Prediction I

Room: Coquina Salon D
Session Chairs: Jonathan Salem, NASA Glenn Research Center; Jon Mackey, University of Akron

8:30 AM

(ICACC-S1-029-2020) Additive manufacturing of Silicon Nitride: First results and way forward

S. Behar Lafenetre*[1]; P. Grasset[1]; L. Cornillon[1]; N. Louh[1]; M. Villemaire[1]; C. Schick[2]; C. Chaput[2]; N. Rousselet[2]; F. Gant[3]

1. Thales Alenia Space, France
2. 3DCeram, France
3. CNES, France

Silicon nitride has very interesting properties for space applications, especially for optical instruments and telescopes. The objective for such structures is chiefly stability to ensure performance, low mass and high stiffness to sustain launch loads. With its high strength and stiffness, low CTE and high toughness, Silicon Nitride is therefore extremely well suited for stable Space structures. Thales Alenia Space has been using Si3N4 for more than a decade, and in that framework has evaluated the ability of the material to address those needs and requirements for complex designs such as tubes, brackets, brazed supports, beams and lightweighted plates. All these structures have been tested, qualified and are now flight proven. In order to further enhance the manufacturing process, save raw material, and access more complex shapes that are not feasible with classical manufacturing techniques, 3DCeram, which has the mastery and know-how of the 3D printing process of technical ceramics, appeared as the right partner for Thales Alenia Space. In the past years, with the financial support of CNES and Thales Alenia Space, 3DCeram has developed a dedicated formulation for Silicon Nitride by additive manufacturing and has shown promising results. Applications as different as structural elements or mirrors are targeted and first mock-ups have been built. This presentation proposes a status of this novel technology.

8:50 AM

(ICACC-S1-030-2020) Failure strength of silicon nitride for space applications

S. Behar Lafenetre*[1]; N. Louh[1]; L. Cornillon[1]; P. Grasset[1]; M. Such-Taboada[2]

1. Thales Alenia Space, France
2. ESA, Netherlands

With its high strength and stiffness and low CTE and high toughness, Silicon Nitride is extremely well suited for stable Space structures. Thales Alenia Space has been using Silicon Nitride for more than a decade, and in that framework has evaluated the ability of the material to address those needs and requirements for complex designs such as tubes, brackets, brazed supports, beams and lightweighted plates. All these structures have been tested, qualified and are now flight proven. In order to improve knowledge and mastering of strength properties, a characterization campaign of the material has been implemented in a study funded by CNES and driven by ESA. This paper describes the design of this test campaign, the choices for the sample types and dimensions, and prediction of the expected results. In addition to the mechanical strength testing, X-ray tomography has been implemented in order to detect flaws beforehand and to investigate the ability to predict failure from the extracted information. This will be especially useful since verification (in particular proof tests that are commonly used on ceramics for Space applications) is based on the relationship between strength and flaws. It is expected that by improving this knowledge a more straightforward verification process can be derived. Final results of the study will be discussed.

9:10 AM

(ICACC-S1-031-2020) Design of damage tolerant and crack-free layered ceramics with textured microstructure

R. Bermejo*[1]; A. Hofer[1]; R. L. Walton[4]; O. Ševeček[3]; G. L. Messing[2]

1. MontanUniversityet Leoben, Materials Science, Austria
2. Pennsylvania State University, USA
3. Brno University of Technology, Czechia
4. Pennsylvania State University, Materials Science and Engineering, USA

Damage tolerant ceramics can be fabricated by combining materials with different microstructures and properties in a layered architecture. One strategy is to induce residual compressive stresses in the embedded layers after sintering in order to shield crack propagation. These embedded layers may be distributed either in a periodic or non-periodic manner. A limiting factor is that the beneficial compressive stress and layers develop tensile stresses in adjacent layers. This work demonstrates the design of damage tolerant and crack-free textured ceramic laminates. Non-periodic architectures were designed by embedding 2 textured alumina (TA) layers between 3 equiaxed alumina-zirconia (AZ) layers. Compressive residual stresses of ~250 MPa were induced in the textured layers. Results from indentation strength tests in non-periodic architectures were compared to periodic architectures with same volume ratio of TA and AZ materials. Crack propagation was arrested in both periodic and non-periodic designs, the minimum threshold-strength being higher in the latter. Non-periodic architectures with compressive layers as thin as ~200 μm showed no evidence of surface edge cracks, yet still reached minimum threshold strength values of ~300 MPa. In addition, the textured microstructure promoted crack bifurcation in the thin compressive layers and thus enhanced the damage tolerance of the material.

9:30 AM

(ICACC-S1-032-2020) Fracture mechanics of dense and porous repeated laminate-ceramics

S. Ikari*[1]; W. Nakao[1]; S. Kawai[2]; T. Sawada[2]

1. Yokohama National University, Graduate school of Engineering, Japan
2. LIXIL, Japan

The dense and porous laminated structure is a new structure that suppresses brittle fracture of ceramics and is expected to improve mechanical properties. The difference from the conventional composite material is that a weak structure is introduced into the material. In this structure, the difference of Young's modulus in the material controls the fracture of the material. We investigated the characteristics of this structure using pottery, which has a low strength among ceramics. In the three-point bending test, we have demonstrated the superior mechanical properties of this structure. Specifically, the maximum strength was improved by 21% and the fracture energy was improved by 35%, compared to the ceramics with

only dense material. Furthermore, the crack growth behavior was analyzed using direct observation with a high-speed camera and the Didital Image Correlation method (DIC method). In addition, the microscopic breakdown inside the material was analyzed using the Acoustic Emission method (AE method). As a result, it has been clarified that ceramics with a dense and porous laminated structure cause peculiar destruction.

10:10 AM

(ICACC-S1-033-2020) R-Curve Measurements of PZT as a Function of Poling Direction

K. T. Strong*[1]; S. Grutzik[2]; C. Diantonio[3]; T. Diebold[1]; T. Chavez[3]

1. Sandia National Laboratories, Material Mechanics and Tribology, USA
2. Sandia National Laboratories, Component Science & Mechanics, USA
3. Sandia National Laboratories, USA

R-curves were measured for ferroelectric lead zirconate titanate (PZT) using Single Edge V-Notch Beams (SEVNB) with notch radii of less than 10μm. A rigid 4 pt. bend fixture that utilizes a piezo-actuator to drive displacement and a pizeo load cell and LVDT to collect load displacement data. Compliance calculations were carried out to determine crack extension and calculation of the stress intensity vs. crack extension curve. Notched samples were electrically biased parallel or perpendicular to notch orientation. Results will be presented and discussed.

10:30 AM

(ICACC-S1-034-2020) Evaluation of competitive model between self-healing and crack propagation in fiber reinforced self-healing ceramics

W. Nakao*[1]

1. Yokohama National University, Japan

In this study, the quantitative evaluation method of competitive relationship between self-healing and crack propagation in fiber reinforced self-healing ceramics was proposed. Furthermore, the competitive model was constructed as a function of temperature and creep test of this model were demonstrated. For this purpose, by using typical self-healing material, which is alumina fiber-reinforced alumina composite having silicon carbide interlayer, the threshold stress was experimentally determined. Investigating the elongation behavior under arbitrary constant stress and active condition of self-healing, crack arrest time and suppression limit time of crack propagation were determined the obtained elongation curves. From these values of the times, the threshold stress for self-healing in the specimen was determined to be 119 MPa at 1100°C. The similar experiment was conducted at 1000°C. It is indispensable to confirm the temperature dependence of the competitive relationship and demonstrate that it is possible to determine the threshold stress for self-healing in order to apply the competitive model to actual material design and further to use the self-healing material as structural material.

10:50 AM

(ICACC-S1-035-2020) On Terminal Crack Velocities in Glasses and Ceramics

G. D. Quinn*[1]

1. NIST, USA

A review of the literature shows that terminal crack velocity, v_{ct}, of glasses should be correlated with the longitudinal elastic wave velocity, and the structure and density. Theoretical predictions of v_{ct} by the Mott-Roberts-Wells model match experimental measurements for some key glasses, including fused silica and low-density borosilicates. It is suggested that the slower v_{ct} results obtained for many other glasses are due to structural variations that cause crack front perturbations. Notions that micro or nano crack nucleation ahead of the fast moving crack tip lead to limits on the crack velocity are in error. These conclusions are most likely applicable to ceramics as well.

11:10 AM

(ICACC-S1-036-2020) Zerodur® strength behavior for space applications

S. Behar Lafenetre*[1]; L. Cornillon[1]; P. Grasset[1]; F. Gant[2]

1. Thales Alenia Space, France
2. CNES, France

Zerodur® is a well-known glass-ceramic used for optical components because of its unequalled dimensional stability under thermal environment. In particular it has been used since decades in Thales Alenia Space's optical payloads for space telescopes, especially for mirrors. As performance of space telescopes is increasing, the size of mirrors increases accordingly, and an optimization of the design is necessary, mainly for mass saving. Therefore the question of the effective strength of Zerodur® has become a real issue. Thales Alenia Space has investigated the application of the Weibull law and associated size effects on Zerodur® in 2014, under CNES funding, to accurately determine the parameters of the Weibull law for Zerodur® when machined in the same conditions as mirrors. The applicability of the 2-parameter and 3-parameter Weibull laws (with threshold strength) have been investigated. Additionally, another strength specificity of Zerodur® was related to sub-critical crack growth before launch. A dedicated test campaign has been implemented over three years, and the results show no effect as expected for the specific application of space optics. This presentation will summarize the results of those test campaigns and confirm that Zerodur® is the best material for ultra-stable Space mirrors.

11:30 AM

(ICACC-S1-037-2020) Direct In Situ Observation of Toughening and Fatigue Behavior in Alumina/Graphene Nanocomposites

Q. Wang*[1]; C. Ramirez[1]; N. P. Padture[1]

1. Brown University, School of Engineering, USA

Despite having increased fracture toughness, conventional ceramic composites toughened by fibers or whiskers have poor cyclic fatigue resistance due to the degradation of the crack-wake bridging in cyclic loading. Graphene, known for its exceptionally high strength and stiffness, is a promising reinforcement material for improving cyclic fatigue properties of advanced ceramics. Recently, many studies have reported that polymers and ceramics containing one-dimensional or two-dimensional carbon nanofillers, such as carbon nanotubes and graphene nanoplatelets, show significantly improved cyclic fatigue resistance. This phenomenon has been explained in terms of toughening mechanism induced by these nanofillers and most of the studies focused on the optimal nanofiller concentration for the max fatigue resistance, however, direct evidence of the processes happening in the carbon fillers under cyclic loading is missing from the discussion. In this work we have performed in situ cyclic loading test of spark plasma sintered graphene alumina composites, achieving stable crack growth for flat punch compression of notched samples and obtaining continuous observation of graphene platelets deformation during cyclic loading. Information extracted accompanied by the Raman spectroscopy studies has permitted to have a deeper understanding of the fatigue resistance mechanisms in this novel material.

Fibers and Coatings

Room: Coquina Salon D
Session Chair: Amjad Almansour, NASA Glenn Research Center

1:30 PM

(ICACC-S1-038-2020) Rare-earth disilicate fiber coatings for SiC/SiC CMCs

E. E. Boakye*[1]; P. Mogilevsky[1]; T. Key[1]; T. A. Parthasarathy[1]; M. Cinibulk[2]; R. Hay[2]; S. Opeka[1]; R. Corns[1]

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Rare-earth disilicates ($RE_2Si_2O_7$) are potential oxidation-resistant alternatives to carbon or BN fiber coatings for SiC/SiC CMCs. Our prior work experimentally demonstrated that rare earth disilicates may work as a weak interface in fiber-reinforced SiC/SiC CMCs. However, the effect of oxidation, especially in water vapor, on their functionality as a weak interface has not yet been tested. In this work, SiC/SiC minicomposites with $Y_2Si_2O_7$ interface were exposed to steam and tensile tested. The effect of steam on the functionality of the $Y_2Si_2O_7$ interface coatings was studied and will be reported.

1:50 PM

(ICACC-S1-039-2020) Experimental Characterization of Elastic Stiffness and Delamination Toughness in Commercial Thermal Barrier Coating Systems

J. Alidoost*[1]; K. J. Hemker[1]

1. Johns Hopkins University, Mechanical Engineering, USA

Layered thermal barrier coating (TBC) systems used in jet engines consist of a nickel-based superalloy substrate, intermetallic bond coat, thermally grown oxide (TGO) and an electron beam physical vapor deposition (EBPVD) yttria-stabilized zirconia (7YSZ) top coat. Thermal protection is provided when the TBC remains attached to the substrate, and mechanism-based lifetime assessment models rely on accurate knowledge of the experimentally measured interfacial fracture toughness and topcoat modulus. Employing a compression edge-delamination (CED) methodology allows for direct measurement of mode-II interfacial toughness of ceramic coatings. The role of mode mix will be emphasized and mode-II delamination experiments of CED specimens extracted from laboratory coupons and engine hardware will be presented. The elastic response of the top coat is critical in the analysis of the CED experiments, and microbending experiments that allow us to measure the elastic modulus of EBPVD YSZ top coats after various stages of thermal cycling will also be discussed.

2:10 PM

(ICACC-S1-040-2020) Effects of Boria on Rare Earth Silicate Environmental Barrier Coatings

R. Guarriello*[1]; E. J. Opila[1]

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

SiC-based ceramic matrix composites (CMCs) have recently come into use for hot section components of turbine engines. These CMCs are comprised of SiC fibers surrounded by a BN interphase, in a SiC matrix. The BN interphase oxidizes to form boria (B_2O_3) on the surface of the CMC. Boria is a strong glass-former with other oxides such as the ytterbium disilicate (YbDS) currently used as an environmental barrier coating (EBC). Little is known about phase equilibria and reactions in the Yb_2O_3-SiO_2-B_2O_3 system. One of the experiments utilized for this study is an idealized interface test between the oxide constituents. Wells are drilled into phase pure samples of the YbDS, and then filled with plugs of pure boria glass. Samples are exposed in air at temperatures up to 1200C for up to 24 hours. Scanning electron microscopy and energy dispersive spectroscopy are used to examine the reactions zones and glass left after the exposures. The remaining glass is digested off the substrate using deionized water and analyzed using inductively coupled plasma-optical emission spectroscopy to measure the amount of dissolved silicon and ytterbium. XRD is used to determine the phases resulting from the SiO_2 and Yb_2O_3 depleted YbDS. The reactions and products observed will inform both phase equilibria in the Yb_2O_3-SiO_2-B_2O_3 system and interactions between the EBCs and boria formed during CMC oxidation.

2:30 PM

(ICACC-S1-041-2020) Single Fiber Creep Performance of Silicon Carbide Fiber Materials

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1. Free Form Fibers, USA

Single fiber creep behavior is a critical area of understanding for the application of ceramic matrix composites (CMCs) in high tech fields such as aviation, aerospace, and nuclear power generation. Free Form Fibers (FFF) has designed and assembled a single fiber creep test system, as well as developed a test methodology for evaluating the high temperature performance of ceramic fibers, in particular for silicon carbide fiber materials as part of SiC-SiC CMCs. Results of creep testing on FFF's LP30-SC SiC fiber as well as Hi Nicalon-Type S are presented across a range of hold temperatures, applied loads, and long-time durations in air (oxygen exposure) and inert (argon) test environments. Post-creep test analysis, including microscopy and residual tensile strength, is also reported.

Fracture Mechanics and Failure Prediction II

Room: Coquina Salon D
Session Chair: Emmanuel Boakye, UES Inc.

3:10 PM

(ICACC-S1-042-2020) Micromechanical Characterization of Damage Evolution in CMCs

B. Swaminathan*[1]; N. R. McCarthy[5]; A. S. Almansour[2]; K. M. Sevener[3]; J. D. Kiser[4]; S. Daly[5]

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2. NASA Glenn Research Center, Mechanical Engineering, USA
3. University of Michigan, Materials Science and Engineering Dept., USA
4. NASA Glenn Research Center, Ceramic and Polymer Composites, USA
5. University of California, Santa Barbara, Mechanical Engineering, USA

Silicon carbide / silicon carbide ceramic matrix composites (SiC/SiC CMCs) are structural ceramics characterized by their low weight, high specific strength, creep resistance, and damage tolerance. In order to accurately life CMCs, it is critical to understand the influence of the constituent landscape (e.g. porosity, constituent properties, geometric and structural variations, and microstructural interactions) on the initiation and accumulation of damage. In this research, an experimental approach combining acoustic emission (AE) with in-SEM (scanning electron microscope) tensile testing was used to examine early microscale damage initiation (i.e. below the proportional limit) and its progression to failure in SiC/SiC minicomposites at the surface and subsurface. SEM-observed damage was correlated to its corresponding AE events, in order to assess the relationships between AE signature characteristics and damage mechanisms such as matrix cracking, interfacial debonding, and fiber breakage. Fiber push-in testing was used to examine interfacial properties. The analysis of microscale damage accumulation was used to assess the applicability of micromechanical models to the observed damage behavior, considering parameters such as the stress-dependent crack opening displacements, and the crack spacing and density.

3:30 PM

(ICACC-S1-043-2020) Use of Acoustic Emission to Assess Damage in EBC-CMC Systems

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2. NASA Glenn Research Center, Environmental Effects and Coatings Branch, USA
3. University of Toledo at NASA Glenn Research Center, Environmental Effects & Coatings Branch, USA
4. Vantage Partners, LLC at NASA Glenn Research Center, Ceramic & Polymer Composites Branch, USA
5. Boise State University at NASA Glenn Research Center, Ceramic & Polymer Composites Branch, USA

SiC fiber-reinforced SiC ceramic matrix composites (SiC_f/SiC) react with oxygen at high temperatures forming a protective silica layer. However, this silica layer reacts with water vapor to form volatile silicon hydroxide ($Si(OH)_4$) which degrades the composite. Therefore, environmental barrier coatings (EBCs) are needed for ceramic matrix composite (CMC) components that are being used in water vapor-rich jet engine environments in order to provide elevated temperature environmental stability and achieve protection against oxidation. In this work, several layers of oxide-based environmental barrier coatings were applied on Hi-Nicalon Type S SiC fiber-reinforced chemical vapor infiltrated (CVI) SiC minicomposites via a slurry-based infiltration approach. Minicomposites were then tested in monotonic tension at room temperature. Acoustic emission damage monitoring technique was used to assess surface and subsurface damage mechanisms in the EBC-CMC system, respectively. EBC/CMC damage onset, progression, and interaction through-thickness and at the matrix/coating interface were characterized and compared to that of the uncoated, pristine CMC.

3:50 PM

(ICACC-S1-044-2020) Experimental and analytical studies on damage evolution behavior of an orthogonal 3-D woven SiC fiber/SiC matrix composite under tensile loading

K. Hachisu*[1]; Y. Ikarashi[1]; T. Ogasawara[1]; T. Aoki[2]

1. Tokyo University of Agriculture and Technology, Japan
2. Japan Aerospace Exploration Agency, Advanced Composite Research Center, Institute of Aeronautical Technology, Japan

This study examined the microscopic damage mechanisms of an orthogonal 3-D woven SiC fiber (Tyranno ZMI) / SiC matrix (CVI+PIP-SiC) composite under monotonic tensile loading at room temperature. The micro-crack propagation behaviors were evaluated by in-situ observation using an optical microscope and digital image correlation technique. The effect of matrix crack propagation in the transverse (90°) and longitudinal (0°) fiber bundles on the stress-strain response was numerically estimated using finite element analysis (FEA). A unit cell model of the orthogonal 3-D woven architecture was modeled in detail. Transverse cracks in the 90° fiber bundles were introduced directly into the unit cell model. The stiffness degradation caused by matrix crack propagation in the 0° fiber bundles was represented using continuum damage mechanics model. A computer code based on homogenization method was developed incorporating the damage mechanics model into the finite element analysis code. The stress-strain curves estimated by FEA showed good agreed with the experimental results.

4:10 PM

(ICACC-S1-045-2020) Advanced Techniques for Design and Analysis of Composite Structures

T. Douglas*[1]

1. Wasatch Composite Analysis, USA

As the benefits of advanced composite materials become known and used by many industries, outside of aerospace, there is an increasing need for fast and accurate design and analysis of these new composite structures. Since often times these composite structures are used in cost critical commercial applications, which require faster design engineering, materials development, and manufacturing with shorter delivery schedules. Due to cost and time constraints, full scale testing is usually not an option for these commercial applications. Therefore, there is an urgent need for experienced composite D&A engineers with an extensive background in composite materials and structures. These engineers relay on many advanced techniques and tools to accelerate the composite design and engineering process.

4:30 PM

(ICACC-S1-046-2020) Failure Analysis of Brittle Single Crystals

J. Salem*[1]

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

Glasses and ceramics in the form of polycrystals and single crystals are used in NASA applications ranging from windows to lenses to centrally-heated laser slabs. Despite standard procedures to design such components, unexpected failures occasionally occur. Examples of NASA mission using glasses and ceramics will discussed along with examples of failures. Emphasis will be placed on the ICESat-2 mission and a solar concentrator. The ICESat-2 (Ice, Cloud, and Elevation Satellite-2) Mission employs yttrium orthovanadate single crystals in laser amplifiers and oscillators. Although the systems are currently flying and successfully operating, some unexpected crystal fractures occurred just prior to flight. The failures were traced to poor crystal quality along with time dependent chemical reactions within the system assembly that increased stress beyond expectations and promoted both fast fracture and stress corrosion. This presentation will discuss failure analysis of the crystals and the chemical reactions that promoted failures, along with corrective actions taken to reduce likelihood of fracture over the three year mission. Re-designed lasers were stored for 12 months and then operated for a total of 1,000 hours without signs of degradation.

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

Environmental and Thermal Barrier Coatings III

Room: Ponce de Leon

Session Chair: Douglas Wolfe, Pennsylvania State University

8:30 AM

(ICACC-S2-031-2020) Effects of Substrate on the Steam Oxidation Behavior of Modified Si/$Yb_2Si_2O_7$ Environmental Barrier Coatings (Invited)

K. Lee*[1]

1. NASA Glenn Research Center, USA

Increased fuel efficiency of gas turbines is obtained through higher thermal efficiency by increasing the overall pressure ratio (OPR). Increased OPR requires higher turbine inlet temperature, which is paced by advances in turbine hot section materials. SiC/SiC Ceramic Matrix Composites (CMCs) are the most promising materials to enable a quantum leap forward in temperature capability compared

to current superalloys. Environmental Barrier Coatings (EBCs) are an enabling technology for CMCs by protecting them from water vapor-induced recession. Spallation of EBC induced by thermally grown oxide (TGO) resulting from steam oxidation is the most critical EBC failure mode. A study was undertaken to investigate the effect of SiC substrate on steam oxidation kinetics of modified $Si/Yb_2Si_2O_7$ EBC. Silicon carbide substrates investigated in this study are CVI (chemical vapor infiltrated) CMC, two MI (melt infiltrated) CMCs with different pedigree, and sintered SiC. Oxidation kinetics were determined using atmospheric pressure steam cycling test in 90% H_2O + 10% O_2. Correlations between oxidation kinetics and substrate chemistry, coupled with chemical analysis of TGO and EBC, are used to explain the effects of CMC substrate on the oxidation behavior of modified $Si/Yb_2Si_2O_7$ EBC.

9:00 AM

(ICACC-S2-032-2020) Comparative Analysis of Furnace Cycle Durability of Thermal Barrier Coatings Applied on Flat and Cylindrical Geometries

E. J. Gildersleeve*[1]; S. Sampath[2]

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

The characteristics of an air plasma sprayed (APS) ceramic thermal barrier coating (TBC) can show a wide range of variability when applying processes optimized for model planar surfaces (i.e. buttons, plates) to complex geometries (i.e. rods, turbine blades). In particular, the differences in microstructure for identical processing parameters are observed when comparing the coating on a flat specimen to one on a cylinder. The mechanical property and performance of the cylindrical TBC differed significantly as well. In the case of flat substrate TBCs, it has been well-documented for Furnace Cycle Testing that by increasing the density of the TBC, the resistance to failure concurrently increases through a combination of toughness and compliance benefits. Results suggest when applying coatings with flat coupon processing parameters onto cylindrical superalloy samples, the FCT durability of the TBC is substantially reduced (< 10% of the life of a flat disk TBC). Furthermore, increasing TBC density to augment the resistance to delamination was not effective. In this study, the different thermomechanical stresses in a cylindrical TBC system were analyzed as compared to a superalloy disk/bond coat/TBC system to provide insights on the differences in failure response. Concurrently, studies were conducted on the influence of TBC microstructure and bond coat type.

9:20 AM

(ICACC-S2-033-2020) Laser Thermal Gradient Testing of Multilayer, Multimaterial Thermal Barrier Coatings

Y. Wu[2]; P. Hsu*[1]; M. H. McCay[2]; E. Croy[2]; Y. Wang[1]; E. J. Gildersleeve[3]; S. Sampath[4]

1. Florida Institute of Technology, Mechanical Engineering, USA
2. Florida Institute of Technology, Center for Advanced Coatings, USA
3. Stony Brook University, Materials Science, USA
4. Stony Brook University, Center for Thermal Spray Research, USA

Three different thermal barrier coatings are tested under the cyclic, laser heating and form a nearly one-dimensional temperature gradient from the top coat to the substrate. A unique laser scanning pattern was developed providing a nearly uniform temperature distribution in the heating surface of the top coat. Each test specimen is a 25.4 mm diameter button with about 3.5 mm overall thickness. The top coat materials are: (1) Porous 7YSZ; (2) Bi-Layer YSZ in which a dense layer of 7YSZ is sprayed followed by thicker porous 7YSZ; and (3) Multilayer YSZ/GZO with a dense 7YSZ at the bond coat/top coat interface, porous 7YSZ as the interlayer, and DVC GZO at the surface. These coatings were applied by air plasma spray. Topcoats were applied on a HVOF coated CoNiCrAlYHfSi bondcoat on a Hastelloy X substrate. During the testing, the heated side is maintained at about 1300°C with compressed air cooling on the bottom side to achieve a temperature of 1000°C for buttons (2) and (3). In the porous 7YSZ case, the top/bottom side temperatures are 1240/1130°C. In each 27 minutes of cycle, the heating dwell time is 20 min and the heating off time is 7 min. The porous 7YSZ case is tested for about 7.75 hr and the other coatings are tested for 50 hr each. Ongoing work examines the relative thermal performance and microstructural changes to the various coatings exposed to this thermal gradient condition.

9:40 AM

(ICACC-S2-034-2020) A Parametric Finite Element Study of the Thermal Barrier Coating Fractures

Y. Wu[2]; P. Hsu*[1]; M. H. McCay[2]; Y. Wang[1]

1. Florida Institute of Technology, Mechanical Engineering Program, USA
2. Florida Institute of Technology, Center for Advanced Coatings, USA

After the laser thermal cycling test of an 8YSZ thermal barrier coating, the specimen is cross sectioned and polished. The SEM images of the cross-section microstructure are taken for metallographic analysis and finite element computation of stress intensity factor and strain energy release rate in a prior study. This paper is a continuation of the previous effort to further explore important issues in computing these fracture mechanics parameters. These issues include the computation domain size relative to the crack size, the distance between the horizontal crack (along the in-plane direction of the top coat) and the thermally grown oxide layer, the variation of boundary conditions. These issues should be parametrically studied so a systematic methodology can be established for fracture mechanics computations. This will be essential for developing a TBC service life model based on the combination of the laser rig test result and finite element analysis.

CMAS Degradation of E/TBC and Mitigation Strategies I

Room: Ponce de Leon
Session Chair: Ravisankar Naraparaju, DLR - German Aerospace Center

10:20 AM

(ICACC-S2-035-2020) Improving the resistance of thermal barrier coatings to molten particulates through blending with rare-earth oxides (Invited)

M. J. Walock*[1]; C. Mock[2]; A. Ghoshal[1]; M. Murugan[1]; M. S. Pepi[3]

1. US Army Research Laboratory, Vehicle Technologies Directorate, USA
2. SURVICE Engineering, USA
3. US Army Research Laboratory, Weapons and Materials Research Directorate, USA

Damage from molten particulates in the hot-section of military gas turbine engines can significantly increase the overall life-cycle cost of the system. In addition, there have been instances where rapid deposition and accumulation of glassy calcia-magnesia-alumino-silicate (CMAS) coatings have resulted in the loss of aircraft and lives. Developing a sandphobic thermal/environmental barrier coating can reduce engine downtime, increase performance in harsh environments, and save lives. To this end, the Army Research Laboratory has been developing unique blended coatings with improved resistance to CMAS damage and accumulation. Specifically, recent efforts have focused on blending rare-earth oxides, such as gadolinia and samaria, with yttria-stabilized zirconia powders for consolidation via air plasma spray onto Inconel 718 discs (1-in diameter). In addition to evaluating the accumulation-resistance of these coatings in the Hot-Particulate Ingestion Rig under engine-relevant conditions, the infiltration-resistance of the samples was assessed under isothermal conditions. As-deposited and tested coatings were characterized using optical microscopy, surface profilometry, and scanning electron microscopy with energy dispersive X-ray spectroscopy and electron backscatter diffraction.

10:50 AM

(ICACC-S2-036-2020) TBC lifetime dependence on CMAS dose rate and history

B. Jun*[1]; E. H. Jordan[2]; R. C. Cooper[2]; N. E. Jonsson[1]

1. University of Connecticut, Materials Science, USA
2. University of Connecticut, Mechanical Engineering, USA

CMAS infiltration into TBCs cause a decrease in lifetime of the TBC. Prior investigation has focused on preloading TBC samples with a critical dosage of homogenized CMAS and observing the infiltration and failure mechanisms at an isothermal target temperature. In real engines, CMAS is neither homogeneous nor applied in bulk prior to heating. The following factors not usually addressed include: 1. heterogeneity of CMAS, 2. deposition rate, 3. deposition history, and 4. temperature gradient. Each of these factors affect the infiltration rate and behavior of CMAS, which in turn affects the lifetime deficit the CMAS causes the TBC. Using a test rig of proprietary design, we are able to feed solid heterogeneous CMAS at very low continuous deposition rates with selected front and back surface temperature histories. Results using this rig addressing the influence of some of the four factors mentioned will be presented.

11:10 AM

(ICACC-S2-037-2020) Influence of Microstructure on Lifetime and CMAS Resistance of EB-PVD Gadolinium Zirconate Thermal Barrier Coatings

C. Mikulla*[1]; R. Naraparaju[1]; U. Schulz[1]

1. DLR - German Aerospace Center, Institute of Materials Research, Germany

Thermal barrier coatings (TBCs) used in high-temperature sections of aero-engines and gas turbines are often subjected to severe degradation by molten calcium-magnesium-alumina-silica (CMAS) minerals mainly found in volcanic ashes (VA), runway dust, desert sands and air pollution. The application of reactive TBCs such as gadolinium zirconate (GZO) on the affected parts is one of the successful approaches to protect the TBCs against CMAS attack. Being exposed to molten CMAS, the coatings form crystalline reaction products that seal pores and gaps and hinder further infiltration of the deposit. However, the microstructure of these coatings plays a significant role on both the infiltration and reaction kinetics of the sacrificial layers with CMAS. GZO layers with different columnar microstructure and porosity were deposited by varying the electron beam physical vapor deposition (EB-PVD) process parameters. Infiltration experiments with CMAS or VA were carried out at 1250°C for different time intervals between 5 min and 50 h. It was found that microstructural features influence greatly the infiltration depth and reaction kinetics. Lifetime of these coatings was tested by doing thermal cyclic testing and a correlation with their microstructure is drawn.

11:30 AM

(ICACC-S2-038-2020) Thermochemical effects related to CMAS-type corrosion of APS Y_2O_3 coatings

P. Mechnich*[1]

1. DLR - German Aerospace Center, Institute of Materials Research, Germany

Yttria (Y_2O_3) is a promising material for thermal and environmental barrier coatings (T/EBC) for Al_2O_3/Al_2O_3 ceramic matrix composites to be used in hot sections of aero-engines. Ingestion and subsequent deposition of airborne inorganic, CMAS-type aerosols on top of T/EBC is considered a major issue for coating lifetime. In particular infiltration of open coating porosity by molten CMAS deposits is considered a major reason for "cold shock" fracture and subsequent T/EBC spallation. The response of air plasma sprayed (APS) Y_2O_3 against the attack of a variety of CMAS-type test dusts is presented in the light of phase formation, solid solubility, and microstructural evolution. APS Y_2O_3 exhibits good resistance versus all CMAS variants at temperatures up to 1400°C. Melt infiltration is effectively mitigated by a low-permeable microstructure and rapid surface crystallization of Y-rich phases such as Yttrium-oxyapatites, Yttrium-garnets, and Yttrium-silicates. Phase formation is governed by the chemical composition of CMAS, where Ca, Si, and Fe are the dominating reactive species. Laboratory scale CMAS-corrosion of APS Y_2O_3 is considered a suitable tool to study fundamental effects related to CMAS corrosion of Yttrium- or other RE-containing T/EBC materials.

CMAS Degradation of E/TBC and Mitigation Strategies II

Room: Ponce de Leon

Session Chairs: Peter Mechnich, DLR - German Aerospace Center; Michael Schmitt, Pennsylvania State University

1:30 PM

(ICACC-S2-039-2020) Novel magnetron sputtered ceramic YSiFe oxide as CMAS-resistant coatings for thermal and environmental barrier coatings (Invited)

R. Naraparaju*[1]; A. Ott[1]; U. Schulz[1]; P. Mechnich[1]

1. DLR - German Aerospace Center, Institute of Materials Research, Germany

A novel YSiFe oxide coating has been developed by reactive magnetron sputtering and a suitable heat-treatment was applied to achieve the crystalline phases that are stable at high temperatures. The heat-treatment includes an isothermal heating at 1250 °C for 1h followed by a rapid cooling to room temperature. The resulting phases X2-Yttria Mono Silicate (YMS), Yttrium Iron oxide and Yttria were found to be nano-crystalline in nature and were homogeneously distributed in the coating. The coating has been subjected to CMAS reaction by performing infiltration experiments at 1300 °C for different time intervals. The YSiFe oxide coating has induced both intrinsic crystallisation of the CMAS melt and formation of reaction products such as oxyapatite and garnet phases. The Fe diffusion from the YSiFe oxide coating into the glass reinforced the reactive crystallisation of CMAS leading to apatite and further garnet formation which consumed CMAS glass and prevented further infiltration. This novel coating has been proven to be a very good CMAS resistant coating and could therefore be a promising candidate for CMAS resistant multilayer EBCs necessary to protect CMC and is also suitable to be applied on top of conventional 7YSZ TBCs.

2:00 PM

(ICACC-S2-040-2020) High-Temperature Interactions between Rare-Earth Pyrosilicate Thermal Environmental Barrier Coating Ceramics and Calcia-Magnesia-Aluminosilicate(CMAS)

L. R. Turcer*[1]; H. Sternlicht[1]; C. Watts[1]; M. Koval[1]; H. Garces[1]; N. P. Padture[1]

1. Brown University, School of Engineering, USA

Higher operating temperatures in gas-turbine engines are achieved by using ceramic-matrix-composites (CMCs) instead of metallic hot-section components. However, in the presence of water vapor active-oxidation of CMCs is prevalent, therefore, dense environmental barrier coatings (EBCs) are needed. At temperatures above 1200 °C, silicate particles (sand, volcanic ash, fly ash, etc.) that enter the engine melt on the hot surfaces and form calcia-magnesia-aluminosilicate (CMAS) glass deposits. These molten CMAS glass deposits can then react with the EBCs and/or penetrate grain boundaries, which can lead to premature coatings failure. Instead of a three-layer system (TBC-Interlayer-EBC), if one material could (1) have a good thermal-expansion match with CMCs, (2) high-temperature phase stability up to 1700 °C, (3) low thermal conductivity, and (4) resistance to high-temperature attack by CMAS, then a one-layer thermal environmental barrier coating (TEBC) could be used. Binary solid-solutions of RE pyrosilicates (e.g. $Y_xYb_{(2-x)}Si_2O_7$) are being examined. Aliovalent doping is also being considered to

lower thermal conductivity. High-temperature interactions between binary solid-solutions of RE pyrosilicate and CMAS are studied, and the nature of these interactions, damage mechanisms, and their mitigation techniques are discussed.

2:20 PM

(ICACC-S2-041-2020) Influence of calcium-magnesium-alumino-silicate (CMAS) on environmental barrier coating materials: Comparative study on pure and composite rare earth silicates

M. Wolf*[1]; D. E. Mack[1]; R. Vassen[1]; O. Guillon[1]

1. Forschungszentrum Juelich, IEK1, Germany

For the corrosive media protection of SiC ceramic matrix composites (CMCs), rare earth silicate environmental barrier coatings (EBCs) are state of the art. The interaction of pure rare earth silicate EBC materials (Yb_2SiO_5, $Yb_2Si_2O_7$) and three ytterbium silicate composites with molten calcium-magnesium-alumuniosilicate (CMAS) were studied at high temperature (1400°C). To evaluate the recession of the different materials after an 8h reaction time, the samples were characterized by SEM and XRD. Additionally, the coefficient of thermal expansion (CTE) was determined to evaluate the suitability of Yb composites as EBC materials for SiC CMCs. Results show monosilicates exhibit a lower recession in contact with CMAS than their disilicate counterpart. The ytterbium composite results are promising for future applications.

2:40 PM

(ICACC-S2-042-2020) Object Oriented Finite Element Analysis of Erosion and CMAS Degradation in Complex Materials

M. Schmitt*[3]; J. Schreiber[3]; B. J. Harder[2]; D. E. Wolfe[1]

1. Pennsylvania State University, USA
2. NASA Glenn Research Center, Environmental Effects and Coatings, USA
3. HAMR Industries LLC, USA

The search continues for advanced thermal and environmental barrier coating (T-EBC) and ceramic matrix composite (CMC) material systems and fabrication methods that can push the boundaries of turbine operation. The aggressive thermomechanical environment targeted for next-generation systems imparts unique operating requirements including resistance to degradation caused by ingestion of foreign particles. These particles can impact the surface and cause erosive wear, or if in certain stoichiometric ranges, can melt and infiltrate and/or react with the protective material system. This study utilizes an object oriented finite element approach to elucidate the impact of fabrication method and coating morphology on erosive particle impact behavior and thermal shock during CMAS infiltration. Particle impact on air plasma spray (APS), electron beam – physical vapor deposition (EB-PVD), and plasma spray physical vapor deposition (PS-PVD) morphologies will be explored for a variety of relevant T-EBC materials and erosion conditions and compared with experimental data. The effect of CMAS infiltration is simulated to assess thermal shock behavior based on common CMAS compositions and predicted reaction products and infiltration depths for various coating microstructures. The results are then used to assist in directing future material design architectures and development.

3:20 PM

(ICACC-S2-043-2020) Na_2SO_4 Interactions with Air Plasma Sprayed $Yb_2Si_2O_7$ /Si/SiC Systems

L. A. Herweyer*[1]

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

Air plasma sprayed (APS) $Yb_2Si_2O_7$ + Si coated SiC specimens were exposed to ~2.5 mg/cm^2 films of Na_2SO_4 from 800-1316°C at times between 0-240h in controlled gaseous environments. SEM, EDS, XRD, TEM, FIB-SEM and SAED were used to determine the $Yb_2Si_2O_7$ coating reactivity, assess the oxidation resistance of the Si bond coat, and identify coating system degradation mechanisms. It was found that Na_2SO_4 reacted with $Yb_2Si_2O_7$ to form Yb_2SiO_5 at both the coating surface and in the bulk, leaving a two phase $Yb_2Si_2O_7$ and Yb_2SiO_5 coating behind. Additionally, a possible sodium silicate reaction product was found to penetrate the entire coating all the way down to the Si bond coat above 1200°C. The Si bond coat oxidized to form a bi-layer SiO_2 and Yb_2O_3-SiO_2-Na_2O thermally grown oxide that grew faster than in dry O_2 environments without Na_2SO_4.

3:40 PM

(ICACC-S2-044-2020) Experimental viscosity of CMAS melts as a function of composition and temperature

R. Webster*[1]; E. Opila[1]

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

Calcium magnesium alumino-silicate (CMAS) attack is a pressing issue in the development of protective barrier coatings for next-generation aircraft engine components. CMAS originates as siliceous debris such as sand or volcanic ash which can be ingested into the engine during flight. At temperatures nearing 1200°C, CMAS can melt and penetrate coating materials, causing premature failure. The composition of CMAS deposits can vary widely and depends on the CMAS source and location of ingest. Additionally, chemical interactions with water vapor from combustion can change the glass composition over time. Properties such as melting temperature and viscosity are affected by CMAS composition and are important parameters to consider in the design of protective coatings. CMAS compositions with varying CaO, MgO, Al_2O_3, and SiO_2 contents were prepared as glasses from reagent grade oxides. A high temperature rotating viscometer was used to experimentally measure the viscosity of glasses over temperatures ~1200-1500°C. Experimental viscosities were compared to values obtained from three different viscosity models used frequently in the literature. The effect of quinary additions such as Fe_2O_3, TiO_2, and Yb_2O_3 on viscosity was also assessed. The interaction of water vapor with CMAS was probed to determine if any changes in glass composition or coating penetration rates are expected in an engine environment.

4:00 PM

(ICACC-S2-045-2020) Dissolution Kinetics of Thermal Barrier Oxides in Molten Silicates

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The degradation of thermal barrier coatings (TBCs) by molten silicates (CMAS) is a fundamental barrier to increasing the operation temperature of gas turbine engines and their fuel efficiency. A promising mechanism for CMAS mitigation has been found in rare earth (RE) zirconate TBCs (e.g., $Gd_2Zr_2O_7$), which react with the molten CMAS to form favorable crystalline products capable of arresting melt penetration. However, for this mechanism to be successful, a rapidly dissolving TBC with favorable diffusion kinetics is required; simultaneously, quantitative data on the kinetics of TBC-CMAS interactions is scarce. In the present work, the dissolution kinetics of RE zirconate samples into CMAS and the ensuing diffusion kinetics of TBC cations were quantitatively investigated using 1D TBC-CMAS diffusion couples. The diffusion kinetics of RE zirconates was similar to that of yttria-stabilized zirconia. Conversely, the RE zirconates dissolved significantly faster, which yielded faster saturation of the CMAS and enabled rapid reactive crystallization. Additionally, the results provided qualitative insight on reaction product formation and attack mechanisms for both polycrystalline and single crystalline RE zirconates. Finite element models were developed using the experimentally-obtained kinetic data to predict dissolution, diffusion, and infiltration behavior within model TBC geometries.

S3: 17th International Symposium on Solid Oxide Cells (SOC): Materials, Science and Technology

Electrocatalysts

Room: Crystal

Session Chair: Prabhakar Singh, University of Connecticut

8:30 AM

(ICACC-S3-026-2020) High Entropy Alloy Fuel Electrode for Direct Internal Reforming of Carbonaceous Fuels in Advanced SOFCs (Invited)

B. Hu*[1]; S. Belko[1]; A. Aphale[1]; R. Kumar[1]; A. Dongare[1]; R. Bhattacharya[2]; P. Singh[1]

1. University of Connecticut, Materials Science and Engineering, USA
2. UES, InC, USA

Solid oxide fuel cells offer fuel flexibility, high energy efficiency, and freedom for clean power generation as well as energy storage. State-of-the-art nickel cermet anodes are prone to carbon formation and pulverization when fueled with carbonaceous fuels. In this technical contribution, we present our research on high entropy alloy (HEA) fuel electrode, comprised of multiple low-cost non-noble metal alloying constituents that have demonstrated carbon resistance during external and internal reforming of methane and alcohol fuels. The well-designed structure and catalytic components provide controllable reforming rates offering ability to achieve homogeneous temperature distribution in cells and stacks. The reformation rates of the HEA catalyst have been measured by a gas chromatograph and compared with that of Ni cermet. Electrochemical performance and stability results of HEA anode-based cells will be presented. Carbon resistance has been examined using scanning electron microscopy and Raman spectroscopy. Absence of carbon deposition on post-test HEA anodes was confirmed while filamentary carbon fibers formed on post-test Ni cermet anodes. A combined approach of experimental and theoretical study of carbon adsorption and deposition have been utilized to develop mechanistic understanding of structure-performance relationship.

9:00 AM

(ICACC-S3-027-2020) Intelligent Behavior of $La(Sr)_{n+1}Fe(M=Mn, Co, Ni)O_{3n+1}$ based Perovskite Oxide Electrodes and Propane fuel SOFC (Invited)

T. Shin*[1]

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Utilization of carbon dioxide or hydrogen complex could be an effective approach for accessing a new form of energy carrier and enabling a carbon-neutral cycle. In particular, reduction of CO_2 or H_2O into CO or H_2 and oxidation fuel devices with renewable electricity has been proposed as an alternative method to store this energy, overcoming the inherent problems with intermittency and geographical distribution of renewable sources. Thus, solid oxide electrochemical cells (SOC), such as solid oxide fuel cell (SOFC) and solid oxide electrolysis cell (SOEC), have received much attractive attention in the energy issues due to the promise of providing high efficiency, low emission and fuel flexibility as the next generation. . In this study, we will discuss that the intelligent performance for tolerance against redox coupling operation could be achieved by using the reversible phase transition on lanthanum ferrite based oxides substituted with several B-site dopants. Doping of some transition metal into the B-site of lanthanum ferrite based perovskite improved the phase stability and reversibility, thereby promoting the further reduction of $Fe^{2+/3+}$ pairs and preventing the formation of impurity during the phase transition: ABO_3 perovskite to A_2BO_4 Ruddlesden-Popper.

9:30 AM

(ICACC-S3-028-2020) Study of Synthesis anomaly in cathode material $PrBaCo_2o_{6-\delta}$

A. S. Bangwal*[1]; P. Singh[1]

1. Indian Institute of Technology(BHU), PHYSICS, India

The general conception is Solid State route need more thermal energy than Auto Combustion route. However, during the synthesis of $PrBaCo_2O_{6-\delta}$, we observed just the opposite. The optimized sintering temperatures are found to be 1150 and 1050 °C for the auto-combustion route (P-ACR) and solid-state route (P-ACR) respectively, although later has large grain size (even after equal calcination treatment). The porous microstructure of P-SSR is suitable for SOFC cathode materials while that of P-ACR is pore-free. Study of HRTEM reveals that there is a crystal growth on a smooth surface with preferred orientation. This synthesis anomaly is due to anisotropic surface nucleation growth. The variation in the Co–O–Co bond angle reveals Jahn–Teller vibrational anisotropy in the-b plane leading to the anisotropic synthesis behavior.

9:50 AM

(ICACC-S3-029-2020) Synthesis and study of Calcium doped High Entropy Perovskite Oxide $La(Co_{0.2}Cr_{0.2}Fe_{0.2}Mn_{0.2}Ni_{0.2})O_3$ for Solid Oxide Fuel Cell Cathode part

S. Gajjala*[1]; R. Koc[1]

1. Southern Illinois University Carbondale, Mechanical Engineering and Energy Processes, USA

In the current research, High Entropy Perovskite Oxide of La(Co0.2Cr0.2Fe0.2Mn0.2Ni0.2)O3 with Calcium 0, 10, 20 and 30% on A-site is synthesized by using polymerizable precursor method and the newly synthesized powders wherein homogeneous single phase with orthorhombic perovskite structure. By varying the level of calcium doping on A-site, the crystal structure, electrical conductivity and microstructural properties of these newly prepared materials are studied. Electrical conductivity measurement was done between the temperature range of 300-900°C in air and the results showed that the conductivity was highly improved by the addition of calcium on A-site and maximum electrical conductivity of 60S/cm was achieved for the sample with 30% calcium on A-site. By tweaking the elements on A-site and site, materials with various characteristics can be synthesized and studied.

Coatings and Contacting Layers

Room: Crystal

Session Chair: Mihails Kusnezoff, Fraunhofer IKTS

10:30 AM

(ICACC-S3-030-2020) MnCo-based spinels on Crofer22APU by electrophoretic method: Compositional modifications and performance in stack

F. Smeacetto*[1]; A. Sabato[1]; H. Javed[2]; E. Zanchi[1]; S. Molin[3]; G. Cempura[4]; K. Herbrig[2]; C. Walter[2]

1. Politecnico di Torino, Applied Science and Technology, Italy
2. Sunfire GmbH, Germany
3. Gdansk University of Technology, Laboratory of Functional Materials, Faculty of Electronics, Telecommunications and Informatics, Poland
4. AGH University of Science and Technology, Poland

Key aspects in obtaining well performing protective coatings for interconnects in solid oxide cells are the deposition method and the sintering treatment. This work focuses on the use of electrophoretic deposition to deposit the MnCo-based spinel on Crofer22APU, from suspension characterization, deposition parameters and sintering steps optimisation. Area specific resistance tests up to 8600 hours at 850°C are evaluated to assess the efficacy of MnCo coating in limiting the oxide scale growth and Cr diffusion. EPD resulted in uniform and homogenous coating having the thickness of 10-15 μm,

showing an ASR degradation rate of 0.5 mΩ cm^2 kh^{-1}. The optimized process was subsequently scaled up to coat real dimension Crofer22APU plates. A 30-layers stack containing 4 repetition units coated with EPD-MnCo was tested at the nominal temperature of 850 °C in SOFC mode, with a 75% fuel utilization for 3000 hours, including thermal cycles. The mechanism by which MnCo-based spinels can influence elements interdiffusion at the steel-oxide-coating interface and relative contribution to the overall ASR is reviewed and discussed by means of advanced transmission electron microscopy and phase composition measurements. Understanding and controlling the evolution of the oxide scale-coating interface provide a deeper insight to further improve coatings durability and performance issues

10:50 AM

(ICACC-S3-031-2020) In-situ Fe-doped MnCo spinel coatings on Crofer 22 APU and AISI 441 interconnects: Microstructural, electrical and oxidation properties

E. Zanchi*[1]; S. Molin[2]; A. Sabato[1]; B. Talic[3]; G. Cempura[4]; A. R. Boccaccini[5]; F. Smeacetto[1]

1. Politecnico di Torino, Applied Science and Technology, Italy
2. Gdansk University of Technology, Laboratory of Functional Materials, Faculty of Electronics, Telecommunications and Informatics, Poland
3. Technical University of Denmark, Energy Conversion and Storage, Denmark
4. AGH University of Science and Technology, Poland
5. University of Erlangen-Nuremberg, Institute of Biomaterials, Germany

Manganese cobaltite-based spinel coatings have been reported to effectively limit oxidation and Cr-evaporation from ferritic stainless steel interconnects in solid oxide cell stacks. Currently, the possibility to improve the coating performance by transition metals doping of the spinel is of great interest; however, doped Mn-Co spinels are usually synthetized before the coating deposition. In this work, Fe-doped Mn-Co spinel coatings are obtained by a single-step electrophoretic co-deposition of $Mn_{1.5}Co_{1.5}O_4$ and Fe_2O_3; a co-deposition mechanism is proposed and discussed. The efficacy of obtained Fe-doped Mn-Co spinels coatings deposited on both Crofer 22 APU and AISI 441 steel is evaluated through a study of the oxidation kinetics and area specific resistance at 750 °C up to 3200 h. Fe-doped coatings show great stability during aging, considerably decreasing the oxidation rate of the substrates. In particular, Fe-modified coating leads to a reduction of ASR of coated interconnects, despite the poorer conductivity of the Fe-doped Mn-Co spinel compared to the undoped Mn-Co. A detailed SEM and TEM post mortem analysis reveals the development of different oxide scale and reaction layers depending on both the coatings and the substrates; oxidation mechanisms for Fe-doped Mn-Co spinel coated Crofer 22 APU and AISI 441 are reviewed and discussed.

11:10 AM

(ICACC-S3-032-2020) Development and testing of MnCo based spinel protective coatings prepared by spray pyrolysis

S. Molin*[1]; B. Kamecki[1]; D. Szymczewska[1]; F. Smeacetto[2]; P. Z. Jasinski[1]

1. Gdansk University of Technology, Laboratory of Functional Materials, Faculty of Electronics, Telecommunications and Informatics, Poland
2. Politecnico di Torino, Applied Science and Technology, Italy

In this work, we have developed and tested interconnect protective coatings based on the MnCo2O4 composition, including spinel modifications. Coatings have a thickness of ~2 μm and were developed by a low-temperature spray pyrolysis process, with no further heat treatment. By using spray pyrolysis fabrication the high-temperature sintering step is omitted, thus no initial oxide formation occurs. Tested materials include: MnCo2O4, MnCo2O4 with the addition of CeO2 and a layered CeO2/MnCo2O4 system. In order to determine corrosion properties, coated Crofer 22 APU substrates were subjected to cyclic oxidation at 700 °C for a total of 3000 hours. Reference samples (uncoated Crofer 22 APU coupons) were oxidized for 1000 hours in a separate exposure to limit potential poisoning of the coated samples. In addition to corrosion exposure, electrical measurements and compatibility with glass-ceramic sealants were assessed. Weight gain and post mortem analyses show a beneficial effect of the applied coatings, reducing the oxide scale formation. Spray pyrolysis is an interesting technology to prepare coatings for the steel interconnects.

11:30 AM

(ICACC-S3-033-2020) Optimization and Validation of LSCo-Mullite Composite Contact Material for Solid Oxide Fuel Cells

Y. Chou*[1]; N. L. Canfield[1]; J. F. Bonnett[1]; J. Choi[1]; J. W. Stevenson[2]

1. Pacific Northwest National Lab, Materials, USA
2. Pacific Northwest National Lab, USA

In previous work we have demonstrated that, by adding mullite of low CTE to high-conducting LSCo matrix, the CTE of LSCo/mullite can be tailored in accordance with the rule of mixtures. To match the composite's CTE to SOFC components, however, a very large amount of mullite needs to be added, which results in substantial reduction in electrical conductivity. In this work, we propose reinforcement with strong short fibers (YSZ and Al_2O_3) to optimize the composite contact materials. To maintain high conductivity, a composition of LSCo/mullite (10 v%) was chosen for optimization. LSCo, mullite and YSZ or alumina fibers at 5 or 10 v% were mixed and die pressed into pellets and rods. Sintered rods were measured for CTE and pellets for electrical conductivity (650-900ºC). Small bilayer coupons were painted with candidate contact material, sintered, and tested for bonding strength in as-sintered and thermally cycled conditions. Results showed that the small addition of strong fibers did improve bonding strength substantially without severely reducing the conductivity. Selected formulation will be tested in a generic stack fixture for cell performance for 500-1000h followed by 5-10 deep thermal cycles.

11:50 AM

(ICACC-S3-034-2020) Improving Anode Current Collection of Anode Supported Tubular Solid Oxide Fuel Cells

Y. Du[1]; H. Feng*[1]

1. Kent State University, USA

For a tubular-supported Solid Oxide Fuel Cells (SOFC), it is critically important to have a reliable current collector on the anode side. Literature indicated that different types of Anode current collectors were used and their performances varied. Considering about the operating conditions of the SOFC, a good current collector must have following characteristics: 1) Good electronic conductivity; 2) Chemical stability; 3) Physical stability; and 4) High temperature tolerance. In this paper aims to improve the current collection in tubular Anode supported SOFC. Through theory discussions and experimental investigations, a material was identified to be high temperature-tolerant, low resistance, durable and low cost. In addition, a universal-removable design of current collector for tubular SOFC was also proposed. The current collector was able to conduct electricity of anode-supported tubular SOFCs at the temperature of 700ºC. The current collector showed an excellent conductivity and heat resistance over 1000-hours stability test.

Simulation and Materials

Room: Crystal
Session Chair: Kevin Huang, University of South Carolina

1:30 PM

(ICACC-S3-035-2020) Atomic-scale Modeling of Materials for Solid Oxide Cells (Invited)

I. Castelli*[1]

1. Technical University of Denmark, Department of Energy Conversion and Storage, Denmark

Over the last decades computer simulations have become increasingly popular as a means to understand and predict materials' properties at various time- and length scales. This has been possible thanks to methodological improvements and increase in computational power, allowing for the treatment of more complex and realistic atomic systems, which has improved the predictive power of these models. In this talk, I am going to describe how Density Functional Theory (DFT) can successfully be used to (i) identify novel perovskite-based materials to be used in Solid Oxide Fuel Cells, as well as to (ii) understand electronic and ionic transport properties of currently used materials at the atomistic level. More in details, (i) I will use a high-throughput approach based descriptors like stability, effective masses and Fermi velocities, to design novel materials by doping SrLaCoO3. (ii) DFT can also be combined with continuum molecular dynamics to study the effects of substitutional defects in the ionic conductivity in Ceria, one of the most studied used ionic and electronic conducting materials. Charge and size of the defect play a fundamental role in enhancing (or blocking) the oxygen transport properties. These fundamental insights into the factors and trends that governs conductivity and diffusion coefficients provide a new route for designing novel functional materials.

2:00 PM

(ICACC-S3-036-2020) Computational Design of Perovskite-type Oxide Electrode to Achieve the Improved Catalytic Activity through Exsolution Phenomena (Invited)

K. Kim[1]; J. Han*[1]

1. POSTECH, Chemical Engineering, Republic of Korea

Redox ex-solution, in-situ synthesis process of metal nanoparticles upon high-temperature reduction has widely been studied as a way to fabricate metal nanocatalyst-decorated oxide electrodes for solid oxide electrochemical cells (SOCs). However, the underlying mechanisms related to this phenomenon are not completely understood. In the first part of this talk, we will discuss about our exploration of the possibility and detailed mechanism for various kinds of B-metal exsolution on double layered $PrBaMn_2O_{5+\delta}$. We will introduce (1) a possible driving force for the exsolution of B-metal nanoparticles (NPs), (2) thermodynamically preferable mechanism for the exsolution of alloy NPs, and (3) how to boost B-metal exsolution to further improve cell performance SOCs with the experimental verifications. In the second part of this talk, we will discuss the practical solutions that effectively accelerate B-site cation exsolutions in perovskite oxide. We control the degree of Co enrichment at the surface of $SrTi_{0.75}Co_{0.25}O_{3-\delta}$ (STC) through lattice strain. DFT show that the tensile strain that plays a role in stretching B-O bond length promotes the Co ex-solution in STC. We successfully demonstrate that this approach can facilitate the exsolution of B-site cations, suggesting a new strategy for improving the surface reactivity of perovskite-type oxides.

2:30 PM

(ICACC-S3-037-2020) Investigation of Prospective Catalyst from Co and Fe doped $LaAlO_3$ Perovskites

X. Gao*[1]; J. Irvine[1]

1. University of St Andrews, United Kingdom

In the case of transition metal-doped perovskite oxides, nanoparticles can be exsolved and dispersed on preferable facets via processing the materials under a reduced atmosphere. Such perovskites, with exsolved nanoparticles, can provide improved catalysis performance to satisfy the demand of energy conversion applications. Among the perovskite (ABO_3) family, Lanthanum Aluminate ($LaAlO_3$) based materials are considered to have potential applications in the energy conversion field. Here, we aim at transition metal (cobalt, iron and both) substituted $LaAlO_3$, by investigating crystal structure, thermal behavior, electrical conductivity, and exsolution studies. Single-phase samples were prepared in for Co, Fe, and Fe-Co both doping in $LaAlO_3$. Mass losing predominately takes place at the temperature rising stage. Matched thermal expansion coefficient has been detected. Fe-Co co-doping could lift the electrical conductivity significantly. Successful exsolution of Co, Fe and Fe-Co nanoparticles exhibiting particle size with 10-50 nm, 30-90 nm, and 25-65 nm has been achieved. After simulating the L_3 and L_2 ionization edge intensities from EELS, the particles are the metal or alloy, but the surface is capped by the thin oxide layer. Hence, we have developed a new material family with exsolved nanoparticles towards the electrochemical catalysis application, such as the fuel electrodes in Solid Oxide Cells (SOC).

2:50 PM

(ICACC-S3-038-2020) Theoretical and Experimental Evaluation of $BaCo_xFe_yZr_zO_{3-d}$ (BFCZ) as cathode for SOFC

J. Liu*[1]; R. Jacobs[2]; B. Na[1]; H. Abernathy[1]; S. Lee[1]; D. Morgan[2]; T. Kalapos[1]; G. Hackett[1]

1. NETL, USA
2. University of Wisconsin, Department of Materials Science and Engineering, USA

A recent high-throughput Density Functional Theory (DFT) study of ~2150 perovskite materials assessed bulk thermodynamic stability via the Python Materials Genomics (Pymatgen) package and predicted oxygen reduction reaction (ORR) catalytic activity with the O p-band center as an electronic structure descriptor. Of the sampled perovskite compositions, BaCoxFeyZrzO3-d(BFCZ) (x+y+z=1) was determined to be a promising new highly active and stable ORR cathode material. Here, perovskite-type oxides $BaCo_xFe_yZr_zO_{3-d}$ (x+y+z=1) are prepared and systematically studied via X-ray diffraction (XRD) analysis and electrical conductivity relaxation (ECR). The microstructure and thermal stability of the materials are characterized by SEM and AFM. The electrochemical performance of the BFCZ is characterized by electrochemical impedance spectroscopy (EIS). The long-term degradation of BFCZ and the grain growth and sintering behavior are studied under polarization and open circuit conditions. The results, as suggested by the prediction of DFT model, not only show high ORR catalytic activity but also exhibit excellent compatibility with a ceria-based electrolyte.

HT Electrolysis

Room: Crystal
Session Chair: John Pietras, Saint-Gobain

3:30 PM

(ICACC-S3-039-2020) Solid Oxide Cells developments: From materials to system (Invited)

J. Mougin*[1]; J. Laurencin[1]; S. Di Iorio[1]; J. Aicart[1]; J. Vulliet[2]; P. Coddet[2]; P. Cloetens[3]; M. Hubert[3]

1. CEA, Liten, France
2. CEA, DMAT, France
3. ESRF, France

Solid oxide cells (SOC) technology is considered for many years for CHP systems when operated in fuel cell (SOFC). It can also be operated in electrolysis (SOEC) to produce H2. Reversible operation (rSOC) allows storing and restituting intermittent renewable electricity. It can also be operated in coelectrolysis by adding CO2 to H2O to produce syngas (H2/CO) that can be transformed in synthetic fuels. This flexibility, complemented by good efficiencies,

constitutes a significant technological and economic potential. CEA R&D activities cover these different application areas, from cell development to stack and system design and operation, supported by multi-physic and multi-scale modeling activities and advanced characterization. Particular emphasis is given to the understanding of performance and degradation controlling parameters at every scale in order to optimize ceramic cells and to design robust and reliable SOC stacks. System integration has been given specific attention allowing the design of complete SOEC system producing 1.2 Nm3 H2/h with an electric yield of 3.5 kWh/Nm3 assuming an available steam source at 150°C. An rSOC system has also been built and its ability to operate in SOEC and SOFC (in H2 and CH4) modes demonstrated, each displaying 3 power levels has been proven. The results showed that all transition cycles could be done in 3 to 10 min without negatively affecting short-term stack performances.

4:00 PM

(ICACC-S3-040-2020) Experimental and Numerical Study on the Performance of Solid Oxide Electrolysis Cells (Invited)

S. Lee*[1]; T. Yang[1]; Y. Fan[1]; J. Liu[1]; T. Kalapos[1]; H. Abernathy[1]; G. Hackett[1]

1. National Energy Technology Laboratory, USA

The performance of solid oxide electrolysis cells (SOECs) is investigated via multiple experiments and an internally developed high-fidelity multiphysics model. It is observed that operating parameters such as gas composition and electrical current affect characteristic phenomena of SOECs. Inspired by the observation, the full parameter space with various steam supply conditions is explored by the numerical parametric study. The numerical simulation is first validated by the performance trends observed in experiments with specific steam supply conditions. Then, the cell performance under different working conditions is analyzed with the full parametric space of steam supply conditions to investigate the global trends of SOEC performance. The global minimum resistance under different working loads is also investigated to better understand the performance of a button SOEC under practical working conditions. Furthermore, the performance degradation due to the Ni redistribution in electrolysis mode is predicted and investigated based on the changes of reaction sites and microstructural properties. This study can provide guidance for the design of efficient solid oxide electrolysis cell as well as reversible solid oxide fuel cell (r-SOFC) systems.

4:30 PM

(ICACC-S3-041-2020) Solid oxide cells as power-to-gas-to-power systems: Experimental and numerical study on their performance and system degradation (Invited)

V. Subotic*[1]

1. Graz University of Technology, Austria

Reversible solid oxide cells (rSOC) enable to generate electricity, heat and valuable fuels in a highly-efficient and environmentally-friendly manner. In order to enhance commercialization of such systems, detailed investigation of process and loss mechanisms as well as possible failure modes that occur during the operation are of crucial interest. Obtaining such information represents a great basis for development of online-monitoring diagnostic tools, the application of which enables in-operando optimization of the system performance and prevent undesired system degradation. For the purpose of this study, a physical model was developed to simulate polarization and electrochemical impedance characteristics, thus separating single losses and making suggestions for the performance improvement. In addition, experimental investigations were performed on single cells of industrial size and 10-cell-stacks, varying the operating parameters, such as gas composition, operating temperature, operating current, etc. Moreover, operating parameters were selected to accelerate degradation in rSOCs in a targeted manner. This ensured identification of specific degradation parameters and their application for development of novel online-monitoring tools.

5:00 PM

(ICACC-S3-042-2020) Highly Efficient SOE System Design - Update on AVL's SOE activities

R. Schauperl*[1]

1. AVL List GmbH, Austria

A central requirement of highly efficient SOE systems concern the steam supply to the stack at high temperatures. Therefore, the focus relies on coupling with industrial processes which allow direct usage of excess steam or the usage of excess heat for steam production. To address these challenges, Pinch-analysis and comprehensive system simulations were conducted and process interface specification requirements of various SOE applications investigated. Process interfaces can differ significantly in terms of available heat, temperature levels and type of media (for input, as well as output interfaces of SOE). The conducted work aimed for finding highest efficient SOE layouts and integration solutions, while keeping the number of system components and complexity as low as possible. The resulting solutions were implemented in system designs of current projects ("AuRora" FFG850459, "HydroMetha" FFG864578), which include linking renewable electricity to gas storages as well as integrating a coupled Co-electrolysis and Methanation plant into industrial processes. These developments lead to highly efficient management of process streams and novel heat exchanger networks. The oral presentation provides the theoretical background, application scenarios, SOE system design variants, BoP development, applied methods as well as results of current projects.

5:20 PM

(ICACC-S3-043-2020) Electrochemical Evaluation under High Temperature CO_2 and H_2O Co-Electrolysis

M. Heringer Boucas*[1]; J. Irvine[1]; L. Sammes[1]

1. University of St Andrews, Chemistry, United Kingdom

The utilization of carbon dioxide (CO_2) to produce green fuels from renewable electricity is an important research avenue for society these days. The Industrial CO_2 by-product is provided by cement manufacture, while the power needed for the conversion is supplied by the offshore wind. In co-electrolysis CO_2 and H_2O (steam) are consumed to mainly produce H_2/CO (synthesis gas). This study aims at evaluating various fuel electrode materials starting with Ni/YSZ and later investigating a range of perovskites such as Ni doped lanthanum-calcium-titanite (LCNT) and Cr doped LSM (LSCM). The LSM/YSZ was applied as the air electrode and YSZ as the thick electrolyte. The electrochemical evaluation (EIS and I-V curve) was carried out at 925°C with 50%H_2O supply under H_2 diluted in N_2 and pure H_2 varying the CO_2/H_2 flow rates for each case, at different voltages applied. Interface and microstructure were investigated by scanning electrons microscope (SEM). The best performance was achieved when wet CO_2 and diluted H_2 in N_2 were used at -1.3V applied. The Rp value encountered was equal to 0.42 $\Omega.cm^2$. It was noticed that with very little H_2, the performance on Ni reduces significantly. When sufficient H_2 is diluted in N_2, Rp and current density values present improvements. It seems mainly related to higher frequency in the polarization arc, suggesting the charge transfer was affected.

5:40 PM

(ICACC-S3-044-2020) Co-Electrolysis: From stack performance map to energy and cost efficient hydrocarbons production

M. Kusnezoff*[1]; S. Megel[1]; C. Rix[1]; P. Adam[1]; G. Herz[1]; E. Reichelt[1]; M. Jahn[1]; N. Trofimenko[1]; A. Michaelis[1]

1. Fraunhofer IKTS, Germany

Co-electrolysis is a unique selling point of high temperature electrolysis (SOEC) and provides opportunity to direct syngas production in composition needed for chemical conversion. The varying gas inlet composition needed for production of syngas with proper hydrogen and carbon monoxide content can affect the power

consumption of SOEC stack. The power consumption and operation of 10-cell SOEC stack utilizing electrolyte supported cell (ESC) and chromium based interconnect (CFY) has been investigated under different inlet compositions and temperatures. It was found that power consumption between stem electrolysis and co-electrolysis operation is caused mainly by open circuit voltage and not by change in stack resistance. High temperature electrolysis has clear advantages in terms of electrical energy consumption if process heat can be utilized for steam generation. Examples of exothermal processes which can be utilized as heat source are Sabatier reactor, Fischer Tropsch synthesis and Haber Bosch ammonia production. The process flow-chart for waxes production by Fischer Tropsch synthesis has been designed and optimized using ASPEN software delivering the total process efficiency estimations for different operationg conditions of stack. Finally the production costs of waxes has been estimated assuming reasonable electricity price, heat losses and known BoP properties.

S4: Armor Ceramics - Challenges and New Developments

Synthesis and Processing I & II

Room: St. Johns

Session Chairs: Lionel Vargas, US Army Research Laboratory; Nicholas Ku, CCDC - Army Research Laboratory

8:30 AM

(ICACC-S4-029-2020) Microstructure and Properties of superhard SiC-bonded diamond materials (Invited)

M. Herrmann*[1]

1. Fraunhofer IKTS, Germany

New, extremely wear-resistant and super hard materials are SiC-bonded diamond ceramics, which can be manufactured by reaction bonding of diamond by liquid silicon infiltration similar to the SiSiC process. Using this approach, components can be produced cost-effectively in large sizes and with complex shapes. They can also be produced as layered materials with SiC-bonded diamond only in areas where it is required. These materials are unique materials with high thermal conductivity (600 W/mK) and excellent abrasive wear resistance. This presentation presents the latest results on the thermal, mechanical and tribological properties of SiC bonded diamond composites as a function of microstructure. The materials show coefficients of friction and a wear behavior under tribological load corresponding to those of PCD and CVD-diamond coatings. Extremely high corrosion resistance was observed in acids, bases and hydrothermal conditions. Even in hot water an extremely low, actually not measurable subcritical crack growth was measured.

9:00 AM

(ICACC-S4-030-2020) Mechanical and thermal characteristics of interfaces and defects in diamond/SiC composites (Invited)

Y. Zhang[4]; C. Hsu[2]; T. Wang[1]; P. Karandikar[3]; C. Ni*[1]

1. University of Delaware, Department of Materials Science and Engineering, USA
2. University of Delaware, USA
3. M Cubed Technology, Inc., R&D, USA
4. University of Delaware, Material Science and Engineering, USA

Due to remarkable mechanical properties, lightweight, availability and unique photonic, electronic and thermal properties, diamond and SiC have received considerable research interest in the microelectronics industry and for structural applications for extreme environments. For large structures such as armors, semiconductor processing platforms, and high energy/power laser mirrors, monolithic or single crystal diamond or SiC are not suitable due to the limitation in dimension, machinability, and property tailorablity. Composites of diamond and SiC realized by advanced processing technologies have therefore become premier candidates for their optimal combination of properties at desired dimension and shape. As with any composite systems, interphase boundaries between diamond, SiC and Si are essential linkages and play critical roles in transferring mechanical force or thermal energy. The processing conditions for these composites which includes high temperature, gas species and pressure, can lead to undesirable phases governed by thermodynamics, and remain to be better understood and controlled. In this talk, I will discuss some common interfacial structures and defects in diamond/SiC composites characterized by SEM and TEM and share some results of mechanistic investigations of their properties and behaviors under *in-situ* mechanical load or heat flux of varied energy and power.

9:30 AM

(ICACC-S4-031-2020) Reaction Sintered Silicon Carbon Diamond Composites via Optimized Particle Distributions

A. A. DiGiovanni*[1]; J. LaSalvia[2]; K. D. Behler[3]; M. Guziewski[1]

1. US Army Research Laboratory, Ceramics and Transparent Materials, USA
2. U.S. Army Research Laboratory, USA
3. U.S. Army Research Laboratory, Multifunctional Materials Branch, USA

Historically, reaction-bonded silicon carbide and related composites have been realized successfully through infiltration of molten silicon that reacts with a carbon source to form a carbide at lower temperatures than required in conventional sintering. Diamond-carbide composites are highly dependent on lower temperatures owing to the metastability of diamond and its rapid conversion to non-diamond carbon above 1600 °C. This low-pressure investigation examines carefully the effect of silicon, diamond, and carbon particle distributions, their packing efficiency as measured by tap densometry, their reaction chemistry as characterized by differential thermal analysis on the formation of the resultant sintered body, the formed microstructure and phase distributions. Packing optimization and efficiency is modeled using a gravity driven LAMMPS POUR molecular dynamic code that accounts for particle mass, radius, and size of n-modal distributions with 1 <= n <= 3. Diamond starting particle sizes range between 10 and 180 microns. Residual porosity, silicon, unreacted phases, graphitization and resulting microstructures are analyzed with backscattered scanning electron microscopy on selected samples.

9:50 AM

(ICACC-S4-032-2020) Densification of Model Hot-Pressed Diamond-Ceramic Composites with Bi-Modal Diamond Particle Size

J. LaSalvia*[1]; K. D. Behler[1]; A. A. DiGiovanni[1]

1. U.S. Army Research Laboratory, FCDD-RLW-ME, USA

Low pressure/high temperature (LPHT) methods such as reaction bonding have been developed to produce low-cost diamond-ceramic composites with high diamond volume loadings, minimal graphitization, and low residual silicon for a range of structural, wear, and thermal management applications. As further exploration of LPHT methods, an initial study on the effects of the volumetric ratio of bi-modal size diamond particles, for a given total volume fraction, on the densification of model diamond-SiB_6 powder mixtures by hot-pressing is reported. Two diamond powders with particle sizes of 109 mm and 10-20 mm, respectively, were mixed with SiB_6 powder in various proportions corresponding to several total volume loadings and three relative volume fractions (at each total volume loading). After mixing, powders were hot-pressed for 1 hr at 1600°C under oxygen-gettered Ar. An LVDT recorded ram displacement from which densification history was determined. Resulting specimens were characterized for density (GeoPyc), phases (XRD), and microstructure (SEM). As expected, the percolation threshold

for total diamond volume loadings were higher for bi-modal versus mono-sized diamond particles. Further findings and experimental procedures will be discussed.

10:30 AM

(ICACC-S4-033-2020) Multimaterial Printing of Silica-Titania Glass: Prediction and Tuning of Ink Rheology (Invited)

N. Dudukovic*[1]

1. Lawrence Livermore National Laboratory, USA

3D printing of titania doped glass can impact applications ranging from microfluidics to optics. Multimaterial additive manufacturing (AM) enables incorporating diverse functionalities into advanced glass optics by spatially controlling dopant concentrations. The development of printable feedstocks relies upon trial and error, which is time-consuming, labor-intensive, and costly. Approaches to overcome these issues by quantifying the rheological behavior, and using it to inform material formulation and printing parameters for direct ink writing (DIW) of silica-titania glass are described. Printable silica-titania inks that can be processed into multimaterial transparent glass with spatial change in refractive index were developed. We investigate the relationship between material properties and printing performance, and develop approaches and predictive tools to enable multimaterial printing of gradient compositions. Three main requirements for successful printing of glass optics will be highlighted: (1) the ability to print void-free monoliths, (2) rheological and compositional compatibility for multimaterial printing, and (3) active micromixing of colloidal materials at high shear. This work was performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344 within the LDRD program 19-ERD-020. LLNL-ABS-770518.

11:00 AM

(ICACC-S4-034-2020) Additive Manufacturing of Transparent Armor Ceramics: One-Step Laser Direct Deposited Magnesium Aluminate Spinel Ceramics

J. Pappas[1]; X. Dong*[1]

1. Missouri University of Science & Technology, Mechanical and Aerospace Engineering, USA

Transparent ceramics are important for a wide range of applications including windows, domes, and high laser threshold optics. The traditional way of manufacturing these components relies on lengthy sintering synthetic procedures with nanopowders. Additive manufacturing (AM) is attractive for the fabrication of these components. This study investigates one-step laser direct deposition of transparent magnesium aluminate spinel (MAS) ceramics, a type of transparent armor materials. Dense MAS ceramic samples are successfully fabricated from a homogeneous mixture of alumina and magnesium oxide precursors without a need of lengthy preprocessing procedures normally required by traditional sintering techniques. The printed samples are verified to be dominated by a single MAS phase through X-ray diffraction peak analysis. The process achieves AM of transparent ceramics from micron-sized powders, instead of nanopowders needed in sintering. Cracking and residual porosity are the key issues affecting the transparency of the fabricated parts. Both issues can be greatly reduced through optimized processing conditions, with the parts shifting from being translucent to transparent. The results of the current study demonstrate the potential of the proposed method in direct printing transparent ceramic parts.

11:20 AM

(ICACC-S4-035-2020) Methylcellulose binder systems for direct ink writing of carbide ceramic suspensions

N. Ku*[1]; B. Gray[1]; J. Pelz[2]; A. T. Rosenberger[1]; L. Vargas[1]

1. CCDC - Army Research Laboratory, Ceramics and Transparent Materials Branch, USA
2. University of California, San Diego, MATS, USA

Direct ink writing (DIW) is an additive manufacturing (AM) method that permits the extrusion of multiple materials into complex structures. This technology can enable the development of mesoscale structures, such as functional gradients or biomimetic structures, which have been shown to exhibit unique fracture paths unlike those found in monolithic structures. However, extruding viscous suspensions of silicon carbide (SiC) and boron carbide (B_4C) in the DIW process remains a difficult challenge. This study investigates the rheological effects of solids loading, methylcellulose binder type, and binder content on both B_4C and SiC suspensions. Samples are tested on a rotational rheometer to determine viscosity as a function of shear rate as well as yield stress as a function of shear stress in an effort to identify an optimal suspension composition for printing. Binder burnout and sintering cycles are also investigated to develop a path forward to achieve fully dense, heterogeneous parts.

11:40 AM

(ICACC-S4-036-2020) Direct Ink Writing for Functionally Graded Ceramics

J. Pelz*[1]; N. Ku[2]; M. A. Meyers[1]; L. Vargas[2]

1. University of California, San Diego, MATS, USA
2. U.S. Army Research Laboratory, USA

Additive manufacturing of functionally graded boron carbide-silicon carbide composites is explored with the goal of improving damage tolerance through extrinsic mechanisms. Traditional powder processing techniques do not allow heterogeneous grading of composition and structure. Thus, a custom direct ink writing system was developed to enable simultaneous extrusion of multiple inks, comprised of high solids-loaded ceramic particulate suspensions, through a single nozzle. This novel technology utilizes an auger to produce heterogeneity in both chemical composition and geometry. Auger design parameters were studied and optimized to enable control over composition variation across multiple length-scales. Micro- and mesostructures were characterized to identify mixing resolution. Several types of functionally graded structures were formed, densified, and tested to determine the effects of heterogenous grading on mechanical properties.

Synthesis and Processing III & IV

Room: St. Johns
Session Chairs: Steve Kilczewski, Army Research Laboratory; Kristopher Behler, U.S. Army Research Lab

1:30 PM

(ICACC-S4-037-2020) Simultaneous improvements of strength and toughness in topologically interlocked ceramics

F. Barthelat*[1]; M. Mirkhalaf[2]

1. University of Colorado, Mechanical Engineering, USA
2. University of Sydney, Mechanical Engineering, Australia

Topologically interlocked materials (TIMs) are an emerging class of architectured materials based on stiff building blocks of well-controlled geometries which can slide, rotate or interlock collectively providing a wealth of tunable mechanisms, precise structural properties and functionalities. TIMs are typically ten times more impact resistant than their monolithic form, but this improvement usually comes at the expense of strength. Here we used 3D printing and

replica casting to explore fifteen designs of architectured ceramic panels based on platonic shapes and their truncated versions. We tested the panels in quasi-static and impact conditions with stereo-imaging, image correlation and 3D reconstruction to monitor the displacements and rotations of individual blocks. We report for the first time a design based on octahedral blocks which is not only tougher (x50) but also stronger (x1.2) than monolithic plates of the same material. This result suggests that there is no upper bound for strength and toughness in TIMs, unveiling their tremendous potential as structural and multifunctional materials. Based on our experiments we propose a non-dimensional "interlocking parameter" which could guide the exploration of future architectured systems.

1:50 PM

(ICACC-S4-038-2020) Effect of Sintering Parameters on Room-Temperature Injection Molded, Pressurelessly Sintered Boron Carbide Components

E. Weaver*[1]; R. Trice[2]; J. P. Youngblood[2]

1. Purdue University, USA
2. Purdue University, Department of Materials Engineering, USA

Boron carbide is an ideal material for lightweight armor applications due to its excellent mechanical properties, such as low density, high hardness, and high Young's modulus. However, the poor pressureless sintering behavior of boron carbide currently limits its widespread use, and current efforts are limited to simple geometries. Room-temperature injection molding, combined with pressureless sintering, allows for the formation of complex, near net shaped components. Achieving high relative densities and good mechanical properties in these pressurelessly sintered parts is challenging, and there are many sintering parameters that must be considered. Boron carbide parts have been produced using a variety of sintering parameters. The effect of these parameters on the microstructure, relative density, and mechanical properties on the boron carbide parts is evaluated and discussed.

2:10 PM

(ICACC-S4-039-2020) Optimizing Dispersion of Boron Carbide and Sintering Aids for Colloidal Processing and Pressureless Sintering

T. Marconie*[1]; J. P. Youngblood[1]; R. Trice[1]

1. Purdue University, Department of Materials Engineering, USA

Boron carbide is an attractive material for armor applications due to its high hardness and low density. However, difficulties with sintering boron carbide to full density often limit parts to simple geometries. Colloidal processing and pressureless sintering with sintering aids are potential methods to create dense and complex-shaped boron carbide parts. To enable this processing route, it is critical to create highly-loaded suspensions with good dispersion of both boron carbide and sintering aid particles to obtain ideal densities, microstructures, and mechanical properties. In this work, dispersion of boron carbide and sintering aids in highly-loaded, aqueous suspensions are studied to determine the effect on sintered properties.

2:30 PM

(ICACC-S4-040-2020) Structures and Compositions of Grain Boundary Complexions in Eu-Doped Boron Suboxide

C. Marvel*[1]; K. D. Behler[2]; J. Synowczynski-Dunn[2]; B. C. Hornbuckle[2]; J. LaSalvia[2]; M. Harmer[1]

1. Lehigh University, Department of Material Science and Engineering, USA
2. U.S. Army Research Lab, FCDD-RLW-ME, USA

Boron suboxide is a promising armor ceramic because of its low density and high hardness, but it exhibits poor fracture toughness. One strategy to improve fracture toughness, and the goal of this work, is to incorporate a variety of grain boundary complexions, including nanolayer films, to weaken grain boundaries and promote alternate fracture mechanisms including crack deflection. As-received boron suboxide powders were co-doped with silica and europium oxide additives up to 5 vol.% (silica) and hot-pressed at 1850°C for 3 hours at ~54 MPa in an attempt to form disordered grain boundary complexions. The resultant microstructures and grain boundary complexions were determined by applying aberration-corrected scanning transmission electron microscopy in combination with ζ-factor microanalysis. Grain boundary misorientations were also determined using precession electron diffraction as the average grain size was on the order of 200 nm. Overall, several distinct grain boundary complexions were observed including ordered monolayers and disordered nanolayers. To verify if the grain boundary compositions and structures are in equilibrium, the experimentally measured grain boundary compositions and structures of each complexion type were used as input parameters for first-principles density functional theory calculations. Experimental and computational results will be presented.

3:10 PM

(ICACC-S4-041-2020) A study of the effect of magnesium on boron suboxide densification and mechanical properties

T. Shoulders*[1]; K. D. Behler[1]; J. LaSalvia[1]

1. CCDC Army Research Laboratory, USA

Boron suboxide is a promising next-generation armor ceramic with 2kg Knoop hardness approaching 20 GPa. Densification is difficult and relies upon hot-pressing B_6O powders with oxide additives or reaction hot-pressing of boron and B_2O_3. In the latter case, the highest levels of densification are achieved with an excess of B_2O_3, a volatile species at typical densification temperatures above 1700 °C. Previous work has shown that commonly used powder sources can lead to the formation of magnesium-containing secondary phases, in some cases leading to enhanced hardness and toughness. The current study aims to understand this relationship between secondary phases and mechanical properties. Three different grades of commercially available amorphous boron powders with nominal magnesium impurity levels of 3, 6, and 8 wt% along with high-purity crystalline boron powders with 0, 2, and 4 w% added magnesium content were used as boron sources. Each boron powder was mixed in a molar ratio of 10:1 with anhydrous B_2O_3 and hot-pressed at 1800 °C for 1h. Bulk elemental analysis (GD-MS and XRF) and phase analysis (x-ray diffraction) were performed on all powders and hot-pressed samples. Ceramic samples were also evaluated with Knoop indentation hardness and Vickers indentation toughness. Correlations in densification behavior, phase content and mechanical properties are analyzed and presented.

3:30 PM

(ICACC-S4-042-2020) Effect of Heating Rate, Temperature and Additive Content on the Densification and Microstructure of Hot-Pressed Boron Suboxide (B_6O)

H. E. Payne*[1]; K. D. Behler[1]; J. LaSalvia[1]; C. Marvel[2]; T. Shoulders[1]; M. Harmer[2]

1. US Army Research Laboratory, FCDD-RLW-ME, USA
2. Lehigh University, Department of Material Science and Engineering, USA

Hot-pressed Boron suboxide (B_6O) has previously been shown to densify using heating rates of up to 50°C/min within the temperature range of 1750 – 1850°C with a uniaxial applied load of ~54 MPa held for up to 3 hours in both inert and vacuum conditions. The addition of silica based sintering additives of up to 5 vol.% silica were used. While mass loss is expected due to the decomposition above 1765°C, silica additives along with a high applied load have been shown to reduce this effect as well as the conversion of B_6O into boron carbide (B_4C) due to powder-die interactions. While B_6O has been

successfully hot-pressed and presented previously using a 1 vol.% silica sintering aid at 1850°C with a ramp of 50°C/min resulting in > 20.5 GPa HK2, a more in depth study of the processing parameters is needed. In this study the effects of heating rate, applied load (ranging from 20 MPa to 54 MPa), the maximum hold temperature and the additive content will be presented with regards to the final density, the densification behavior, the mass loss, the poorly densified B_4C region, the microstructure and hardness of the B_6O. This study determines a parameter space that promotes near full density with minimal decomposition and conversion to B_4C. Experimental procedures and results will be presented.

3:50 PM

(ICACC-S4-043-2020) Flash Sintering of Boron Carbide

A. T. Rosenberger*[1]; R. E. Brennan[2]; S. Raju[1]; A. L. Fry[3]

1. Oak Ridge Associated Universities, USA
2. US Army Research Laboratory, USA
3. U.S. Army Research Laboratory, ORISE, USA

Flash sintering of B_4C has seen limited investigation. A flash sintering furnace setup capable of applying either DC or AC electric fields with current or voltage control was developed. Initial experiments under fixed DC voltage conditions observed flash sintering in B_4C at low furnace temperatures of 400-450°C, but with densification localized to a small diameter channel between the electrodes. The effects of temperature profile, electrical parameters, electrical contact, and specimen geometry on densification and current localization were systemically evaluated. A 1V/s voltage ramp applied at constant temperature of 550°C was found to prevent current localization, but did not result in uniform densification. Specimen geometry had a significant effect on current localization, attributed to temperature gradients within the specimen. The power density is also proposed as a mechanism that influences densification in flash sintering of B_4C.

4:10 PM

(ICACC-S4-044-2020) Multi-Phase Armor Production and Characterization Involving B_4C–SiC–TiC with SPS Technique

G. Uysal Sapanci*[1]

1. ROKETSAN, Turkey

B_4C is commonly used material for armor applications due to its light and high hardness. However, the high cost is one of the most important obstacles in the front. Sintering of B_4C requires sintering with at 2200C to bring it to the theoretical density. However, the mechanical properties of B_4C are not good enough to be used in industrial applications. Numerous studies have been carried out on the production of B_4C containing composite materials by the addition of different compounds such as SiC, TiC. The addition of these compounds improves properties and reduces the sintering temperature. In this study, optimum B_4C-SiC-TiC ceramic composite compositions were designed and produced by SPS bearing in mind the hardness–toughness–cost–lightness-performance relationship. The addition of SiC did not affect the hardness and toughness, but the addition of TiC resulted in increased toughness in spite of decreased hardness. When microstructure analyzes and XRD patterns were evaluated together, residual graphite and SiC were found to exist in B_4C, and when TiC was added, residual graphite and small amounts of SiC, as well as TiC and B_4C, reacted to form TiB_2. When SiC and TiC were added together, hardness decreased whereas fracture toughness increased. Due to the fact that B_4C is a light material, all additives added increase the weight, while the costs were reduced because all additives are cheaper than B_4C.

S5: Next Generation Bioceramics and Biocomposites

Next Generation Bioceramics IV

Room: Coquina Salon C
Session Chairs: Nikhil Kamboj, Tallinn University of Technology; Ashutosh Goel, Rutgers University; Bryan McEntire, SINTX Technologies

8:30 AM

(ICACC-S5-026-2020) Biological Crystallization of Ultrahard Teeth and Translation to Multi-Functional Materials

D. Kisailus*[1]

1. University of California, Riverside, Chemical and Environmental Engineering, USA

Natural systems have evolved efficient strategies to synthesize composites from a limited selection of available materials that often exhibit exceptional mechanical properties that are frequently superior to those exhibited by engineering materials. These systems have accomplished this by establishing controlled synthesis and hierarchical assembly of nano- to micro-scaled building blocks. This requires organic that is used to transport mineral precursors to organic scaffolds, which not only precisely guide the formation and phase development of minerals, but also significantly improve the mechanical performance of otherwise brittle materials. Here, we investigate the formation of heavily crystallized radular teeth the chitons, a group of elongated mollusks that graze on hard substrates for algae. From theinvestigation of synthesis-structure-property relationships in these unique organisms, we are now developing and fabricating multifunctional engineering materials for energy conversion and storage. We discuss the crystallization of these materials and their subsequent impact on performance.

8:50 AM

(ICACC-S5-027-2020) Nitric Oxide Releasing Ceramics for Prevention of Periprosthetic Joint Infections (Invited)

B. J. McEntire*[1]; G. Pezzotti[2]

1. SINTX Technologies, Research & Development, USA
2. Kyoto Institute of Technololgy, Ceramic Physics Laboratory, Materials Science Division, Japan

Perioperative infections are leading causes for the failure of orthopedic implants, with reported rates of between 2.7 and 18%. Their incidence is expected to increase due to demographics and the prevalence of antibiotic resistant bacteria. One preventive approach that has generated interest is the use of nitric oxide (NO) releasing polymeric coated implants. Unfortunately, their clinical use has been limited due to inadequate NO payloads and uncontrolled release. Silicon nitride (Si_3N_4) is a bioceramic with inherent NO donor properties that may overcome some of these limitations. Si_3N_4 undergoes slight surface dissolution which releases minute amounts of silica (SiO_2) and ammonia (NH_3) into the biological milieu. Then, a series of off-stoichiometric reactions generates free electrons and various short-lived reactive nitrogen species (RNS, i.e., NO, N_2O, $^{-}OO\text{-}N{=}O$). These moieties penetrate the bacterial membrane and induce nitrosative stress which eventually kills the pathogen. The NO releasing capabilities of Si_3N_4 may make it an effective short- and long-term orthopedic implant in the prevention of periprosthetic joint infections.

9:10 AM

(ICACC-S5-028-2020) Additive manufacturing of silicon-wollastonite/bioactive glass based biomaterials by Selective Laser Melting (Invited)

N. K. Kamboj*[1]; M. A. Rodríguez Barbero[2]; C. Rodrigo[2]; J. Kazantseva[3]; I. Hussainova[1]

1. Tallinn University of Technology, Mechanical and Industrial Engineering, Estonia
2. Instituto de Ceramica y Vidrio (ICV-CSIC), Spain
3. Cellln Technologies LLC, Tallinn, Estonia

The repair and regeneration of load-bearing defects by bioceramic scaffolds remain a significant clinical challenge resulting from disease or trauma. The needs for repair and regeneration are one of the key assets for the synthesis of the scaffolds for load-bearing repair applications. The criteria include (1) porous structure, (2) geometry, (3) biodegradability, (4) biomolecules delivery, (5) mechanical competence, which is generally seen as a problem. In-order to fulfill all these requirements, we developed silicon-wollastonite/bioglass based composite scaffolds with high strength and different geometries via selective laser melting which can be easily tailored with shape and size to the diseased or injured area avoiding the binder addition and post-processing stages. The scaffolds showed a high bioactivity and controlled growth of the hydroxyapatite-like layer on the surface of the structures. The as-obtained scaffolds significantly expresses osteogenic genes (RUNX2, OSAD and OCN) and moreover the inflammatory factors were also studied onto the scaffolds. The findings demonstrate that as-produced composite scaffolds can pave the way for treating cortical bone and maxillofacial defects for large load-bearing repair applications.

9:30 AM

(ICACC-S5-029-2020) Modified polymer-derived SiOC ceramics for medical applications

M. Gaweda*[1]; W. Simka[2]; M. Sowa[2]; A. R. Boccaccini[3]; M. T. Sitarz[1]

1. AGH University of Science and Technology, Faculty of Materials Science and Ceramics, Poland
2. Silesian University of Technology, Department of Inorganic Chemistry, Analytical Chemistry and Electrochemistry Faculty of Chemistry, Poland
3. University of Erlangen-Nuremberg, Institute of Biomaterials, Germany

The common name of the SiOC-based ceramics – black glass – origins in the presence of the so-called free carbon phase in the material. However, the carbon ions bonded into the amorphous silica matrix have a supreme role in the process of shaping properties of the black glasses such as high thermal and chemical stability, corrosion resistance and the durability of the material. Preparation of amorphous and homogenous SiOC ceramics is possible by ceramization of the certain polymers containing silicon-carbon bond in their structure. In this work, ladder-like polisilsesquioxanes obtained by the sol-gel method were selected. Due to the modifiability of the chosen path, it is possible to implement other ions into the structure of the material to enhance properties or ad a new one. From the viewpoint of designing materials of high biocompatibility and bioactivity, most attractive is the application of bioelements. As the main point of the study, the modification by phosphate ions was chosen. The materials were obtained in the form of layers and examined to describe their structure, microstructure and functional properties. Most crucial in vitro tests of bioactivity (the so-called Kokubo test) and biocompatibility (LDH-activity, WST-8) were conducted on the most promising materials. The results showed the impact of the modification on the particular properties of the polymer-derived SiOC ceramics.

10:10 AM

(ICACC-S5-030-2020) Structural design of borosilicate based bioactive glasses (Invited)

A. Goel*[1]; N. Stone-Weiss[3]; R. Youngman[2]; H. Eckert[6]; A. Pedone[5]; E. M. Pierce[4]; N. J. Smith[7]

1. Rutgers University, USA
2. Corning Incorporated, Science & Technology Division, USA
3. Rutgers University, Materials Science and Engineering, USA
4. Oak Ridge National Lab, Environmental Sciences Division, USA
5. University of Modena and Reggio Emilia, Italy
6. University of Muenster, Germany
7. Corning Incorporated, USA

A paradigm shift from the "trial–and–error" approach to "materials–by–design" approach is required to develop new-generations of resorbable bioactive glasses with controlled release of functional ions tailored for specific patients and disease states, whereby material functions and properties can be predicted from first principles. Realizing this goal, however, requires a thorough understanding of the complex sequence of reactions that control the dissolution kinetics of bioactive glasses and the structural drivers that govern them. Using a mix of both experimental and computational results describing the structure and dissolution behavior of glasses designed in the Na2O–B2O3–P2O5–SiO2 glass system, the presentation will highlight some major experimental challenges or choices that need to be carefully navigated to unearth the mechanisms governing the chemical degradation behavior and kinetics of boron-containing bioactive glasses, and to accurately determine the composition–structure-property relationships.

10:30 AM

(ICACC-S5-031-2020) Impact-resistant biological coatings of the mantis shrimp dactyl club

D. Kisailus*[1]

1. University of California, Riverside, Chemical and Environmental Engineering, USA

There is an urgent need for light weight high-performance impact/ballistic resistant materials in many engineering areas. Natural systems have provided invaluable information for new material designs with broad application. One incredible example is found in the dactyl clubs of the smashing mantis shrimp. These clubs can reach ~20 m/s at an acceleration of ~10,000g, striking thousands of times without breaking, thus displaying significant high strain rate impact resistance. Here, we utilize a combination of microscopy, spectroscopy and nanomechanical testing to uncover ultrastructure-mechanical property relationships in the outermost coating. The ultrathin (~ 70 μm) coatings found in the surface of these dactyl clubs consist of densely packed (~ 88 vol%) mineral nanoparticles in an organic matrix. We reveal the particles are mesocrystalline and attached at low angle grain boundaries. We observed significant energy dissipating mechanisms including particle rotation, translation and fracture, and plastic deformation of organic phase. Imperfections in the nanocrystals such as dislocations and amorphization are also generated during the impact events. Finally, the observed damping behavior of these HAP-based nanoparticles are greater than that demonstrated in technical ceramics as well as metals. We synthesize biomimetic structures that implement these designs for engineered coatings.

10:50 AM

(ICACC-S5-032-2020) Synthesis and characterization of electro thermally poled Li_2O doped Borate- based bioactive glass

S. Jaiswal*[1]

1. IIT (BHU), Varanasi, Ceramic Engineering, India

Electrically conductive borate-based, 1393 bioactive glass in substitution to Li_2O was prepared by the conventional melting technique in an Alumina crucible at 1000°C for 2 hours. The AC

conductivity and permittivity of Li_2O substituted glass samples at temperatures from room temperature to 500°C and 0.1-1MHZ frequency were measured by impedance spectroscopy. It was observed that the substitution of Li_2O for B_2O_3 enhanced the electrical properties and bioactivity of bioactive glasses. The glass samples were electro thermally poled by applying voltages of 10 kV at 500°Cfor 2 hours. The impact of poling on the bioactivity was investigated by XRD, SEM/EDS, and AFM analysis. The poling prompts morphological changes in the boron-rich layer and to changes in the development rate of amorphous calcium phosphate. Such a result shows that the combined effect of change in chemical composition and electrothermal poling can be utilized to increase the bioactivity of bioactive glasses in terms of increasing the rate of formation of HCA.

11:10 AM

(ICACC-S5-033-2020) Studies on In vitro bioactivity, Cytocompatibility and mechanochemical properties of SrO modified 1393 glass scaffold

A. Ali*[1]

1. Indian Institute of Technology(BHU), Ceramic Engineering, India

This investigation deals with evaluation of desired physico-chemical and biological compatibility of SrO substituted 1393 glass scaffolds. Strontium substituted 1393 bioactive glass scaffolds with general formula $54.6SiO_2$-$6Na_2O$-$7.9K_2O$-$7.7MgO$-(22-X) CaO-$1.74P_2O_5$-XSrO (mol%; where X=1.1, 5.5, 11, 22) have been prepared (S1, S2, S3, S4 and S5) through sol gel method followed by foam replica. In vitro bioactivity study confirmed by XRD, FTIR, SEM-EDX, and pH showed better bioactivity after SrO addition to the parent glass system. Assessment of cellular attachment, viability and metabolic activity using mouse fibroblast L929 was also found better for strontium incorporated glasses. Physico-mechanical properties like compressive and flexural strength and modulus of elasticity with the average apparent porosities (AP) of '55-60%' were also significantly increased with the increase in SrO percentage in the scaffolds. In vitro studies insinuates combine properties of calcium and strontium in newly formed apatites $[Ca_{(10-x)}Sr_x(PO_4)_6(OH)_2]$ responsible for better bioactivity and cytocompatibility. The results demonstrated that the newly formed apatites are better in metabolic activity, in vitro bioactivity, cellular compatibility and mechanical performance, which is a good indication for their consideration in neo bone tissue regenerative application.

11:30 AM

(ICACC-S5-034-2020) ZnO modified 1393 bioactive scaffolds with enhanced cytocompatibility and mechanical performance

R. Pyare*[1]

1. Indian Institute of Technology(BHU), Ceramic Engineering, India

Zinc is one of the most essential and vital trace elements for our skeletal system and is accountable for formation, development and maintenance of healthy bones. In present investigation we have fabricated zinc based alkali phosphosilicate 1393 glass scaffolds by traditional melt-quench, followed by foam replica technique. This experiment deals with the evaluation of ZnO modified porous (Apparent porosity >60%) 1393 glass scaffolds (Z1, Z2, Z3 and Z4) with the general formula $(54.6-X)SiO_2$-$6Na_2O$-$7.9K_2O$-$7.7MgO$-$22CaO$-$1.74P_2O_5$-XZnO (where X=0.0, 0.5, 1.0, 2.0; mol%) prepared through melt route followed by foam replication for its desired physico-chemical and biological properties. In vitro bioactivity of these scaffolds was evaluated using XRD, FTIR, SEM-EDS and behavioral changes in pH. Cell adhesion and biological compatibility study was carried out using mouse fibroblast L929 cells via MTT assay. The results indicate that the ZnO substituted 1393 derived scaffolds have enhanced the apatite like calcium phosphate nodules formation, biomineralization, cytocompatibility and physico-mechanical properties and thus we found our new ZnO substituted 1393 glass scaffold seems to be a suitable candidate for neo-bone tissue regenerative medicinal application.

S6: Advanced Materials and Technologies for Rechargeable Energy Storage

Li-ion Battery Cathodes

Room: Tomoka A

Session Chairs: Jagjit Nanda, Oak Ridge National Lab; Ilias Belharouak, Oak Ridge National Lab

8:30 AM

(ICACC-S6-024-2020) Advanced Characterisations of Nanostructured Li-rich Disordered Rock-salts (Invited)

M. Diaz Lopez*[1]; P. Chater[2]; H. Playford[1]; Y. Joly[3]; P. Bordet[3]; V. Pralong[4]

1. STFC Rutherford Appleton Laboratory, ISIS, United Kingdom
2. Diamond light source, United Kingdom
3. Institut Néel, CNRS, France
4. CNRS ENSICAEN, France

Li-rich cation disordered rocksalts (RS) are an emerging family of cathode materials displaying higher capacities (200-350 mAh g^{-1}) than Li-rich layered oxides (150-250 mAh g^{-1}). Disordered RS can present multivalent transition metals, mixed O^{2-}/F^- anions or oxygen redox, [1–3] offering hope to significantly improve the performance of rechargeable Li-ion batteries. Our work focuses on the full description of structure and charge evolution of nanostructured RS synthesized by mechanochemical alloying. We have carried out a comprehensive study combining neutron and X-ray total scattering,[3] advanced High Energy Resolved Fluorescence Detected X-ray absorption and emission spectroscopy[4] to understand their performance. Additionally, data collections were carried out operando using new cells designs with minimal parasitic scattering from the cell. This study provides essential information for understanding the performance of nanostructured RS with great implications on the design and development of future cathode materials.

9:00 AM

(ICACC-S6-025-2020) Towards cobalt free cathode materials for Lithium ion batteries (Invited)

M. Wohlfahrt-Mehrens*[1]; P. Axmann[1]; M. Mancini[1]; N. Jobst[1]

1. ZSW - Zentrum für Sonnenenergie- und Wasserstoff-Forschung, Baden-Württemberg, Germany

Lithium ion batteries are widely used in many applications and are the most promising energy storage systems for future mobility. Nevertheless they still require further improvements in order to meet the energy and cost requirements for the next generation of electric vehicles. The development of cobalt free cathode materials with high capacity and high voltage is necessary. Co-free, Li-rich $Li_{1+x}Ni_{0.5}Mn_{1.5}O_{4.0}$ (0<x<1) compounds are very promising candidates for high energy applications. The lithium–nickel–manganese oxide compounds can be tailored with respect to composition and crystal structure in order to reach high capacities about 250 mAh g^{-1} with good cycling stability. We present full cell data with $Li_{1+x}Ni_{0.5}Mn_{1.5}O_{4.0}$ (0<x<1) compounds as cathode materials and either graphite or silicon/carbon composites as anodes [1]. $LiNi_{0.5}Mn_{1.5}O_4$/$Li_{1.3}Ni_{0.5}Mn_{1.5}O_4$ blend cathodes with adapted extra lithium can be used to compensate irreversible capacity losses of silicon based anodes in full cells. We will also present full cell data using various electrolytes and will discuss the main aging mechanisms in full cells. Acknowledgment: The authors acknowledge financial support from BMBF within the LiEcoSafe project under contract number 03X4636A.

9:30 AM

(ICACC-S6-026-2020) Interface Modification To Enhance Disordered Li-Rich Material Performances (Invited)

J. Colin*[1]; D. Peralta[1]; A. Boulineau[1]; J. Ducros[1]; J. Martin[1]

1. Université Grenoble Alpes, CEA-LITEN, France

As the development of electric vehicles has become an important economical and political issue, the look for higher energy material has never been so important. However the development of classic layered cathode with increasing Ni content will soon reach its limit. In addition, the use of critical raw materials (especially cobalt) for battery manufacturing has also become a concern, so that only cobalt-free materials may be envisaged. Only few materials seem able to cope with these specifications: lithium rich layered oxide, 5V spinel and the disordered Li-Rich materials as Li_2VO_2F. The latter one presents a much higher theoretical capacity of 462mAh/g but also work at lower potential [1]. However, it presents a strong capacity fading that has been ascribed to electrolyte stability. We will present the results of two strategies that have been applied to modify the reactivity at the material/electrolyte interface. In a first one electrolyte degradation has been studied and additives have been propose to stabilize it. In a second one we developed a coating strategy with AlF_3. Such coating strategy requires a adapted process due to the metastability of the material that degrades upon air/moisture exposure and during thermal treatment. The electrochemical performances as well as the surface chemistry will be discussed.

Solid Electrolytes

Room: Tomoka A

Session Chairs: Olivier Guillon, Forschungszentrum Juelich; Palani Balaya, National University of Singapore

10:20 AM

(ICACC-S6-027-2020) Processing of LATP-Based Solid Electrolytes for All-Solid-State Li-Ion Batteries (Invited)

M. J. Hoffmann*[1]; N. Schiffmann[1]; E. C. Bucharsky[1]; G. Schell[1]

1. Karlsruhe Institute of Technology (KIT), Institute of Applied Materials (IAM-KWT), Germany

Lithium titanium aluminium phosphate (LATP) is a potential candidate as solid state electrolyte with a high Li-ion conductivity. LATP with the basic composition $Li_{1,3}\ Al_{0,3}Ti_{1,7}(PO_4)_3$ was prepared by a modified sol gel method. The compositional range was systematically varied to study the impact on the sintering behaviour and the microstructural evolution. Complete densification has been achieved by pressureless sintering in the temperature interval from 800°C to 1100°C and soaking times of 10 to 480 mins. The resulting phase composition and crystal structure was obtained from XRD analysis. Electrical properties, such as ionic conductivity, were determined by impedance spectroscopy in the frequency range from 0.1 Hz to 1 MHz. A maximum conductivity in the order of 10^{-3} S/cm at room temperature is measured for different process parameters resulting in a different phase assemblage and relative density. Furthermore, a strong anisotropy in thermal expansion is observed, causing microcracking in the coarse-grained materials, which finally leads to a reduction in ionic conductivity. Finally, with regard to the intended use of LATP as a solid electrolyte in a battery, thin sheets were prepared by tape casting which were characterized in terms of density, structure and ionic conductivity. Potential strategies for a bonding between electrolyte and electrodes are discussed.

10:50 AM

(ICACC-S6-028-2020) Understanding Li Plating/Stripping Behaviors on Oxide Solid Electrolytes using the In-Situ SEM Technique (Invited)

M. Motoyama*[1]

1. Nagoya University, Department of Materials Design Innovation Engineering, Japan

Oxide solid electrolytes such as $Li_7La_3Zr_2O_{12}$ (LLZ) have been expected to be able to prevent the dendrite growth of Li because their shear moduli are enough large (usually more than 30 GPa). However, for any all-solid-state cells, the short-circuit event occurs during Li plating/stripping cycles. The reason for this has not yet been understood. Hence, in this study we aim at obtaining fundamental understandings on the Li plating/stripping processes for oxide solid electrolytes using an in-situ scanning electron microscope (SEM) observation technique. The voltage transient and in-situ SEM images were measured during Li plating on a Cu-coated-Ta-doped LLZ plate at 100 μA cm^{-2} at room temperature. Li whiskers started growing after the voltage peak. After 450 seconds, the absolute voltage suddenly dropped off to almost 0 V. This means the short-circuiting event occurred. It was found that local plating current density likely reached almost 100 mA cm^{-2}. It is considered that a significant increase in local plating current density caused the short-circuiting event. We will also show the results on the nucleation and growth of Li on LiPON glass electrolyte, which does not contain grain boundaries for comparison.

11:20 AM

(ICACC-S6-029-2020) Understanding Li-ion transport and conductivity in Ga- and Al-substituted garnet-type $Li_7La_3Zr_2O_{12}$ solid electrolytes using atomistic simulations (Invited)

J. Carrasco*[1]; F. Garcia Daza[2]; M. Bonilla[2]; E. Akhmatskaya[2]

1. CIC Energigune, Spain
2. Basque Center for Applied Mathematics, Spain

$Li_7La_3Zr_2O_{12}$ is a promising solid electrolyte for solid-state Li-ion batteries. However, fast Li-ion mobility required for battery applications only emerges at high temperatures, upon a phase transition to cubic structure. A well-known strategy to stabilize the cubic phase at room temperature relies on Li^+ substitution by Al^{3+} or Ga^{3+}. Yet, fundamental aspects regarding this aliovalent substitution remain poorly understood. We examine here why, despite having the same formal charge, Ga^{3+} substitution yields higher conductivities (10^{-3} S/cm) than Al^{3+} (10^{-4} S/cm). To this end, we combine for the first time molecular dynamic simulations and advanced hybrid Monte Carlo sampling techniques to unveil the atomistic origin of this phenomenon. We find Ga^{3+} ions tend to allow limited Li^+ diffusion within their immediate surroundings, whereas Al^{3+} induce a complete blockage of neighboring Li^+ diffusion paths. The effect is magnified at low temperatures and explains the higher conductivities observed for Ga-substituted garnets. We also use an effective medium approximation and comparison with reported experimental data to estimate the conductivities of cubic/tetragonal phase mixtures. We put forward that phase coexistence could have a significant impact on the conductivity of Al-substituted $Li_7La_3Zr_2O_{12}$, particularly at low Al contents.

11:50 AM

(ICACC-S6-030-2020) Exploration of the system Li-P-S-O

A. Neveu*[1]; C. Jordy[2]; V. Pelé[2]; V. Pralong[1]

1. CNRS ENSICAEN, France
2. SAFT, France

Li-ion batteries are nowadays mature systems whose production and use are constantly increasing. However, the electrolyte used in current lithium batteries is composed of lithium salt dissolved in an organic solvent. The latter poses safety problems, particularly

during overvoltage. One solution considered to make the battery safer would be to replace the solvents with an ionic conductive solid compound. This is why in recent years scientists have been trying to develop these solid systems. Several families of materials have been developing and have led to major improvements in this technology (NASICON, perovskites, Garnets). Several compounds were discovered in the pseudo-binary diagram xLi_2S-$(x-1)P_2S_5$ such as $Li_4P_2S_6$, $Li_7P_3S_{11}$, Li_3PS_4, Li_7PS_6. In 2011, R. Kanno and al. had discovered a new phase: $Li_{10}GeP_2S_{12}$ showing ionic conduction of 12 mS/cm. Unfortunately, this structure is unstable against lithium metal and germanium remains a very expensive element. In order to improve the stability of this structure, a partial substitution of sulfur with oxygen was made and allowed 7 cycles to be performed with lithium metal. Very recently, the germanium-free phase $Li_{9.6}P_3S_{12}$ has been obtained and has better stability against lithium at the price of lower conductivity. Thus, the $Li_{3.2}PS_{3.7}O_{0.3}$ phase seems to be a promising phase from a conductivity and stability point of view.

12:10 PM

(ICACC-S6-031-2020) Development of air stable Li-La-Zirconate (cubic garnet) based solid electrolyte for all-solid-state Li-ion batteries

S. Kobi*[1]; A. Verma[1]; A. Mukhopadhyay[1]

1. Indian Institute of Technology Bombay, Metallurgical Engineering and Materials Science, India

Despite being promising as a potential solid electrolyte for Li-ion batteries, Li-La-zirconate (LLZO) shows instabilities in stoichiometry, phase and integrity upon exposure to atmospheric moisture and CO_2. We have obtained new understandings on such instability of sintered Al-doped LLZO based on systematic sets of studies conducted during storage in ambient air for different durations. Exposure to air under ambient conditions led to the formation of Li_2CO_3 layer on the surface and $La_2Zr_2O_7$ in the bulk. Internal stresses associated with the formation of $La_2Zr_2O_7$ from within the bulk of LLZO caused spontaneous cracking, fracture and, eventually, disintegration. Concomitantly, the Li-ion conductivity decreased by ~3 orders of magnitude upon exposure to air for ~25 days. Following this up, our more recent work has indicated that when Al is replaced by Mg as the dopant, the desired cubic garnet phase can still be stabilized at room temperature and similar Li-ion conductivity be obtained for sintered LLZO. More importantly, formation of the deleterious phases and associated cracking upon long term exposure to air can be considerably supressed (and avoided) by Mg-doping; thus causing no notable decrease in the Li-ion conductivity upon exposure for ~25 days. Accordingly, Mg-doped LLZO is being envisaged as a promising air stable solid electrolyte for Li-ion batteries.

Beyond Li-ion battery

Room: Tomoka A

Session Chairs: Chongmin Wang, Pacific Northwest National Lab; Dany Carlier, ICMCB

1:30 PM

(ICACC-S6-032-2020) Development of Electrode Materials for Multivalent Rechargeable Battery (Invited)

Y. Orikasa*[1]

1. Ritsumeikan University, Applied Chemistry, Japan

Multivalent-ion rechargeable batteries are promising candidates as the next generation of rechargeable batteries. The advantages of metal anode such as magnesium or calcium are their high theoretical capacity, available stocks and the absence of dendrite formation. For an intercalation host, the use of multivalent cations increases charge carrier density and reduces the occupation of host-structure sites by charge carrier cations, improving key aspects of device performance such as energy density. However, multivalent ions exhibit slow diffusion kinetics in solid electrodes and intercalation hosts that can operate at room temperature are currently limited to Chevrel structured compounds [1]. In this presentation, the key aspects of designing electrode materials will be introduced. Although the radius of the magnesium-ion (72 pm) is almost identical to that of the lithium-ion (76 pm in a hexagonally-coordinated environment), the divalent character of magnesium-ion double the charge density over that of the monovalent lithium-ion. The reported first-principles calculations using the structure of typical lithium-ion active battery materials provide the large diffusion barriers for various multivalent ions [2]. Our recent study to overcome this slow kinetics for multivalent-ion will be also discussed.

2:00 PM

(ICACC-S6-033-2020) Designing Electrode Architectures for Safer Rechargeable Batteries (Invited)

V. Pol*[1]

1. Purdue University, Chemical Engineering, USA

ViPER (Vilas Pol's Energy Research) laboratory at Purdue University focuses its research activities on the development of high capacity electrode materials, their engineering for longer cycle life and improved battery safety. Considering the advantages and limitations of known synthesis techniques, a solvent-less, single step processing technology has been developed to fabricate a variety of unique anode and cathode materials for Li-ion, Na-ion, K-ion and Li-S batteries. Moreover, the talk will demonstrate how tailored spherical, solid, dense carbon particle anodes could make Li-ion batteries safer via distributing current uniformly on the particle surface during charging, minimizing excess SEI formation and dendritic growth. ViPER's recent efforts on structural, morphological, compositional and electrochemical properties of various fascinating electro-chemistries with transformative technological aspects will be discussed.

2:30 PM

(ICACC-S6-034-2020) Investigation of Metallic Dendrite Growth in Electrolytes

C. Lei*[1]; A. V. Virkar[1]

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

Recently, sodium-based energy storage devices are raising more and more interest because of the sodium abundance. Also, sodium ion batteries operating at room temperature are under development. One of the modes of failure of these devices is the formation of metallic sodium dendrites. In this work, a two-phase material Na-β″-alumina+3YSZ has been used to investigate the sodium dendrite growth. The mixed Na^+ ion and O^{2-} ion electrolyte was fabricated by vapor phase conversion. An embedded hole, which was achieved during the initial uniaxial pressing stage, was formed. Subsequently, a small plastic tube with $NaNO_3$ solution was put on the surface of the disc. Silver wires were put into the solution and the embedded hole. By applying voltage, the in-situ formation of sodium dendrites was observed. The current response and the voltage between the sample were recorded, and EIS was measured. Furthermore, $AgNO_3$ solution was used to investigate the in-situ formation of the sharp dendrite. Several drops of $AgNO_3$ solution were placed onto the surface of the glass slide. Two silver wires were immersed inside of the solution. For the cathode electrode, the silver wire was protected by a thin layer of wax; only the tip was exposed to the solution. By applying the voltage, the silver dendrites shoot out from the tip of the silver wire. Current and voltage response between the sample were recorded. EIS was measured.

Characterization of Materials for Batteries and Capacitors

Room: Tomoka A
Session Chairs: Dany Carlier, ICMCB; Chongmin Wang, Pacific Northwest National Lab

3:20 PM

(ICACC-S6-035-2020) Insights Gained by Microscopy and Spectroscopy Diagnosis of Materials for Better Batteries (Invited)

C. Wang*[1]

1. Pacific Northwest National Lab, USA

Diagnosis based on microscopy and spectroscopy have been realized to be one of the essential approaches for gaining insights as how an electrode material failure, therefore feeding back for designing and creating new materials with enhanced battery performances. In this presentation, we will focus on recent progress on ex-situ, in-situ, operando and cryo S/TEM studies for probing into the structural and chemical evolution of electrode materials for lithium ion batteries and beyond. I will highlight several recent key observations, which even appear to be well documented, while essentially are poorly understood, and therefore act as the bottleneck for the advances of both cathode and anode for better batteries. It would be expected that this presentation can stimulate new ideas as how to attack these bottlenecks to advance the electrode materials design for better batteries.

3:50 PM

(ICACC-S6-036-2020) Charge Distribution Guided by Grain Crystallographic Orientations in Polycrystalline Battery Materials (Invited)

Z. Xu[1]; Z. Jiang[2]; Y. Liu[2]; F. Lin*[1]

1. Virginia Tech, Chemistry, USA
2. SLAC National Accelerator Lab, USA

Architecting grain crystallographic orientation can modulate charge distribution and chemomechanical properties for enhancing the cycle life, safety, and energy density of polycrystalline Li ion and Na ion electrode materials. However, incisively probing the interplay between charge distribution, grain crystallographic orientation, and electrochemical performance remains a daunting challenge. In this work, we elucidate the spatially resolved charge distribution in nickel-rich Li layered oxides with different grain crystallographic arrangements and establish a model to quantify the charge heterogeneity in these polycrystalline battery particles. While the holistic "surface-to-bulk" charge propagation prevails in polycrystalline particles, the crystallographic orientation-guided charge propagation governs the charge distribution in the local charged nanodomains. Compared to the randomly oriented grains, the radially aligned grains exhibit a lower cell polarization and higher capacity retention upon battery cycling. The radially aligned grains create less tortuous Li ion pathways thus improving the charge homogeneity as statistically quantified from over 20 million nanodomains in polycrystalline particles. The present study provides a fundamental guidance for understanding the charge distribution and chemomechanical properties of polycrystalline battery materials.

4:20 PM

(ICACC-S6-037-2020) Tungsten oxide and carbide composite electrodes for electrochemical capacitors synthesized by Hot Filament Vapor Deposition technique

D. Soares*[1]; R. Vicentini[2]; G. Singh[1]; A. Peterlevitz[2]; H. Zanin[2]

1. Kansas State University, Mechanical and Nuclear Engineering Dept., USA
2. Advanced Energy Storage Division, Center for Innovation on New Energies; Carbon Sci-Tech Labs, School of Electrical and Computer Engineering, University of Campinas, Brazil

Since the 1960s tungsten carbide has been extensively studied for catalytic reactions. Furthermore, due to its low cost, good electronic conductivity, corrosion resistance this species is also a candidate to renewable energy storage applications. Specifically, for supercapacitors, another material of interest are transition metal oxides, once then the stored charge has contribution also from its pseudocapacitance. Nevertheless, such family of materials does not usually provide cyclability. Therefore, the purpose of this work is to present a material that has both pseudocapacitance and cyclability. To this end, the supercapacitor electrode synthesized consisted of a tungsten carbide (W2C) and oxide (WxOy) film, synthesized by hot wire chemical vapor deposition (HWCVD) technique in one single, which can be interesting for manufacturing. With respect to electrochemical performance, the material's efficiency increased during cyclability, from 59% at first cycle to 85% after 10,000 cycles. Additionally, the capacitance was enhanced by 160% after 10,000 cycles. Thus, this material presented higher efficiency and capacitive properties after 10,000 cycles, proving it can be a feasible candidate towards energy storage devices in terms of its electrochemical behavior and manufacturing.

S7: 14th International Symposium on Functional Nanomaterials and Thin Films for Sustainable Energy Harvesting, Environmental, and Health Applications

Nanomaterials for Energy Conversion and Storage and Catalysis III

Room: Flagler A
Session Chair: Narsingh Singh, University of Maryland Baltimore County

8:30 AM

(ICACC-S7-024-2020) Origins of extraordinarily high C-rate of nonstoichiometric $TiNb_2O_7$ Li-ion Battery Anode: Enhanced polaron-dominating carrier transport and volume expansion (Invited)

H. Choi*[1]

1. University of Cologne, Germany

Titanium niobium oxide ($TiNb_2O_7$) has recently attracted great attention as anode material for lithium ion batteries (LIBs) due to its high practical capacity of ~280 mAh g^{-1} and high C-rate. In general, defect engineering for wide gap oxide anode, such as $TiNb_2O_7$ is mostly intended to increase electrical conductivity by making oxygen deficiency, which increases electron carrier concentration. In this study, defect equilibria and the effects of defects on the electrical and ionic conductivity were investigated using density functional theory (DFT) calculations. Interestingly, polaron formation energy was negative, which means dominance of polaron-hopping governing carrier transport. In addition, the polaron on Ti_{Nb} anti-site defects increases cell volume of $TiNb_2O_7$ and lowers Li-ion diffusion energy barrier. We experimentally demonstrated with fiber $TiNb_2O_7$ having a specific discharge capacity of ~ 254 mAh g^{-1} at 1 C and a superior rate capability (~ 183 mAh g^{-1} at 100 C) by reduction process.

9:00 AM

(ICACC-S7-025-2020) Black and colored titania films for enhanced photocatalysis under visible light

M. Wong*[1]; M. Pylnev[1]

1. National Dong Hwa University, Materials Science and Engineering, Taiwan

As a multifunctional material, titania has been widely used and investigated for numerous applications, in which photocatalysis of titania for clean energy and environment has drawn the most attention. Lots of efforts were made to modify titania to increase its optical absorbance in the visible region and other aspects to enhance its overall photocatalytic performance. The recently reported

particles of black TiO_2 powder are usually in a core-shell structure of amorphous shell and crystalline core, but little is known about the structure and composition of thin film black TiO_2. Various black H-TiO_{2-x} films in different architectures such as monolithic, bilayered, gradiently- self-doped (oxygen vacancies) and co-doped titania (B,C,N) were prepared in-situ by sputtering in Ar/H_2 plasma or by post-treatment in hydrogen plasma. The structure and properties of the black titania films were analyzed. For example, thin film black H-TiO_{2-x} samples with the enhanced photocatalytic performance were prepared by hydrogen plasma treatment of pure titania film. The black titania film is an ensemble of gradiently hydrogen-and self- doped layer of H-TiO_{2-x} with a limited thickness on a crystalline TiO_2 inner layer. The differences of different black and colored titania films of various sources are compared and the applications and the mechanisms defining the unique properties are discussed.

9:20 AM

(ICACC-S7-026-2020) Atomic Layer Deposition as a Tool to Produce Ceramic Photonic Materials with High-temperature Stability

K. P. Furlan*[1]; T. Krekeler[2]; M. Ritter[2]; R. Blick[3]; R. Zierold[3]; G. A. Schneider[1]; R. Janssen[1]

1. Hamburg University of Technology, Institute of Advanced Ceramics, Germany
2. Hamburg University of Technology, Electron Microscopy Unit, Germany
3. Universitaet Hamburg, Center for Hybrid Nanostructures, Georgia

The fabrication process defines the 3D structure and composition of a photonic material and thereby, its properties. 3D inverse opal photonic crystals (i-PhCs) are usually produced by the infiltration of a polymeric template by either colloidal routes, chemical vapor deposition or atomic layer deposition (ALD). In this talk, different ceramic i-PhCs produced via ALD will be presented and their behavior when exposed to high temperatures depicted. Special focus will be given on the refractory material mullite produced by an ALD super cycle approach, in which a superposition of sub-nm sized laminates of SiO_2 and Al_2O_3 were subsequently generated and looped, aiming to reach the stoichiometric range for the formation of monophasic mullite. Two different silica precursors were used (APTES and 3DMAS), and combined with TMA, water and ozone to generate the films and further infiltrate the PhCs. The produced materials showed conversion to mullite at 1000 °C, comparable to the mullitization temperature of sol-gels type I, attributed to the high compositional mixing homogeneity provided by ALD. The i-PhC fabricated by such ALD super cycle showed a remarkable temperature stability and a photonic stopgap is identified even after exposure at 1400 °C, which might allow its application as a refractory photonic material.

9:40 AM

(ICACC-S7-027-2020) Data-driven Creation of an Inorganic Nanotube Database

I. Castelli*[1]

1. Technical University of Denmark, Department of Energy Conversion and Storage, Denmark

Reducing dimensionality has led to a new realm of materials exhibiting interesting structural, electronic and catalytic properties. While two dimensional materials are the focus of many theoretical and experimental research groups, one dimensional materials are less well studied. Most of the theoretical work on 1D materials is focused on explaining phenomena at the atomic-scale rather than performing a high-throughput search of new inorganic nanotube materials. Here, we investigate by means of density functional theory (DFT) sub-nanometer tubes that exhibit different properties compared to their 2D counterparts while being selective in size and composition. Sub-nanometer tubes have the advantage of tunable properties as well as fewer atoms in the unit cell making them suitable for DFT calculations. I will elucidate designing rules to produce nanotubes with controlled dimensions. Additionally, data-driven design rules guide the search towards nanotubes that can be used in a variety of applications. In detail we focus on properties like bandgaps, stabilities and adsorption energies that lead to predictive models assisting in designing new nanotubes before doing DFT calculations. The generalization from existing data helps to accelerate the search for novel materials for applications in batteries, (photo-)catalysis, chemical storage and nanofluidics.

Nanomaterials for Energy Conversion and Storage and Catalysis IV

Room: Flagler A
Session Chair: Thomas Fischer, University of Cologne

10:20 AM

(ICACC-S7-028-2020) Towards stable and low-PGM fuel cell cathode with Hierarchical Nanostructured Ceramic Thin Film as non-carbon support

G. Rossetti*[1]; F. Di Fonzo[2]; A. Casalegno[1]

1. Politecnico di Milano, Italy
2. Istituto Italiano di Tecnologia, Italy

To date fuel cell technology, especially for automotive applications, is on the brink of large-scale commercialization. Nevertheless, there is still concern about the expensive materials and the long-term stability of the devices. The aim of this contribution is to show our work advances in the research of a high surface area, durable ceramic support. Starting from the physical vapour deposition process developed, a nanostructured titanium nitride film has been shown to be a promising alternative to the traditional carbon support for platinum. However, the low electrical conductivity of these materials is a bottleneck to their applicationin this field. Another interesting aspect of this material is its hierarchical structure that can assure a high surface area to the catalyst. By an optimized atomic layer deposition process, low platinum loading has been reached (around 0,1 mg/cm^2) with a better utilization of the precious metal. Furthermore, recent improvements have been done to increase the electrical conductivity of the titanium nitride support and the optimum compromise between electrical conductivity, surface area and morphology has been found. This, combined with the superior durability of the titanium nitride (15% decrease in ECSA after 15000 cycles between 1-1,5 V), could lead to a durable and cheaper alternative to the current state of the art.

10:40 AM

(ICACC-S7-029-2020) Nanostructured Ceramics for Hybrid Organic Photoelectrochemical Water Splitting: The Role of Charge Selective Layers

A. Alfano*[1]; A. Mezzetti[1]; F. Di Fonzo[1]

1. Italian Institute of Technology, Center for NanoScience and Technology, Italy

Hybrid organic photoelectrochemical water splitting is gaining momentum in the field of solar conversion technologies. Hybrid organic photoelectrodes are based on reasoned coupling of multiple layers, where the final goal is to minimize both photogenerated charges recombination rate and reaction overpotentials. To this extent, charge selective layers play a relevant role in overall system efficiency. Materials employed for this application need to satisfy stringent requirements in terms of energetic alignment, electrochemical stability and optical transparency. In this work, transition metal dichalcogenides and metal oxides for charge selective layers applications are being investigated and optimized to realize high performing hybrid organic photocathodes. By tuning the deposition conditions, we are going to elucidate how nanostructuration of these layers greatly contributes to the overall performances of the full

device. In depth characterization will be also discussed, with main focus on the charge transport properties of these nanostructured thin films within a photoelectrochemical cell. The best performing materials will be then employed to realize a complete hybrid photocathode architecture, capable of achieving photocurrents exceeding 5 mA/cm^2 and a remarkable stability over several hours of continuous operation.

11:00 AM

(ICACC-S7-030-2020) Characterization of Piezoelectric MEMS Vibration Energy Harvesters using Lead-free $BiFeO_3$ Film under Random Vibration

S. Murakami*[1]; T. Yoshimura[2]; M. Aramaki[2]; K. Satoh[1]; Y. Kanaoka[1]; K. Tsuda[3]; N. Fujimura[2]

1. Osaka Research Institute of Industrial Science and Technology, Research Division of Electronic and Mechanical Systems, Japan
2. Osaka Prefecture University, Graduate School of Engineering, Japan
3. Osaka Research Institute of Industrial Science and Technology, Research Division of Product Reliability, Japan

Recently, considerable attention has been directed toward vibration energy harvesters aimed at realizing energy sources for micro electronic systems. We have focused on MEMS piezoelectric vibration energy harvesters (PVEH) owing to the direct conversion of vibration energy into electrical energy with a high-power density. Although $Pb(Zr,Ti)O_3$ (PZT) is widely accepted as the piezoelectric material, we have already demonstrated that MEMS-PVEHs employing the lead-free $BiFeO_3$ (BFO) films show the high-power density which is comparable to the best performing PVEHs using the PZT films. In this study, we fabricated MEMS cantilever-type PVEHs employing (100)-oriented BFO films. The output power was investigated at various accelerations under random vibration whose power spectral density (PSD) was constant in the frequency regime between 135 and 175 Hz. The PSD of the output power shows that cantilever vibrates with a high mechanical quality factor at the resonant frequency of 153.8 Hz. The power generation under the random vibration is proportional to the square of the acceleration, and agrees well to the power generation estimated from the measurement result under sinusoidal vibration. The comparison of the power generation under random and sinusoidal vibrations is also discussed. Acknowledgments: This work was supported by JST CREST (JPMJCR16Q4).

S8: 14th International Symposium on Advanced Processing and Manufacturing Technologies for Structural and Multifunctional Materials and Systems (APMT14)

Advanced Manufacturing and Processing II

Room: Coquina Salon A

Session Chairs: Shu Yin, IMRAM, Tohoku University; Pavol Sajgalik, Institute of Inorganic Chemistry, Slovak Academy of Sciences

8:30 AM

(ICACC-S8-026-2020) Harnessing spontaneous polarisation to enhance chemical processes (Invited)

S. Dunn*[1]

1. LSBU, Engineering, United Kingdom

Ferroelectric materials have an inherrent dipole that can be harnessed to drive a number of unique features. Over the past ten years there has been increased understanding of what unique chemistry the dipole can drive. Aspects of photochemistry, such as those associated with enhanced exciton lifetimes, energy harvesting through vibration and photovoltaic devices are all applications that are increasingly studied. Most recently the use of thermal fluctuations that have been investigated for disinfection and the production of hydrogen from water. Water splitting by thermal cycling of a pyroelectric element acting as a charge source is an attractive method to produce hydrogen using transient low-grade waste heat. This presentation will take a meandering review of the growing body of work that focuses on ferroelectric materials in energy conversion technologies. Current work is focused on alternative reactions and increasing the surface area of the electrode to enhance the rate of conversion by releasing more charge.

9:00 AM

(ICACC-S8-027-2020) Hydroxylated High-entropy Alloy as Highly Efficient Catalyst for Electrochemical Oxygen Evolution Reaction

P. Ma*[1]; S. Zhang[1]; M. Zhang[1]; J. Gu[2]; L. Zhang[1]; W. Ji[2]; Z. Fu[2]

1. Wuhan University of Technology, China
2. Wuhan University of Technology, State Key Lab of Advanced Technology for Materials Synthesis and Processing, China

CoCrFeNiAl high-entropy alloy (HEA) with uniform element distribution, abundant nanophases, high electronic conductivity and high corrosion resistance in alkaline electrolyte is not only regarded as a potential OER catalyst, but also as a favorable support for the electrocatalyst. In this work, CoCrFeNiAl HEA supported Co,Fe,Ni-(O)OH electrocatalyst was fabricated through direct hydroxylation of CoCrFeNiAl HEA, followed by in situ electrochemical activation, which delivers OER current intensity of 10 mA cm^{-2} at overpotential of 240 mV and a Tafel slope of 52.7 mV dec^{-1}.

9:20 AM

(ICACC-S8-028-2020) Growth of YAG films on YAG single-crystal substrate via a sol-gel method

I. Milisavljevc*[1]; Y. Wu[1]

1. Alfred University, New York State College of Ceramics, USA

Fabrication of thin films on various substrates via the sol-gel method has been recognized in recent years as more convenient and less expensive method, which allows better control over compositions and film uniformity. However, there is less information in the literatures on the mechanism of the interface formation and, especially, the epitaxial relationship between the film and the substrate during the sol-gel film deposition. In this study, $Y_3Al_5O_{12}$ (YAG) sol-gel films were deposited using the spin-coating technique on optically polished and cleaned YAG crystal substrates. After deposition, the films were dried and annealed in the air environment. Crystal structure and morphology of the grown films were investigated using XRD, SEM, and AFM, while the optical properties were evaluated using UV-Vis spectroscopy. The structural relationship between the deposited YAG film and the substrate was studied using the transmission electron microscopy.

9:40 AM

(ICACC-S8-029-2020) Textured Monoclinic Lanthanum Titanate Prepared by Magnetic Alignment

O. Van der Biest*[1]; L. Zhang[1]; Z. Sun[1]; J. Vleugels[1]

1. K U Leuven, Materials Engineering, Belgium

Monoclinic lanthanum titanate (La2Ti2O7) ceramic was reported to exhibit strong piezoelectric and electro-optic effects with a very high Curie temperature (1500 °C). It has a high relative dielectric constant (42-46) with a low-temperature coefficient and a low dielectric loss at microwave frequency. La2Ti2O7 is a promising candidate for high-temperature piezoelectric and electro-optic devices, low temperature coefficient capacitance materials and microwave frequency dielectrics. Earlier work showed that

crystallographic texturing of La2Ti2O7 may be a valuable way to improve its piezoelectric properties. In the present work La2Ti2O7 powder was synthesized by reacting lanthanum nitrate with TiO2 powder. Crystallographic alignment was obtained by electrophoretic deposition of the powder in a 9.4 Tesla strong magnetic field. The magnetic alignment mechanism was analyzed according to monoclinic crystal symmetry. The (-4, 0, 1) plane of La2Ti2O7 was aligned perpendicularly to the imposed magnetic field direction. The three principal magnetic axes were determined relative to the crystallographic axes: one of the magnetic axes has an angle of 74.94° to the a axis, the other has an angle of 66.30° relative to the c axis and the third magnetic axis is along the b axis. Moreover, a uniform and morphologically isotropic microstructure is observed in the sintered La2Ti2O7 with a substantial crystallographic alignment.

10:20 AM

(ICACC-S8-030-2020) Design of Alumina/Titanium Composites and their Multifunctions with Room-temperature Crack Healing (Invited)

S. Shi[1]; S. Chou[1]; T. Goto[1]; T. Sekino*[1]

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

Ceramic-based composites consisted of alumina (Al_2O_3) matrix and fine titanium (Ti) dispersion were designed and fabricated by controlled powder-metallurgical and sintering methods. The composites exhibited enhanced fracture toughness and good electrical conductivty due to the dispersion of metallic Ti phase, which was applicable to electro-discharge machining. When the composites were heat-treated or chemically-treated using alkaline soluton, nanostructured titania such as nanorods, nanosheet and nanoporous layeres were formed on the surface of Ti in Al_2O_3. These surface nanostructured titania exhibited photocatalytic properties. In addition, the composite exhibited crack-healing function when cracked composites were electrochemically-anodized in aqueous solution at room-temperature without heating, which was the first achievement of the room-temperature crack-healing in ceramic-based composites. These results imply that the fine-Ti dispersed Al_2O_3 is multifunctional ceramic-based composites having balanced mechanical, electrical and advanced properties including room-temperature repairable function and photocatalytic performance. This it is considered that the composites might be utilized for "multi-task" applications. Detailed relationships between processes, micro/nano/interface structure, mechanical, electrical and crack healing characteristics will be discussed.

10:50 AM

(ICACC-S8-031-2020) Laminated Object Manufacturing of Ceramic-based Composites (Invited)

N. Travitzky*[1]

1. University of Erlangen-Nuremberg, Materials Science, Germany

It is an incontrovertible fact that the wide industrial use of advanced ceramic materials depends on the technological availability to fabricate near-net-shaped three-dimensional ceramic-based parts with required geometry. Owing to the inability of current technology-related methods to produce porous ceramic structures with complicated internal structure and desired microstructures and properties, additive manufacturing (AM) techniques such as 3D-Printing (3DP), Selective Laser Sintering, Melting or curing (SLS, SLM and SLC), Extrusion Free Forming (EFF), Stereolithography and Laminated Object Manufacturing (LOM) are becoming increasingly important. In this talk, the emphasis is placed on the fabrication of dense and porous, oxide as well as non-oxide ceramic-based composites with relatively complex geometries by LOM process, the associated problems addressed, and the possible routes to circumvent these problems highlighted. Microstructures and mechanical properties of the fabricated composites will be present.

11:20 AM

(ICACC-S8-032-2020) Controlling Grain Growth of Alumina by Solute-Drag and Solute-Acceleration

R. Moshe[1]; P. Ghosh[1]; L. Rudnik[1]; R. Marder[1]; W. D. Kaplan*[1]

1. Technion - Israel Institute of Technology, Dept. of Materials Science and Engineering, Israel

Microstructural evolution of alumina is strongly affected by dopants, such as MgO which promotes sintering and limits grain growth. Impurities, such as CaO and SiO_2, are known to cause exaggerated grain growth. Unlike segregating dopants which reduce grain boundary mobility by solute-drag (such as MgO and C), CaO increases the rate of grain growth, and the increased mobility due to Ca segregation is believed to be due to an increase in vacancy concentration in the immediate vicinity of the grain boundaries. Co-doping with Mg and Ca leads to a higher Mg solubility limit, and thus more Mg at the grain boundaries in balance with Mg in solution, and thus a reduced grain boundary mobility. Presumably grain boundary motion in alumina is via the motion of disconnections, which has been experimentally demonstrated for $SrTiO_3$. How dopants, including carbon, interact with disconnections will be discussed.

11:40 AM

(ICACC-S8-033-2020) Bioprocess-Inspired Microscale Additive Manufacturing of Multilayered TiO_2/Polymer Composites with Enamel-like Structures and Excellent Mechanical Properties

L. Lei*[1]

1. Wuhan University of Technology, China

Enamel is an excellent organic-inorganic composite biological material. This microstructure exhibits outstanding mechanical performances and durability. Herein, a new research direction "bioprocess-inspired fabrication" is proposed for achieving a multilayered organic-inorganic columnar structure. Inspired by the formation process of enamel, a microscale additive manufacturing method is used to mimic this process, and rutile titanium dioxide nanorods, a polymer and graphene oxide (GO) are sequentially assembled in a layer-by-layer fashion to form a structure of orderly inorganic minerals wrapped in an organic framework. In particular, GO can both serve as a substrate for TiO_2 nanorods and interact with polymers, jointly leading to the strength of the composite materials. Impressively, this enamel-like structure material using inexpensive and abundant materials has hardness and ultrahigh Young's modulus comparable to natural enamel, and viscoelastic propertysuperior to most solid materials, with striking mechanical performances and vibration resistance. Consequently, our biomimetic synthetic approach can provide an in-depth understanding of the formation process of biomaterials, but also enables the exploration of a new avenue for the preparation of organic-inorganic composite materials.

Functional Materials and Composites

Room: Coquina Salon A
Session Chair: Tohru Sekino, Osaka University

1:30 PM

(ICACC-S8-034-2020) Morphology Control and Gas Sensing Property of Nitride / Oxynitride Based Materials (Invited)

S. Yin*[1]

1. IMRAM, Tohoku University, Japan

In the present talk, the synthesis and characterization of morphology controllable nitrides and oxyoxides will be introduced. In a typical synthesis process, the precursors with various morphologies synthesized by hydrothermal process were utilized for the nitridation treatment. As an example, GaN was prepared from α–GaOOH

precursors by a direct nitridation method under NH_3 flow. The nitridation was carried out at various temperatures to obtain GaN with different oxygen contents, which played important role to H_2 gas sensing response. Although the oxide based materials showed very limited gas sensing property, the nitride and oxynitride based materials showed quite enhanced sensing sensitivity. The gas sensing sensitivity of the GaN with 2.07 wt.% of oxygen content was 10 times higher than that with the lowest oxygen content of 1.9 wt.%. The sensors also showed high stability and repeatable property after being exposed in H_2 gas at 500°C. Furthermore, the morphology controllable aluminum nitride such as needle-like, nest-like and plate-like AlN were also successfully synthesized by the similar manner.

2:00 PM

(ICACC-S8-035-2020) Generation of highly-active battery-materials with unique spray pyrolysis equipment

T. Jähnert[1]; K. Weber*[1]

1. Glatt Ingenieurtechnik GmbH, Germany

The aim of this project is to generate powder materials, which can be used in the next generation of lithium-batteries. Cathode-active and solid electrolyte materials were generated and tested for their functionalities. Glatt developed a special technology for generating highly functionalized powder particles. This APPtec uses a pulsating stream of hot gas to generate or modify powders. Inexpensive raw materials are dissolved and then sprayed into the pulsating gas stream. There the material is dried, calcinated and reacted to form functional oxides in nano- to micrometer-size. The advantage of this process arises through the unique thermodynamic conditions in the reactor, which can lead to higher heat transfer to the particles and the formation of unique structures and very active materials. With this technology lithium-manganese-nickel oxide (LMNO) was made and tested as cathode-material. Processing parameters were optimized and stoichiometry and doping of the material adjusted to maximize the capacity and stability of the LMNO. In similar experiments lithium-lanthanum-zirconium oxide (LLZO) was synthesized, optimized and tested as a solid electrolyte. The presentation will show the development of LMNO with capacities over 130 mAh/g, high stability and the possibility for easy upscaling and the generation of LLZO and testing of ceramic films of this material.

2:20 PM

(ICACC-S8-036-2020) Influence of Crystal Orientation on Dielectric Properties of [001] Textured $BaTiO_3$ Fabricated by Template Grain Growth

S. Lee*[1]; J. Wang[1]; D. Kim[1]

1. Korea Advanced Institute of Science and Engineering (KAIST), Dept. of Mater Sci & Eng, Republic of Korea

Highly textured polycrystalline bulk $BaTiO_3$ was fabricated using conventional template grain growth (TGG) method. Preferentially oriented grains of bulk ceramic showed anisotropic dielectric properties along with its various directional bias. Hydrothermally synthesized one-dimensional $BaTiO_3$ particles performed as a seed material. [001] directionally grown single crystal sub-micron sized templates were uniformly aligned within the 50nm $BaTiO_3$ equiaxed matrix particles. Although as-sintered microstructure of textured ceramic displayed spherical grain morphology, X-ray diffraction patterns confirmed the maintenance of preferential [001] oriented crystallography. Dielectric constant comparison between parallel and vertically sliced samples with respect to casted direction was conducted to investigate the influence of crystal orientation under applied electric field.

2:40 PM

(ICACC-S8-037-2020) Stable Pd@Cu Core–Shell Nanocubes with Finely-Tuned Sizes for the Reduction of Nitroaromatics

G. Zhang*[1]; J. Feng[3]; Z. Fu[2]

1. Wuhan University of Technology, School of Chemistry, Chemical Engineering and Life Science, China
2. Wuhan University of Technology, State Key Lab of Advanced Technology for Materials Synthesis and Processing, China
3. University of California, Department of Chemistry, USA

This paper reports a seed-mediated growth of Pd@Cu core-shell nanocubes with controllable sizes from 49 to 103 nm in an aqueous system using cetyltrimethylammonium bromide (CTAB) capped Pd nanocubes with sizes of 21 nm as seeds. During the growth, ascorbic acid (AA) was served as the reductant and hexadecylamine (HDA) as the capping agent and ligand to form coordination complex with Cu^{2+}. The sizes of Pd@Cu nanocubes could be finely tuned by changing the volume of Pd seeds while maintaining the amount of $CuCl_2$ precursor. Cu atoms initially nucleated on one or two faces of a Pd seed via localized epitaxial growth. Due to the lattice mismatch between Pd and Cu (7.1%), Pd cores were not located in the center of Cu shells during the initial deposition of Cu. Interestingly, most of Pd cores could transfer to the center of Cu nanocubes by dissolving and re-growing of the boundary to decrease the interfacial strain when sufficient amount of $CuCl_2$ precursor and reaction time were provided. The Pd@Cu nanocubes exhibited strong LSPR signals and long-term stability which could be low-cost potential substitute for Au and Ag for applications in sensing, surface-enhanced spectroscopy, optoelectronics and photocatalysis. Comparing the rate constant of p-nitrophenol reduction reaction over Pd@Cu nanocubes with different sizes as catalyst, 83 nm Pd@Cu nanocubes showed the highest catalytic activity.

3:00 PM

(ICACC-S8-038-2020) Micro-cantilever testing of Si_3N_4 based ceramics with different sintering additives (Invited)

P. Sajgalik*[1]

1. Institute of Inorganic Chemistry, Slovak Academy of Sciences, Ceramic Department, 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 based ceramics. The role of intergranular grain boundary films (IGBF) is a core of the present paper. Role of IGBFs is documented on the bulk ceramics. The fracture toughness and bending strengths are evaluated. Despite the literature data, the fracture toughness and strength of Lu_2O_3 doped ceramics is the highest compared to the La_2O_3 doped Si_3N_4 ceramics. This behavior is tried to be explained by the ab initio model simulations including an individual chemical composition of IGBF films. These model ab initio experiments are confronted with the model experiments that use cantilevers of several micrometers in size, which were used for determination of IGFs strength. The cantilevers were cut from the real microstructure using a FIB technique. The geometry and location of cantilever was carefuly selected in the microstructure in order to have a right position for measuring the strength of IGBF. The discussion of this paper is trying to give the sound explanations of the role of the IGBFs in silicon nitride based ceramics, which has been conducted for several decades in the ceramics community.

3:30 PM

(ICACC-S8-039-2020) B and B-W nanoparticle preparation by pulsed discharge of compacted powder

H. D. Nguyen*[1]; M. Ngo[1]; Y. Tokoi[3]; T. Do[2]; T. Nakayama[1]; H. Suematsu[1]; K. Niihara[1]

1. Nagaoka University of Technology, Japan
2. Nagaoka University of Technology, Nuclear System Safety Engineering, Japan
3. National Institute of Technology, Oyama College, Department of Innovative Electrical and Electronics Engineering, Japan

In this study, B and compounds of B-W nanoparticles were prepared by discharging a pulsed current through B micron powder as a raw material in Ar gas. By charging three banks of capacitors (capacity of each one is 10μF) to 6.2 kV and discharging through the B micron powder, due to Ohmic heating, B micron powder was heated, melted and vaporized. Then, B vapor was quenched rapidly and became B nanoparticles. In the experiment, copper and tungsten rods were used to compress B micron powder. Prepared powder was B spherical nanoparticles with little amount of Cu impurity and compound of spherical B-W nanoparticles by using Cu electrodes and W electrodes, respectively. By TEM bright field images, size of nanoparticles was confirmed to be less than 100 nm for both cases of Cu electrodes and W electrodes. B nanoparticles are expected to be used in semiconductors while the success of preparing B-W nanoparticles is promising for preparation of tungsten boride nanoparticles for application of superhard materials.

3:50 PM

(ICACC-S8-040-2020) Fabrication of Porous and Magnetic Ceramic Microparticles via Stop Flow Lithography

A. Alcaraz-Ramirez*[1]

1. Purdue University, Materials Engineering, USA

Fabrication of microparticles with active properties as self propulsion and mobility with enhanced mechanical, thermal and chemical properties are of special interest for emerging technologies such as micro-robotics and drug delivery. Particles having porosity and magnetic behavior can have a tunable trajectory along a rectangular path. The porous characteristic of a particle assists to increase its surface area allowing the particle to be infiltrated with a catalyzer. On the other hand, the magnetic behavior of the particles allows a complete controllable trajectory with defined magnetic fields. Non-intrusive medical interventions with microparticles are an alternative to chemotherapies by reducing the side effects of the treatment. The method used to fabricate the particles was Stop-flow lithography, which is a method that combines microfluidics and photolithography properties for the continuous fabrication of microparticles with predefined shapes. We were able to fabricate particles porous and magnetic ceramic particles in the 10-50 μm range with tunable thicknesses of 30-120 μm with predefined shapes as hexagons, discs, rods and triangles. The material that we used was a UV sensitive preceramic polymer which is compatible for functional modifications. This polymer allows it usage as either polymer or ceramic material upon conversion.

S9: Porous Ceramics: Novel Developments and Applications

Innovations in Processing Methods and Synthesis of Porous Ceramics I

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

1:30 PM

(ICACC-S9-001-2020) Porous polymer derived ceramics (Invited)

C. Vakifahmetoglu*[1]

1. Izmir Institute of Technology, Materials Science and Engineering, Turkey

Porous ceramics are key enabling components in different demanding applications because of their favorable set of properties. The production of porous ceramics from preceramic polymers offers advantages in terms of simple processing methodology, low processing cost, and ease of control over porosity and other properties arising from the composition of the resultant ceramics. A vast amount of research has been conducted towards the fabrication and characterization of porous ceramics produced from different polymeric precursors. The potential of polymer-derived-ceramics can only be fully achieved if the type of used fabrication method is tailored taking into account the specific application of interest. Here, different processing strategies employed to obtain, from polymer precursors, ceramic foams, membranes, aerogels or porous ceramics for which porosity higher than 50 vol% has been deliberately introduced, including also components with high specific surface area or hierarchical porosity will be mentioned. The different processing strategies, such as replica, sacrificial template, direct foaming, etching, aerogel forming, fiber spinning and automated manufacturing, are explained and the published literature has been reviewed with particular concern for the critical issues that affect the pore size, the amount of porosity and strength of the ceramic components

2:00 PM

(ICACC-S9-002-2020) Porous SiOC bulk ceramic based on perhydropolysilazane (PHPS) and polysiloxane (PSO) pyrolysis

N. Yang[1]; K. Lu*[1]

1. Virginia Tech, USA

A new route for the synthesis of micro- and meso-porous silicon oxycarbide (SiOC) is studied based on the pyrolysis of perhydropolysilazane (PHPS) and polysiloxane (PSO) as preceramic materials. The similar hydrophobicity of the two species as well as the high reactivity of the monosilanyl and silanyl groups of PHPS with the hydroxy end groups of PSO results in homogeneous mixing of the two polymer precursors. Polysiloxane forms the matrix of the bulk ceramic. PHPS, with the repeating unit of (SiH_2-NH), can convert into silica glass throughout the SiOC matrix. When silica is etched away as a pore-forming agent through HF acid treatment, high surface area SiOC bulk ceramic forms. Based on the model of nanodomains in the polymer-derived SiOC, the effect of PHPS content on the thickness of the interdomain boundary (δw) is investigated. The thickness of δw will be related to the etchability of nanoscale silica domains.

2:20 PM

(ICACC-S9-003-2020) Automated 3D-Assembly of Modular Ceramic Composite Structures

J. M. Biggemann*[1]; M. Stumpf[1]; P. Greil[1]; T. Fey[1]

1. Friedrich-Alexander University Erlangen-Nürnberg, Department Material Science and Engineering, Germany

Current ceramic manufacturing aims for near net-shaped components with high geometric complexity, accuracy, individual shape and size. Pick and place robots enhance the precision and lower the

discard in large-scale industrial manufacturing. Pick and placers equipped with an automatic optical inspection (AOI) system enable simultaneous dimensional characterization, tolerance sorting and flexible placing of miniaturized ceramic components (=building blocks). In this work, we present a unique processing technique for modular ceramic composites utilizing a fully automated assembling technology. Ceramic building blocks of various shapes (e.g. cubic, triangular, osteomorphic) and sizes (0.3 – 10 mm) were fabricated via transfer molding. Single building blocks are picked up by an assembly head from a tray feeder and assembled to complex 3D-structures. Position, size and rotation angle are recognized by the AOI system. The building blocks are connected with an UV-curing adhesive by an integrated dispensing unit. A placing precision of < 78 µm was derived by µCT reconstructed 3D-analysis. Building blocks were assembled to high toughness brick-and-mortar structures with a K_{Ic} up to 14.5 MPa $m^{0.5}$. Dense and porous modular structures can deliberately fabricated utilizing the building block approach by varying the dimensions, shape and placing gaps of the building blocks.

2:40 PM

(ICACC-S9-004-2020) Fabrication and Characterization of Mechanical Properties for Hydroxyapatite – Poly(lactic acid) Unidirectional Porous Scaffolds

Y. Zusho*[1]; S. Kobayashi[1]; T. Osada[1]

1. Tokyo Metropolitan University, Mechanical Systems Engineering, Japan

Although autologous bone is currently used for repairing bone defects, this method is highly invasive and poses a heavy burden on patients. Thus, the use of artificial bones has been expected. Among the artificial bone materials, hydroxyapatite (HA) have been attracting much attention. In this research, in order to overcome the problem that artificial bone has low initial strength after implantation, poly(lactic acid) (PLA) was coated on HA unidirectional porous body prepared by freeze-casting method. HA, which is non-bioabsorbable, is expected to remain in the body. On the other hand, bioabsorbable PLA is expected to reinforce at an early stage of implantation and gradually replaced to bio-bone. For HA porous body, the porosity was constant in the plane perpendicular to the freezing direction regardless of the distance from the bottom in the freezing step. The pore size increased in proportion to the distance from the bottom, and became almost constant after a certain distance. In order to investigate the PLA coating method that most effectively improves the mechanical properties, the coating state of PLA was observed and the compression test was performed. As a result, the behavior of the stress-strain diagram of the HA-PLA composite porous body was different from that of the HA porous body, and the mechanical properties were improved.

Engineering Applications of Porous Ceramics I

Room: Coquina Salon F
Session Chair: Tobias Fey, Friedrich-Alexander University Erlangen-Nürnberg

3:20 PM

(ICACC-S9-005-2020) Hierarchical Pore Structures via Freeze Casting With Preceramic Polymers and Block Copolymers

C. T. Kuo*[1]; L. M. Rueschhoff[2]; M. B. Dickerson[2]; K. Faber[3]

1. California Institute of Technology, Material Science, USA
2. Air Force Research Laboratory, Materials and Manufacturing Directorate, USA
3. California Institute of Technology, USA

Porous ceramics are largely used for filters and catalyst supports due to their chemical and temperature stability. Three characteristics are particularly crucial for these applications: surface area, permeability, mechanical robustness. High surface area allows more active sites for chemical reactions or capture. High permeability promotes fluid transport to reaction/capture sites. Mechanical robustness ensures prolonged product life. However, optimizing structures for surface area, permeability, mechanical robustness remains a challenge as they are often inversely related to one another through porosity and pore size. To address this challenge, we propose a porous composite with two structural elements: The first is a SiOC template with dense pore walls produced by freeze casting of preceramic polymers. Micrometer pores provide rapid fluid transport and low back pressure. By re-infiltrating with a fugitive phase of self-assembled block copolymers, an interior surface coating of SiOC adds porosity of a second length scale. The derived nanometer pores provide high surface area. Evolution of the hierarchical structure was investigated with microscopy techniques. The surface area, porosity, mechanical and flow properties were examined to provide an understanding of how complex structures influence material response and to assess the composite's potential as a filtration material.

3:40 PM

(ICACC-S9-006-2020) Ag or Cu-modified geopolymer foams, 3D-printed scaffolds, and granules for water treatment

T. Luukkonen*[1]; J. Yliniemi[1]; G. Franchin[2]; P. Colombo[2]

1. University of Oulu, Fibre and Particle Engineering Unit, Finland
2. University of Padova, Industrial Engineering, Italy

Point-of-use (POU) water disinfection with Ag-modified ceramic filters is a promising approach for improving access to safe drinking water. The POU technology can be more sustainable than centralized water treatment in developing countries. However, the firing of ceramics results in emission of environmentally detrimental gases and particulate matter, especially if low quality fuels are used. Geopolymers are ceramic-like amorphous aluminosilicates that do not require high-temperature sintering for consolidation. In the present work, different methods for fabricating highly porous metal-containing geopolymer filters were employed, and their characteristics were compared. Metakaolin-based geopolymers were prepared using direct foaming, granulation, or 3D printing. Metals (Ag or Cu) were added directly to paste (as salt or nanoparticles), via ion exchange, or by dipping the filter into colloidal Ag solution. Filters were characterized in terms of mechanical strength, porosity, permeability, and stability of added metals. Results indicate that the properties of filters can be readily controlled by the selection and tuning of the preparation method and they are in many cases comparable to those of conventional ceramics. The porous components in this study were prepared at 25–60°C, and thus they represent a potentially more sustainable and cost-effective approach for water treatment filters.

4:00 PM

(ICACC-S9-007-2020) Porous ceramics - from processing to novel applications

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1. Friedrich-Alexander University Erlangen-Nürnberg, Department Material Science and Engineering, Germany
2. Institute of Glass and Ceramics, Material Science and Engineering, Germany

Cellular materials offer a wide spectrum of applications such as catalyst support structures, lightweight materials, energy adsorption or energy storage materials. Due to several ways of processing and different materials, a wide range of material properties e.g. thermal conductivity, mechanical strength or damping can be adjusted, measured and verified, with regard to the expected properties. Various techniques for processing porous ceramics and their corresponding composites independent of material are presented. Especially in heterogeneous and homogeneous porous structures and their composites, only global effective material properties can be determined and measured. For example, the knowledge on the

predominating influence of the microstructure on the global properties is the key for designing materials with desired properties. To fill this gap and enable a "look-in" a microstructure model derived from μ-CT measurements (see highlights in Figure 1) carried out at certain processing steps can be used as model for FEM-calculations. Combining estimated material properties by experiment with microstructure models offers the possibility to carry out different simulations over different hierarchical levels in order to design the structures for future applications of porous ceramics.

4:20 PM

(ICACC-S9-008-2020) Influence of the texture on the conductive-radiative behavior of SiC-based cellular ceramics up to very high-temperature

B. Rousseau*[1]; B. Afeef[1]; V. Jerome[2]; F. Yann[1]

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Refractory cellular materials constitute an attractive class of materials for designing high-temperature energy conversion systems and processes. Their thermal behavior at high temperatures is poorly known today, whereas such information is crucial for the global optimization of the involved industrial systems and processes. The texture possessed by such materials hampers the establishment of robust predictive laws. To tackle this issue, one proposes a numerical route which combines the generation of cellular ceramics and a massively parallel finite element conduction-radiation solver. Three SiC-based cellular structures where the textural features are tuned will be confronted for same porosity and mean cell size: cubic-cell, Kelvin-cell, and random open-cell structures. To neglect convection and study the conduction-radiation characteristics of the samples, numerical experiments are performed within fictitious vacuum medium while being exposed to severe gradients (1200-1800 K). The conductive-radiative coupling will be discussed according to the Stark number variations. This number allows us to differentiate heat transport dominance either by conduction or by radiation. A practical guideline will be proposed to tune the thermal conductivity at high-temperatures according to the textural features.

4:40 PM

(ICACC-S9-009-2020) Development of radiant porous burners working at 1500°C: Composition and microstructure of Si-SiC based reticulated structures

G. Bianchi*[1]

1. SUPSI, DTI-MEMTI, Switzerland

Different approches can be followed to increase the efficiency of a radiant porous burners. To name a few, one is the optimization of the thermo-fluid dyinamic properties by engeneering the pore morphology exploiting additive manufacturing and another is enabling higher working temperature of the ceramic radiant body by enhancing the thermo-physical and thermo-mechanical properties. For state of the art Si-SiC emitting body in radiat porous burners the lifespan decreases as the the working temperature approches the Si melting point of 1412°C, which is considered their short time maximum working temperature. In this work is addressed the development of Si-SiC based materials suitable for combustion environments reaching 1500°C. Several strategies can be applied to move the long term working temperature above the Si melting point. The effects of oxidation at 1500°C in air were evaluated for different variations in the matrix composition and in the microstructure. In the short term scale the modified materials showed a promising increase in the oxidation resistence.

S10: Modeling, Genome, Informatics, and Machine Learning

Informatics, Genome and Machine Learning

Room: Coquina Salon G

Session Chairs: Jingyang Wang, Shenyang National Laboratory for Materials Science, Institute of Metal Research, Chinese Academy of Sciences

1:30 PM

(ICACC-S10-001-2020) Machine Learning Approach to Predict Phase Stability of Complex Oxide Systems (Invited)

D. Shin*[1]; J. Lee[2]; I. Jung[2]

1. Oak Ridge National Laboratory, USA
2. Seoul National University, Republic of Korea

The current paradigm of oxide thermochemistry research heavily relies on either experimental measurements or density functional theory-based first-principles simulations. Herein, we present an emerging machine learning approach that can leverage existing rich experimental data accumulated over many decades. We show that cation polyhedra which efficiently capture both chemistry and topology of complex oxides can be used as descriptors to represent their thermochemistry. Predicted formation enthalpies of ternary/quaternary oxides in Li-Fe-P-O, from the well-trained surrogate machine learning models, have been used to predict isothermal section of Li_2O-Fe_2O3-P_2O_5, as an example. The demonstrated approach can be extended to other higher-order oxide systems to rapidly predict their phase stability, where crystal structures of constituent oxides are available.

1:55 PM

(ICACC-S10-002-2020) Machine Learning for Novel and Improved Ceramic and Single-Crystal Scintillators (Invited)

G. Pilania*[1]; A. Talapatra[1]; C. Stanek[1]; B. P. Uberuaga[1]

1. Los Alamos National Laboratory, Materials Science and Technology Division, USA

With the advent of data-centric approaches, we are entering a new paradigm in which materials informatics and data-mining can augment chemical intuition in the search for new materials. We are applying such approaches to the search for novel scintillators. In many areas in which radiation detection or imaging is required, a key component of the detector system is the scintillating material. However, almost universally, the scintillator is chosen off-the-shelf, without any special consideration for the system at hand. We propose that, by combining experimental synthesis and characterization with first-principles calculations and machine learning (ML), we can optimize materials for the given application, enhancing properties that are critical at the expense of others that may be less important. This talk will specifically focus on the ML-enabled screening approach of this project in which we build physics-informed statistical learning models to predict the fundamental properties of potential scintillating materials. This provides invaluable information for down-selecting materials for further experimental interrogation. ML predictions combined with on-demand electronic structure computations and targeted experiments lead to an integrated approach capable of providing both new insights into what drives performance in these scintillators and optimizing them for a specific application.

2:20 PM

(ICACC-S10-003-2020) Machine learning approach to predict thermal expansion of oxides

S. A. Utlak*[1]; D. Shin[1]; C. Bridges[2]; S. Lee[3]

1. Oak Ridge National Lab, Materials Science and Technology Division, USA
2. Oak Ridge National Laboratory, Chemical Sciences Division, USA
3. Oak Ridge National Lab, Computer Science and Mathematics Division, USA

A physics-based model with the ability to predict thermal expansion does not currently exist. Leveraging the machine learning (ML) methodology to determine such a property would be a powerful asset in the workflow of evaluating material behaviors. Hence, a ML approach was used to compute the thermal expansion of oxides. Experimental measurements were compiled for input to train the model, and crystalline lattice cation polyhedral geometries were used as an initial descriptive feature to characterize both oxide chemistry and topology. Good agreement between predicted and actual data was obtained, and the model was validated by well reproducing oxide expansion values not included in the initial data set. Thus, we have demonstrated a data analytics approach for reliable calculation of a thermophysical property.

2:40 PM

(ICACC-S10-004-2020) Computational study of physical properties of 55 chalcogenide crystals

S. A. Hasan*[1]; K. Baral[1]; W. Ching[2]

1. University of Missouri, Kansas City, Department of Physics and Astronomy, USA
2. University of Missouri-Kansas City, USA

We report the results of a comprehensive study on the electronic structure, optical properties and mechanical properties of 55 binary, ternary and quaternary chalcogenide crystals calculated using density functional theory methods. The comparative study of a large number of chalcogenide crystals using the same computational method enables us to draw insightful conclusions for the first time. In particular, the concept of total bond order density (TBOD), a single quantum mechanical metric characterizing the internal cohesion of a crystal is used to compare and correlate with the calculated physical properties of these crystals. This could facilitate the exploration and development of new chalcogenide crystals and glasses for potential novel applications.

Multi-scale Modeling of Processing and Performances I

Room: Coquina Salon G

Session Chairs: Dongwon Shin, Oak Ridge National Laboratory; Ghanshyam Pilania, Los Alamos National Lab

3:20 PM

(ICACC-S10-005-2020) Modeling of ablation roughness of carbon/carbon composites from micrometer to centimeter and its consequences on transfer phenomena (Invited)

G. L. Vignoles*[1]; X. Lamboley[1]; J. Lachaud[2]; C. Levet[1]; J. Couzi[3]; J. Mathiaud[3]

1. University Bordeaux, LCTS - Lab for ThermStructural Composites, France
2. University Bordeaux, I2M, France
3. CEA, DAM/CESTA, France

During atmospheric reentry of space objects, or during solid rocket motor firing, the thermal protection materials are exposed to extreme heat fluxes; they are consequently ablated and their surface morphology changes, with the acquisition of specific features at various scales. The impact of these morphology changes on the exchanged heat flux is strong and has deep consequences on the amount of lost matter during a mission. We describe in this presentation a multi-scale study of the roughness acquired by carbon/carbon composites. First, a micrometer-scale roughness arises from reactivity differences in the composite, e.g. protruding fiber tips emerging from a weaker interphase or matrix. Several important results on roughness acquisition and on the resulting effective reactivity will be described. Second, a millimeter to centimeter roughness under the form of "scallops" is obtained on the same materials, without any relationship with its texture length scales. We prove, on the basis of numerical simulations, that this roughness arises from (i) the impact of the micro-roughness on the development of turbulence and (ii) the interaction of transition turbulence with the wall recession leading to "dune-like" shapes.

3:50 PM

(ICACC-S10-006-2020) Ab initio modeling of high entropy alloy ceramic (TiNbTaZrMo)C (Invited)

W. Ching*[1]; S. San[1]

1. University of Missouri-Kansas City, USA

We report the recent progress in the ab initio modeling of a high entropy alloy (HEA) composite (TiNbTaZrMo)C ceramic. A supercell of 500 atoms in FCC lattice is constructed with the random solid solution model such that each transition metal bonds to one C atom as nearest neighbor. After full structural relaxation, the electronic structure, interatomic bonding and mechanical properties are calculated. It is found that the FCC lattice is severely distorted and transformed into a non-cubic monoclinic-like structure. The mechanical properties of this ceramic composite is found to be extremely strong and compare well with experimental data. Detailed analysis of the electronic structure and bonding indicate the significantly strong bonding between metal ions and C is the source of this excellent property. Extension to other HEAs and the effect of variations of the compositions will also be discussed.

4:20 PM

(ICACC-S10-007-2020) Non-Destructive Electrical Characterization of Ceramic Microstructures: Inside and Outside the Furnace

M. C. Golt*[1]; E. Hernandez-Rivera[1]; K. Strawhecker[1]; S. Kilczewski[1]

1. CCDC - Army Research Laboratory, USA

Measurements of electrical properties can be fast, non-destructive, volumetric, and relatively easy to perform in-situ. Since these measurements involve the measure of the interactions of traveling electrons with the microstructure, they provide atomic resolution-a necessary capability when measuring grain boundary properties. Frequency-dependent impedance measurements provide distinction between the contributions of the grains from the grain boundaries. Analytical and numerical modeling can then be used to reconstruct microstructures, providing knowledge of the largest features in the microstructure, the grain size distribution, all the way down to the number of defect atoms at the grain boundary. What's more, we show that these measurements can be performed in real-time on the evolving microstructure during sintering, providing valuable insights into the dynamics of the sintering process. We will discuss how we have used electrical measurements to characterize ceramic microstructures, non-destructively, on large specimens, over a variety of compositions. We show how bulk electrical measurements compare with AFM and APT measurements, and how to relate processing with structure and performance. Finally, we detail how these experiments validate numerical models that help to visualize, understand, and predict the relationship between the measurement and the structure.

4:40 PM

(ICACC-S10-008-2020) Modelling of heat and mass transfer based on X-ray Computerized Tomography scans in porous carbon/carbon composites

C. Charles*[1]; G. Vignoles[1]; C. Descamps[2]

1. LCTS - CNRS, France
2. Safran ceramics, France

Cf/C composites are thermo-structural materials often used in aerospace or aeronautics industries. They contain carbon fibers and pyrocarbon matrix, often added by CVI process (Chemical Vapor Infiltration). The quality of the manufactured composites is highly related to the processing conditions. Rigorous modelling of physi-co-chemical phenomena is very helpful to optimize the CVI process. It is decisive to assess, at initial stage and during the densification, the preform properties, which are: - Geometrical characteristics such as porosity, surface area, pore size distribution - Gaseous transport properties like continuous diffusivity, Knudsen diffusivity, Darcian permeability - Heat transfer properties such as thermal diffusivity Calculation of these properties allow connecting the evolutions of the transport properties inside the porous media along the way of their infiltration. We present an approach on a family of porous media images of carbon/carbon composite preforms at several stages of matrix infiltration, for which we have been able to produce numerical estimates of the aforementioned properties. Experimental validations have been brought, especially with respect to Knudsen diffusion. Moreover, interesting correlations are seen to arise between the non-dimensional forms of the transfer coefficients and their evolution trends during progressive pore filling.

5:00 PM

(ICACC-S10-009-2020) Molecular Template Model Building Methods for Amorphous Molecular Solids (Invited)

P. Rulis*[1]

1. University of Missouri - Kansas City, Physics and Astronomy, USA

Plasma enhanced chemical vapor deposition (PECVD) may be used as a growth processes for thin-film amorphous molecular solids. Under the low-temperature and non-thermodynamic PECVD conditions the substructure of molecular precursor source ingredients is retained in the resultant amorphous thin film. However, the detailed structure of the thin film is subject to the variation of growth parameters such as substrate temperature, gas pressure, plasma energy, precursor flow rate, etc. As may be expected, the film properties are dependent on the atomic scale structure. Experimental optimization of the growth process for a target application is time consuming and expensive such that computational methods that can realistically model the possible resultant structures and enable the search for models with the desired properties. In this presentation progress on the development of such an approach will be presented. The application of atomic scale model generation to amorphous hydrogenated boron carbide will be shown using template-driven explicit bond formation methods, showcasing their capabilities and limitations. Comparison of the resultant models with variance data from fluctuation electron microscopy analysis of comparable growth samples will be used to help judge the product quality.

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

Sustainable Energy Concepts and Applications II

Room: Tomoka B

Session Chairs: Tadachika Nakayama, Nagaoka University of Technology; Sungwook Mhin, Korea Institute of Industrial Technology

9:00 AM

(ICACC-S11-023-2020) Synthesis of transition metal (TM: Co, Mn, Ni, Fe) based sulfides as an efficient electrocatalysts for overall water splitting

K. Park*[1]; J. Jeon[1]; J. Lee[3]; H. Han[4]; N. Oh[1]; S. Mhin[2]

1. Hanyang University, Division of Materials Science and Engineering, Republic of Korea
2. Korea Institute of Industrial Technology, Heat treatment R&D group, Republic of Korea
3. Korea Institute of Industrial Technology, Republic of Korea
4. Hongik University, Department of Materials Science and Engineering, Republic of Korea

Electrochemical water splitting via electrolysis has been actively studied to produce a sustainable hydrogen source for eco-friendly and efficient renewable energy. Currently, noble metal-based oxides, such as RuO_2 and IrO_2, show high electrochemical performance of water splitting. However, the scarcity and high cost of these noble metals severely hinder their large-scale practical application. Alternatively, sulfides, nitrides, phosphides, selenides, and carbides based on transition metal have been widely investigated for overall water splitting. In this work, facile synthesis of the transition metal-based sulfides for high-performance electrocatalyst is proposed using hydrothermal method. Morphological and crystallographic characteristics were examined by field emission scanning electron microscopy (FESEM), transmission electron microscopy (TEM), X-ray diffraction (XRD) spectroscopy. Also, chemical compositions and elemental chemical states were further investigated by X-ray photoelectron spectroscopy (XPS). Combined with structural characterization of the transition metal-based sulfides, electrocatalytic mechanism of the oxygen evolution reaction (OER) and the oxygen reduction reaction (ORR) in alkaline media is discussed.

9:20 AM

(ICACC-S11-024-2020) Electronically Double-layered Metal Boride Hollow Nanoprism as an Excellent and Robust Water Oxidation Electrocatalysts (Invited)

H. Han*[1]; Y. Hong[3]; S. Mhin[2]

1. Hongik University, Materials Science and Engineering, Republic of Korea
2. KITECH, Republic of Korea
3. KITECH, Republic of Korea

Metal–metalloid compounds have been paid much attention as new high-performance water oxidation catalysts due to their exceptional durability for water oxidation in alkaline media originated from the multi-dimensional covalent bonding of the metalloid with the surrounding metal atoms. However, compared to the excellent stability, a relatively low catalytic activity of metal-metalloids often limits its practical application as high-performance water oxidation catalysts. Here, we first disclose a novel self-templating strategy combined with atomic layer deposition (ALD) to design the electrochemically active and stable quaternary metal boride (vanadium-doped cobalt nickel boride, VCNB), hollow nanoprism by inducing electronic double layers on the surface.

9:50 AM

(ICACC-S11-025-2020) Optimization of oxygen concentration in rGO/g-C_3N_4 heterojunction for photoelectrochemical water splitting

M. Je*[1]; Y. Sim[2]; U. Sim[2]; H. Choi[1]

1. University of Cologne, Germany
2. Chonnam National University, Republic of Korea

Recently, 2D carbon-based heterojunction materials have exhibited high photoelectrochemical activities for water splitting. Due to excellent compatibility with other 2D materials and versatile functionalities, rGO and g-C_3N_4 have attracted strong research interests in junctions. However, the functionality of rGO-C_3N_4 compoosites is not very reliable, which means that photoelectrochemical activities for water splitting largely fluctuates with fabrication conditions. In this study, we systematically and theoretically explored the effect of O/C ratio of rGO and possibility to fine tune the photochecmial activity of rGO-C_3N_4. Interestingly, from our DFT calculations, the work functions of pristine rGO (PrGO) and defective (DrGO) were found to be linearly increase with O/C ratio. Especially, PrGO and DrGO having O/C ratio range from 4% to 13% were optimum conditions for the photocatalyst in junctions with C_3N_4, considering feasibilities of charge separations and water redox levels. Our DFT calculation further revealed that the charge transfer only occurs in PrGO with 4% O/C ratio and DrGO with specific O/C ratio. Free energy diagram and volcano plot of H adsorbed on PrGO and g-C_3N_4/DrGO heterojunction was investigated to further understand the HER mechanisms. The possibility of optimizing photocatalytic activity of rGO-C_3N_4 composite was well verified with our experiments.

10:30 AM

(ICACC-S11-026-2020) Phase Engineered 2D Transition Metal Dichalcogenides and their Applications (Invited)

W. Choi*[1]

1. University of North Texas, Department of Materials Science & Engineering, USA

Recent advances in atomically thin two-dimensional transition metal dichalcogenides (2D TMDs) have led to a variety of promising technologies for nanoelectronics, energy storages, and electro-catalysis, to name a few [1]. The 2D TMDs have recently been reported to exhibit superior properties for the hydrogen evolution reaction and other electrochemical reactions. The rich chemistry of 2D TMDs combined with engineering of their electronic and opto-electronic properties make these materials very attractive for energy storages and energy conversion catalysis. This talk will present two important subjects; (1) Synthesis of 2D TMDs and their engineering in energy bandgap and optoelectronic properties, (2) Recent developments and challenges in using 2D TMDs as active materials in energy storages. In addition, 2D MoS_2 coating in Li-metal anodes for advanced rechargeable batteries will be introduced - 2D MoS_2 coated Li-metal demonstrated a three-fold improvement in cycle-life than using bare Li-metal. In a Li-S full cell configuration, we obtain a specific energy density of ~600 $Whkg^{-1}$ and a Coulombic efficiency of ~98% for over 1200 cycles at 0.5 C. These new materials can lead to open the way for advances in energy storages and catalysis [2].

11:00 AM

(ICACC-S11-027-2020) Mapping defect equilibria using density functional theory calculations and thermodynamic modeling for photochemical reaction boosting (Invited)

H. Choi*[1]

1. University of Cologne, Germany

Photochemical and electrochemical reactivities of nanomaterials are determined by several factors, which are difficult to measure only via experiments, such as optical transition characteristics, mechanism of carrier transport, and electron orbital shapes of surface atoms. Point defects of photocatalytic semiconductors largely govern the photocatalytic activity by determining the charge transfer efficiency, carrier lifetime, photoabsorption efficiency, and band levels, and so on. It is ideal to map the defect equilibria of candidate materials for water splitting photocatalytic materials for efficiency processing optimizations. However, it is extremely time-consuming and exhaustive work to derive defect formation energies and relate the effect of each defect and the resultant photochemical reactivity. Modern quantum mechanical simulations based on density functional theory (DFT) are capable of providing reliable values of thermodynamic parameters and electronic structures. Hence, DFT calculations are widely used in analyses and designs of photocatalysts. In this talk, I will present my original strategies to boost water splitting efficiencies of photocatalytic semiconductors and electrocatalysts: theoretical mapping of the beneficial and deterimental point defects in photocatalytic semiconductor solids.

11:30 AM

(ICACC-S11-028-2020) First-Principles calculations on charge carrier effective masses of $La_{0.5}Sr_{0.5}FeO_3$ under high temperature and low oxygen partial pressure (Invited)

D. Lee*[1]; Y. Shin[1]

1. Pohang University of Science and Technology(POSTECH), Materials Science and Engineering, Republic of Korea

Lanthanum ferrite, $LaFeO_3$, a mixed ionic and electronic conductor, is a promising material for various applications, such as Solid Oxide Fuel Cells (SOFCs), electrolysis membranes, catalyst, gas sensor and thermoelectric (TE) devices. Especially, it has shown superior electronic and ionic conductivity when it is doped with other ionic species such as Sr, Ca, Ba and other transition metals. However, there are still a couple of issues that need to be taken cared for $LaFeO_3$ to be used for various industrial applications. In this study, first-principles density functional theory calculations are employed to understand the electronic structure and charge carrier effective masses of $La_{0.5}Sr_{0.5}FeO_3$ under high temperature and low oxygen partial pressure. Our study shows that the variation of oxygen partial pressure significantly affects the electronic structure of $La_{0.5}Sr_{0.5}FeO_3$ and thus affects the carrier mobility. Our calculation results will be a cornerstone for controlling the electronic properties of $LaFeO_3$ by using various dopants.

S12: On the Design of Nano-Laminated Ternary Transition Metal Carbides/Nitrides (MAX Phases) and Borides (MAB Phases), and their 2D Counterparts (MXENES, MBENES)

Current Progress in MXenes II

Room: Coquina Salon F
Session Chairs: Surojit Gupta, University of North Dakota; Athanasios Gkountaras, CNRS

8:30 AM

(ICACC-S12-025-2020) Structure-Property Relationships of MXenes

S. B. Sinnott*[1]

1. The Pennsylvania State University, Materials Science and Engineering, USA

The discovery and design of new materials is an important part of the process by which application improvement takes place. Material modeling methods are now widely applied in pursuit of these objectives. This presentation will review the evolution of some common material modeling methods and their integration with cutting-edge experimental techniques to elucidate the structure-property relationships of MXene materials. In particular, high-throughput density

functional theory calculations are used to identify stable MXene materials under a variety of compositions and environmental conditions. The affect of heterogeous structures on the properties of MXenes will also be examined.

9:10 AM

(ICACC-S12-026-2020) Energy storage properties of MXenes (Invited)

D. Cakir*[1]; C. Sevik[2]; F. Peeters[3]; O. Gulseren[4]

1. University of North Dakota, Physics and Astrophysics, USA
2. Eskisehir Technical University, Turkey
3. University of Antwerp, Belgium
4. Bilkent University, Turkey

Energy is key to progress in both developed and under-developed countries. A modern energy infrastructure requires technology for storage such as batteries and capacitors. Efficient energy storage requires novel materials such as two-dimensional (2D) materials composed of stacked sheets where each molecule-thin sheet has properties not found in conventional three-dimensional materials. In this respect, transition metal carbides/carbonitrides (MXenes) are the newest class of 2D materials, and they offer great potential in chemical energy storage applications. In this talk, I will discuss the computational design of MXenes and MXene based hybrid structures for battery and supercapacitor applications. I will mainly focus on the methods to enhance the energy storage properties of MXenes.

9:40 AM

(ICACC-S12-027-2020) Understanding Pseudocapacitive Energy Storage in MXenes (Invited)

D. Jiang*[1]

1. University of California, Riverside, USA

Pseudocapacitive energy storage can involve either surface redox or intercalation process. In this talk, we will present our recent theoretical studies to understand the surface redox chemistry of MXenes in sulfuric acid and to predict the surface charge vs. potential curve. In addition, we will apply the method to screen different MXenes and to come up with descriptors to quickly obtain trends for carbides and nitrides. Last, we will show ab initio molecular dynamics simulations of the proton transport across the interface and in confined water layers for MXenes and MXene/graphene heterostructures.

10:30 AM

(ICACC-S12-028-2020) Termination Species and Chemical Bonding in MXenes Investigated by X-ray Spectroscopy and ab initio Theory (Invited)

M. Magnuson*[1]

1. Linkoping University, Sweden

The electronic structures and the adsorption sites of the termination species in the 2D ceramic materials MXenes are investigated by X-ray diffraction and X-ray spectroscopy compared to ab initio electronic structure calculations. Valence band studies using synchrotron radiation reveals how the structures react to increasing temperature. Calculated spectra using density-functional theory (DFT) including core-to-valence dipole matrix elements are found to yield consistent spectral functions of experimental data. By varying the termination species, constituting elements and structures in MXenes, a change of the electron population cause a change of covalent bonding between the laminated layers, that enables tuning of the macroscopic properties of the materials. The role of functional –F and –O and –OH temination groups at the interfaces and their local symmetries at different adsorption sites are discussed. Synchrotron radiation techniques such as bulk-sensitive X-ray absorption, EXAFS and X-ray emission spectroscopies are shown to be particularly useful for detecting detailed symmetry in the electronic structure that yield anisotropic information about internal monolayers and termination groups at the interfaces. Angle- and polarization-resolved measurements are shown to reveal differences in orbital occupation across and along the laminate basal planes.

11:00 AM

(ICACC-S12-029-2020) MXene surface chemistry (Invited)

P. Persson*[1]

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

Since their discovery in 2011, MXenes have outperformed existing materials for a range of applications such as energy storage, water filtering, electromagnetic shielding, as catalysts for H_2 evolution from water and as effective materials for capturing CO_2 to name but a few examples. Their outstanding performance is accredited to a range of properties, e.g. hydrophilic and conductive, that can be attributed to a rich transition metal chemistry. Ultimately, the range of properties are dictated by the tailoring potential of the MXenes. The general formula to describe MXenes, $M_{n+1}XT_x$, identifies that the tailoring potential in the MXene family is vast in contrast to commonly employed 2-dimensional structures. In addition to choice of thickness (n) and X element, M can be a single transition metal element, or an ordered or disordered combination between multiple M elements. The thinner the MXene (low n), the more dependent are the emerging properties on the surface elements. Therefore, the range and corresponding potential mix of terminating elements or molecules is an apparent key in determining the final MXene properties. On top of this, the surfaces may be further decorated by functional elements. This work primarily adopts advanced electron microscopy methods to reveal the state of the art available tailoring of the MXene structure and chemistry, owing to recent advances in tuning of surface terminations, surface decoration and emerging properties.

S13: Development and Applications of Advanced Ceramics and Composites for Nuclear Fission and Fusion Energy Systems

Coating Technologies for Reactor Components

Room: Coquina Salon H

Session Chair: Konstantina Lambrinou, SCK-CEN

8:30 AM

(ICACC-S13-030-2020) Multifunctional nanoceramic barrier for DEMO breeding blanket concepts

B. Paladino*[1]; M. Vanazzi[1]; D. Iadicicco[1]; P. Munoz[2]; T. Hernandez[2]; S. Bassini[3]; M. Utili[3]; F. Di Fonzo[1]

1. Italian Institute of Technology (IIT), Italy
2. CIEMAT, Spain
3. ENEA, Italy

The breeding blanket will be an in-situ source of Tritium for future generation fusion reactors. A permeation barrier is required to confine it. Two are the eligible breeding materials: eutectic Pb-16Li and a pebble bed of Lithium ceramics. These both lead to severe corrosive attack on steel. We report on nanoceramic Aluminum Oxide coatings grown by Pulsed Laser Deposition (PLD) technique. Coatings appear compact and well-adherent on the substrates, showing metal-like mechanical properties combined with high hardness. They have been tested as permeation barriers (with Hydrogen and Deuterium) at different temperatures (from 350°C to 650°C). They showed a permeation reduction factor (PRF) up to 10^5 at 650°C. These results have been confirmed also under 1.8 MeV electron irradiation. In addition, samples have been exposed to corrosion tests (both Pb-16Li and Lithium ceramic pebbles) up to 10000 hours. No corrosive attacks on the steel substrate were

detected. Finally, Atomic Layer Deposition technique has been recently employed as a complementary technique. Preliminary results confirm the feasibility of the technique. To conclude, in a DEMO-oriented perspective, Oxide coatings present interesting properties as multifunctional protective barriers, proving to be one of the few possible solutions for the material-related issues of all the breeding blanket concepts currently considered.

8:50 AM

(ICACC-S13-031-2020) Ceramic oxide coatings for Water Reactors: From selection of materials to coating development

M. Cabrioli*[1]; E. Frankberg[1]; M. Vanazzi[1]; K. Van Loo[2]; J. Vleugels[2]; K. Lambrinou[3]

1. Istituto Italiano di Tecnologia (IIT), Italy
2. Katholieke Universiteit Leuven (KU Leuven), Belgium
3. SCK-CEN, NMS, Belgium

Radical performance and safety improvements at economically attractive conditions are fundamental basis of Accident Tolerant Fuel (ATF) concepts. Coatings were recently regarded as near-term evolutionary option to integrate engineered surface properties, for enhanced corrosion resistance and accident tolerance, in the fabrication of traditional cladding materials for water cooled reactors. In this work, a preliminary screening of several ceramic oxides is presented. Candidate materials are produced in the form of sintered pellets, tested and characterized in the framework of the European project IL TROVATORE. Bulk polycrystalline specimens are exposed to pure water at 360°C and 18.7 MPa, to simulate the Pressurized Water Reactor (PWR) environment during normal operation. Mass changes, monitored during the exposure, are correlated to surface and structural modifications of the samples. In addition, most promising oxides are considered for the fabrication of protective coatings, to be deposited by the Pulsed Laser Deposition (PLD) technique on standard substrates as Zirconium alloys and nuclear grade steels. To conclude, the feasibility of coating deposition by means of PLD technique is proven for relevant materials. Moreover, their interaction with PWR ambient is tested, as first stage towards the assessment of their performance as ATF solution in water cooled reactors.

9:10 AM

(ICACC-S13-032-2020) Development of coatings on SiC for LWR fuel cladding

P. Mouche*[1]; T. Koyanagi[1]; Y. Katoh[1]

1. Oak Ridge National Laboratory, USA

The dissolution of silica in oxidizing chemistries and the possibility of networked micro-cracks enabling fission gas release point to the need for an additional barrier on SiC/SiC composites for light water reactor use. One of the major challenges is overcoming the evolving stresses from irradiation swelling of the SiC, as well as intrinsic radiation effects in the coating and the interface. Therefore, fundamental research is required to determine the key parameters that lead to an adherent, ductile, corrosion resistant coating during normal operation. To understand the as-deposited mechanical properties of the currently fabricated coatings, x-ray residual stress analysis, scratch tests, and adhesion tests were compared to wider autoclave, thermal cycling, and micro-mechanical testing. Data were compared between first generation CrN, TiN, and Cr coatings deposited via unfiltered cathodic arc and newer Cr and Ti high pulse magnetron sputtered and partially filtered cathodic arc coatings. New coatings have shown lower impurity levels, better interfacial adhesion, and tunability of residual stress. This work was sponsored by the U.S. DOE, Office of Nuclear Energy, AFC of NTRD program under contact DE-AC05-00OR22725 with ORNL managed by UT Battelle, LLC., and Westinghouse Electric Company/General Atomics FOA projects.

9:30 AM

(ICACC-S13-033-2020) Micro-mechanical testing of Cr coated Silicon Carbide

D. Patel*[1]; T. Koyanagi[2]; P. Mouche[2]; Y. Katoh[2]

1. University of Michigan, Nuclear Engineering, USA
2. Oak Ridge National Laboratory, USA

SiC/SiC composite concept is among the leading candidate accident tolerant fuel cladding material due mainly to its excellent steam oxidation resistance and high temperature mechanical performance. However, prototypical BWR coolant environments have shown concerning SiC dissolution, rather than passivation. Furthermore, fission product gas retention under irradiation is another concern. Coating the outer surface of the cladding can mitigate corrosion and improve hermiticity. This research compares mechanical performance of Cr coatings on SiC using nanoindentation techniques. Two types of pure Cr coatings on monolithic chemical vapor deposited SiC were tested. To study the bonding performance, micro-cantilevers were fabricated at SiC/Cr interface and tested using cube-corner nanoindentation tip. Excellent bonding performance is seen during micro-cantilever testing: the coating showed plastic deformation without interface failure. In addition, to investigate the plastic deformation behavior of the coating, strain rate sensitivity of the coatings using conventional nanoindentation tests will be investigated: ranging from conventional strain rates (10^{-3} - 1 s^{-1}) to very high strain rates (up to 20 s^{-1}). This is confusing. See my RESsolutions comments.

9:50 AM

(ICACC-S13-034-2020) Particle Atomic Layer Deposition of Tungsten Nitride as a Hydrogen Environmental Barrier Coating

S. Bull*[1]; W. W. McNeary[3]; C. Adkins[2]; T. Champ[1]; C. Hill[1]; R. O'Brien[2]; C. Musgrave[1]; A. W. Weimer[1]

1. University of Colorado Boulder, Chemical Engineering, USA
2. Idaho National Lab, USA
3. National Renewable Energy Laboratory, USA

Nuclear thermal propulsion is an attractive in-space propulsion technology due to its large specific impulse. This specific impulse is achieved by using energy released from uranium fission to heat hydrogen gas to over 2500°C before expelling it from a supersonic nozzle to generate thrust. However, hydrogen diffuses into the nuclear fuel elements and embrittles them. In this research we investigate the use of nanoscale tungsten nitride (WN) coatings to address this issue. Ultrathin WN films were deposited by atomic layer deposition (ALD) on zirconia nanoparticles and yttria stabilized zirconia (YSZ) micropowders. Differential thermal analysis (DTA) was used to investigate the efficacy of the W/WN films in preventing hydrogen attack. The temperature at which hydrogen reacted with the sample increased with film thickness, thereby indicating that the film inhibited this reaction. However, the existence of a hydrogen reaction peak in the thickest film indicated a material with a smaller hydrogen diffusivity is necessary for a substantial decrease in hydrogen interaction with the substrate. Therefore, density functional theory was used to compare W to other high temperature refractory ceramics as alternative coating materials. This is the first study for ALD of WN on particles from bis(t-butylimido)bis(dimethylamino)tungsten(VI).

Chemical Compatibility and Corrosion

Room: Coquina Salon H
Session Chair: Konstantina Lambrinou, SCK-CEN

10:30 AM

(ICACC-S13-035-2020) Application of Empirical Transition-State Theory to Corrosion of SiC in Light Water Reactors

P. J. Doyle*[1]; S. Raiman[2]

1. University of Tennessee, Nuclear Engineering, USA
2. Oak Ridge National Lab, USA

Understanding the hydrothermal and radiation-accelerated corrosion of nuclear ceramics is of critical importance for the safe, reliable, and cost-effective operation of light water reactors (LWRs). Historically, corrosion predictions, and chemical reaction analyses more generally, have focused on the use of the Arrhenius equation which allows accurate prediction of reaction rates as a function of activation energies, absolute temperature, and an empirical pre-exponential factor. However, Arrhenius lacks a scientific interpretation of the pre-exponential factor, which could aid in the understanding of reaction mechanisms. Transition State Theory (TST) was introduced in 1933 to address this problem by describing the factor in terms of physical constants, reaction temperature, and the reaction's activation entropy. Despite applicability to a wide range of chemical systems, TST has been underutilized and inconsistently applied, especially in corrosion science and the surface sciences more generally. In the present work, a unifying standard is proposed and applied to literature data, yielding adjustments to the reported activation entropy ranging from -61 to +53 J/mol-K. The method was then applied to the hydrothermal corrosion of SiC with and without radiation damage to estimate activation energies and entropies. Mechanistic insights from the activation parameters are suggested.

10:50 AM

(ICACC-S13-036-2020) Hydrothermal Corrosion and Irradiation of Dual-Purpose Coatings on Silicon Carbide

S. S. Raiman*[2]; P. J. Doyle[1]; T. Koyanagi[2]; Y. Katoh[2]

1. University of Tennessee, Nuclear Engineering, USA
2. Oak Ridge National Laboratory, USA

Silicon carbide ceramic matrix composite (CMC) materials have shown promise as next generation light water reactor (LWR) fuel cladding. Major unresolved issues include compatibility with high temperature water, and hermeticity of CMC cladding. To resolve these issues, dual-purpose coatings have been proposed. This talk presents recent experimental results in which SiC materials with CrN, Cr, and TiN coatings were exposed in a continuously refreshing autoclave for up to 2000h in BWR conditions (288°C water with either 2 wppm dissolved oxygen or 150 wppb dissolved hydrogen to simulated boiling water reactor normal water chemistry (NWC) and hydrogen water chemistry (HWC)) or simulated PWR primary water (320°C with 3 wppm dissolved hydrogen). Autoclave tests are compared to samples irradiated at the MIT nuclear reactor to assess the combined effects of irradiation and corrosion, and proton irradiation experiments are used to further understand in-reactor performance of coating candidates.

11:10 AM

(ICACC-S13-037-2020) Evaluation of Coatings on SiC Ceramics for Hydrothermal Corrosion Protection

W. Kim*[1]; D. Kim[1]; H. Lee[1]; J. Park[1]

1. Korea Atomic Energy Research Institute, Republic of Korea

Silicon carbide (SiC)-based ceramics and composites have benefits such as excellent high-temperature mechanical properties and irradiation tolerance. They also exhibit a low chemical reactivity with high-temperature steam and thus a reduced hydrogen production in severe accidents. Owing to these excellent safety features under severe accident conditions, there have been large efforts on applying the SiC composites to nuclear fuel claddings and fuel components for an enhanced accident tolerance of light water reactor (LWR). Among the various technical issues, hydrothermal corrosion and fission gas retention capability are considered to be critical concerns for the application of SiC composites in LWR fuel claddings. Development of coatings on the surface of SiC composite claddings are underway as a possible way of corrosion protection and gas hermeticity. In this study, we applied various metallic and non-metallic coatings in order to mitigate the hydrothermal corrosion of SiC ceramics. Microstructures, integrity and corrosion behavior of various coatings on SiC ceramics are presented.

11:30 AM

(ICACC-S13-038-2020) High Temperature Oxidation Behaviors of SiC Under Air/Water Vapor Ingress Conditions

Y. Cho[1]; K. Lu*[1]

1. Virginia Tech, USA

SiC has been used in TRISO fuel particles developed for high-temperature gas-cooled reactors (HTGRs). Despite the well-recognized safety, durability, and efficiency, TRISO fuel behaviors under air and steam ingress accidental conditions are unknown. This study focuses on the oxidation behaviors of bulk SiC under air/water vapor ingress conditions with low partial pressures at elevated temperatures. CVD SiC samples have been exposed to 1400 °C for ~12 h in Ar/water vapor/air mixed atmospheres. Weight changes, phase evolution, surface and cross-section morphologies are analyzed. No significant weight changes are observed after the tests, but SiO_2 layers with a cristobalite phase formed on the surface become thicker with increasing the temperature and time. The oxidation kinetics are derived by evaluating the formations of gaseous species and condensed solids. Also, oxide thickness and weight change at various atmospheres are estimated using analytical models.

11:50 AM

(ICACC-S13-039-2020) High Temperature Oxidation Behaviors of Nuclear Graphite Under Water Vapor Ingress

Y. Cho[1]; K. Lu*[1]

1. Virginia Tech, USA

Graphite matrix for high temperature gas-cooled reactors (HTGRs) contains TRISO fuel particles, removes fission heat, and reduces neutron fluence and thermal stress. One major concern for the graphite matrix in this accident is low oxidation stability at high temperatures. An oxidizing atmosphere will lead to oxidation and burn-off of the graphite matrix and finally result in fuel failure. Little research has studied the effects of water vapor with very low partial pressures for accidental conditions of HTGRs. To ensure the safety, durability, and efficiency of TRISO fuels for next-generation reactors, comprehensive understanding of oxidation behaviors of nuclear graphite under accidental conditions is of paramount importance. This study is to understand the oxidation behaviors of nuclear graphite under water vapor ingress at elevated temperatures. Oxidation tests are conducted at ~1400 °C for ~12 h in a mixed Ar/water vapor atmosphere. In-situ and post-oxidation behaviors including weight change, pore evolution, and gaseous products are investigated. The oxidation rates increase with the temperature and time, showing different oxidation regimes. Depth profiles of oxidant concentration and oxidation depths are estimated using analytical models.

12:10 PM

(ICACC-S13-040-2020) Differences in steam oxidation kinetics and scale volatility between monolithic SiC variants and SiC/SiC composite

K. Kane*[1]; S. Uwanyuze[2]; B. A. Pint[1]

1. ORNL, MSTD, USA
2. University of Connecticut, Materials Science and Engineering, USA

Accident tolerant fuel cladding can provide increased coping time during beyond design basis accidents in light water reactors and SiC/SiC composites are attractive candidate particularly because of their very high temperature oxidation resistance in steam environments (>1700°C). In order to model this behavior in accident scenarios, dense SiC (e.g. CVD or Hexoloy) specimens are often substituted in laboratory testing. The current project compared the oxidation kinetics, reaction products and volatility rates under various high temperature steam conditions in order to compare to SiC/SiC composite specimens, which have limited availability. This work was sponsored by the U.S. Department of Energy Office of Nuclear Energy, Advanced Fuels Campaign.

Material Technologies for Enhanced Accident Tolerance LWR Fuels and Core I

Room: Coquina Salon H
Session Chair: Weon-Ju Kim, Korea Atomic Energy Research Institute

1:50 PM

(ICACC-S13-041-2020) In situ characterization of the damage and fracture in SiC cladding and TRISO fuel particles at elevated temperatures

D. L. Liu*[1]; S. Knol[2]; M. Davies[3]; A. Vreeling[2]; P. Xu[4]; R. Lu[4]; E. J. Lahoda[4]; R. O. Ritchie[5]

1. University of Bristol, School of Physics, United Kingdom
2. NRG, Netherlands
3. USNC, USA
4. Westinghouse Electric Company, USA
5. Lawrence Berkeley National Laboratory, USA

Nuclear reactor core components usually work under extreme conditions with high temperatures and neutron irradiation. Accordingly, characterizing damage and fracture in these components requires in situ observations to discern the underlying physical mechanisms. In this work, in situ C-ring compression combined with X-ray computed micro-tomography (XCT) and digital volume correlation methods have been conducted to reveal deformation and fracture mechanisms in four types of SiC-SiC cladding materials with different coating/composite ratio at temperatures up to 1200°C in Air and inert gas Ar. The correlation between the microstructure, the crack initiation and eventual failure modes will be presented and discussed. In addition, uniaxial compression tests of dedicated TRISO fuel particles have been undertaken at 1000°C with real-time 3D XCT imaging of the evolution of damage. Specifically, vertical cracks were found to form inside the Hertizan zone rather than outside; the initiation and propagation of these cracks vary significantly with particle types. It is emphasized that the fracture behavior of these materials can be markedly different between ambient and elevated temperatures; indeed, we found that the relaxation of residual stresses played a critical role in modifying the high-temperature mechanical behavior and irradiation performance of these materials.

2:10 PM

(ICACC-S13-042-2020) Characterization of heterogeneity distribution of SiC_f/SiC_m composite using X-ray computed tomography (XCT)

J. Nance*[1]; H. T. Nagaraju[2]; G. Subhash[2]; R. Haftka[2]; B. Sankar[2]

1. University of Florida, Material Science Engineering, USA
2. University of Florida, Mechanical and Aerospace Engineering, USA

Continuous silicon carbide fiber and silicon carbide matrix (SiC_f/SiC_m) composites is a promising candidate material for nuclear fuel cladding applications. However, the fabrication process is inherently prone to introduction of heterogeneity in the microstructure and geometry. The former includes variations in the fiber diameter, fiber angle orientation, porosity, etc. The latter includes variations of tube wall thickness, weave angle, inner/outer diameter, etc. These variations can manifest into a large distribution of mechanical properties. Utilizing XCT, digital image processing techniques, and statistical analysis, the current study provides quantitative description of the above heterogeneities for ongoing mechanistic analysis of the deformation behavior of these composites.

2:30 PM

(ICACC-S13-043-2020) X-ray tomography characterization of neutron irradiated SiC–SiC composites

J. D. Arregui-Mena*[1]; T. Koyanagi[2]; E. Cakmak[2]; G. Singh[3]; C. Deck[4]; Y. Katoh[2]

1. Oak Ridge National Lab, Nuclear Materials Science & Technology Group, USA
2. Oak Ridge National Laboratory, USA
3. University of Tennessee, USA
4. General Atomics, USA

SiC–SiC composites are being developed as a cladding material for light water reactor (LWR) fuels. These materials are manufactured to withstand the extreme environment of a nuclear reactor. Among the most critical challenges to the SiC–SiC composite cladding is the ability to contain gaseous fission products generated by the burning fuels. Potential loss of gas tightness arises from a combination of irradiation-induced thermal conductivity reduction, swelling gradient, and the resulting complex stress state within the cladding. To understand the complex irradiation effects on micro-cracking in SiC–SiC composites x-ray computed tomography (XCT) was performed with neutron-irradiated samples under a prototypical heat flux. The analysis of the XCT scans revealed the crack propagation mechanisms in these materials. These results will inform the designers and manufacturers of the directions of architectural and fabrication process improvements toward improved cladding performances in the operating reactor environment.

2:50 PM

(ICACC-S13-044-2020) Application of laser flash method in measurement of thermal diffusivity of SiC composite tubes

T. Koyanagi*[1]; H. Wang[1]; J. D. Arregui-Mena[1]; Y. Katoh[1]

1. Oak Ridge National Laboratory, USA

Thermal diffusivity is one of the most important physical properties for assessing the performance of SiC/SiC composite fuel cladding of light water reactors. It is used to calculate thermal conductivity along with density and specific heat. However, there is a significant lack of data for tube materials because measurement is challenging owing to the specimen geometry. The thermal diffusivity of SiC composite plate has been studied, but it was unknown whether it would be different for tubular SiC composite. The objective of this study is to develop a method for accurate measurement of thermal diffusivity of SiC composite tubes. Laser flash apparatus (Netzsch LFA467 HT) was used to conduct thermal diffusivity measurements of curved SiC composite coupons machined from tubes. The system

has the unique capability of monitoring the temperature of a selected area on the specimen, which enabled improvement of measurement accuracy; by minimizing the analyzed area, the effect of specimen curvature on the thermal diffusivity became ignorable. Furthermore, this study will explore modeling of transient heat transport of a curved coupon using FEM to improve analysis of the thermal diffusivity experiment. This study was supported by US DOE Nuclear Energy and Westinghouse Electric Company/General Atomics FOA projects under contract DE-AC05-00OR22725 with ORNL managed by UT-Battelle, LLC.

Material Technologies for Enhanced Accident Tolerance LWR Fuels and Core II

Room: Coquina Salon H
Session Chair: Takaaki Koyanagi, Oak Ridge National Laboratory

3:30 PM

(ICACC-S13-045-2020) Thermomechanical Analysis of SiC-SiC Cladding with U_3Si_2 Fuel System

G. Singh*[1]; R. Sweet[1]; A. Nelson[2]; J. Harp[2]; B. Wirth[1]; Y. Katoh[2]

1. University of Tennessee Knoxville, USA
2. Oak Ridge National Laboratory, USA

Silicon carbide fiber-reinforced silicon carbide matrix (SiC-SiC) composite cladding and uranium silicide (U_3Si_2) fuel is an accident tolerant fuel concept that has the potential to deliver superior performance and enhanced safety for light water reactors. In this work, the thermomechanical analysis of SiC-SiC cladding with U_3Si_2 fuel under normal operating conditions was performed. The analysis was carried out using the fuel performance code Bison and employed the latest available properties for the SiC-SiC and U_3Si_2 materials. The results show that the U_3Si_2-SiC fuel system can lower the fuel temperature by several hundreds of degree celcius, have relatively flat temperature distribution in the fuel pellet which can possibly mitigate fuel cracking, and exhibit a significant delay in fuel-cladding gap closure when compared to UO_2-SiC fuel system. Through sensitivity analysis, this work also identified the key material properties needed for this fuel system for further evaluation and qualification purpose.

3:50 PM

(ICACC-S13-046-2020) Impact of Control Blade on the Deformation Behavior of SiC-SiC Channel Box in a BWR

G. Singh*[1]; J. Gorton[1]; D. Schappel[1]; B. S. Collins[2]; N. Brown[1]; B. Wirth[1]

1. University of Tennessee Knoxville, USA
2. Oak Ridge National Lab, USA

Silicon carbide fiber-reinforced silicon carbide matrix (SiC-SiC) composite is an accident tolerant material concept for applications in LWR fuel cladding and core structures. This work focuses on evaluating the deformation behavior of the SiC-SiC channel box in BWR conditions for three different positions of the control blade in the fuel assembly (fully withdrawn, half inserted and fully inserted). The neutron flux and temperature distribution were calculated using the coupled MPACT and CTF codes. The structural analysis to evaluate the distortion behavior was performed using the fuel performance code BISON as well as the commercial software Abaqus. The results indicate that channel box will undergo time dependent lateral bowing which will be governed by the SiC-SiC swelling under irradiation. The bowing behavior is strongly dependent on the control blade position, and will lead to temporary interference between the control blade and channel box. The developed stresses are expected to generate microcracking in the channel box.

4:10 PM

(ICACC-S13-047-2020) Grid-to-Rod Fretting Wear of Accident-Tolerant Fuel Claddings in Pressurized Water

J. Qu*[1]; C. Kumara[1]; B. Reed[1]; R. Lu[2]

1. Oak Ridge National Laboratory, Materials Science and Technology Division, USA
2. Westinghouse Electric Company, USA

Pressurized water nuclear reactors (PWRs) can experience grid-to-rod fretting (GTRF), which may produce progressive wear damage on the fuel claddings causing subsequent leakage of radioactive fuel. Accident-tolerant fuel (ATF) concepts are being developed for light water reactors. One approach is to use new cladding materials or coatings with higher resistance to the high temperature steam. This study investigated the wear resistance and wear modes of candidate ATF cladding materials in a simulative GTRF environment using a unique autoclave fretting rig. 20-hr and 100-hr fretting tests were conducted on a candidate Cr-based coating and a silicon carbide cladding against production Zr alloy grids under a realistically low load (~0.5 N) in D.I. water at a temperature of 204 ºC under 23 bars. Test conditions were designed to mimic the environment in an industrial full-assembly reactor core simulator. Both candidate ATF cladding materials showed significantly improved resistance to both oxidation and fretting wear compared to a production Zr alloy cladding. The surface roughness of the harder ATF claddings needs to be carefully controlled to avoid the risk of increased wear on the softer grid.

4:30 PM

(ICACC-S13-048-2020) Status of Accident Tolerant Fuel SiC Modeling at General Atomics

C. P. Ellis*[1]; R. Hon[1]

1. General Atomics, Nuclear Technologies and Materials, USA

Advanced fuel performance codes are required to properly model Accident Tolerant Fuel (ATF) designs incorporating silicon carbide (SiC) materials, specifically SiC-SiC ceramic matrix composite and monolithic SiC. This work will provide an overall status of recent activities related to SiC modeling at General Atomics, covering a broad range of computer codes and methods. Irradiation testing of SiC composite cladding with ADOPT UO_2 fuel is scheduled to occur in the Advanced Test Reactor in 2020. Modeling of the fuel system was performed using MATLAB and ANSYS to ensure the cladding remains intact and provides a hermetic barrier to fission product release. Results including predicted SiC cladding stresses and fuel rod temperature profiles will be discussed. Preliminary evaluation of SiC cladding performance under accident conditions was performed and compared to typical light water reactor materials. Calculations used TRACE, FRAPCON and FRAPTRAN codes. For the worst accident, results showed SiC cladding can provide over 2 hours of additional coping time compared to Zircaloy. Initial work comparing BISON to experimental mechanical test results (specifically expanding plug data) showed excellent agreement. In addition, a multi-organizational SiC BISON user group was formed to coordinate SiC-based improvements to the code and expedite ATF modeling development. Its participants and status of objectives will be presented.

4:50 PM

(ICACC-S13-049-2020) Accelerated Development of MAX Phases for Accident-Tolerant Fuels (ATFs)

K. Lambrinou*[1]

1. SCK-CEN, NMS, Belgium

This lecture presents the challenges involved in the accelerated development of MAX phase coating materials for accident-tolerant fuel (ATF) clads intended for use in Gen-II/III light water reactors (LWRs). Innovative ATF clads are designed so as to overcome the inherent technical shortcomings of standard zircaloys.

The MAX phases are layered ternary carbides/nitrides described by the $M_{n+1}AX_n$ general chemical formula, where M is an early transition metal, A is an A-group element, X is C or N, and n = 1, 2 or 3. Material development in the nuclear sector typically involves successive cycles of material production, neutron irradiation and post-irradiation examination, until the new materials comply with the property requirements of the end application. Such an approach is both time-consuming and costly, while great financial/technical benefits could result from an accelerated material development approach where application-driven material design, material production and material performance assessment are interconnected. This approach relies on the use of material screening tools, such as ion/proton irradiation, to reliably assess material performance prior to in-pile testing. This work describes the stepwise development and performance assessment (coolant compatibility, radiation tolerance) of MAX phase coating materials in the (Zr,Nb,Ti,Cr)-(Al,Sn)-C system designed for deposition on zircaloy substrates.

S14: Crystalline Materials for Electrical, Optical and Medical Applications

Optical Material IV

Room: Halifax A/B

Session Chairs: Tohru Suzuki, National Institute for Materials Science; James Wollmershauser, U.S. Naval Research Laboratory

8:30 AM

(ICACC-S14-022-2020) Structural and Electrical Properties of Co^{2+} ions Substituted Fe_2O_3-Li_2O-B_2O_3-Bi_2O_3 Glass Ceramics

S. Kumar*[1]

1. Deenbandhu Chhotu Ram University of Sc. Tech. Murthal, Physics, India

Glass-ceramics containing TMI are suitable for use as gas sensors, solid-state electrolyte materials for batteries and medical tools. The Co^{2+} ions substituted Fe_2O_3-Li_2O-B_2O_3-Bi_2O_3 glasses were synthesized via melt quench technique. The XRD patterns of the as-prepared and annealed samples at 400ºC for 04 hours & 06 hours containing CoO and/or Fe_2O_3 confirm the glassy phase and crystalline nature i.e. presence of cubic Fe_3O_4 phase. The particle size (D) increases as the concentration of iron ions decreases in the prepared samples. For annealed samples, the intensity of all peaks decreased (i.e. D) with an increase of CoO contents in glass matrix and study reveals that Fe^{4+} ions dominate over cobalt ions in the glass-ceramics matrix. FTIR studies suggest a significant shifting and change in area under the curves with a decrease of Fe_2O_3 content confirms the formation of non-bridging oxygen (NBOs). AC & DC conductivity of prepared samples has also been measured in the temperature range of 343K to 573K shows that prepared glasses were semiconducting. A decrease in dc conductivity on increasing the cobalt oxide content in place of iron oxide marked the blocking effect of iron. Different parameters (viz. Debye temperature(D), iron ion concentration (N), the distance between ion sites (R) etc. corresponding to small polaron hopping conduction were successfully evaluated and analyzed.

8:50 AM

(ICACC-S14-023-2020) Processing and characterization of high-entropy sesquioxide optical ceramics (Invited)

G. Zhang[1]; Y. Wu*[1]

1. Alfred University, Kazuo Inamori School of Engineering, USA

Equal molar proportion of high-entropy sesquioxide $Gd_{0.4}Dy_{0.4}Yb_{0.4}Lu_{0.4}Y_{0.4}O_3$ transparent ceramics were successfully fabricated with La_2O_3 and ZrO_2 as sintering additives through a vacuum sintering. Synthesized material has a density of 7.59g/cm^3 measured via the Archimedes method, with a relative density of 99.98%. Measured diagonal length of indention was 50.42 ± 0.79 μm, leading to a Vickers hardness of 7.15 ± 0.22 GPa. The highest in-line transmittance in visible and IR range is nearly 80%. By incorporating the high entropy concept with transparent ceramics, this will largely broaden the family of transparent ceramics.

9:20 AM

(ICACC-S14-024-2020) Control of transparency in polycrystalline ceramics by colloidal processing and SPS (Invited)

T. S. Suzuki*[1]

1. National Institute for Materials Science, Ceramics Processing Group, Japan

Transparent polycrystalline ceramics provides flexibility in size and shape design to apply for a wide field. However, extremely low porosities are necessary for high transparency and high temperature needs to eliminate pores. Large grain growth occurred at high temperature. Spark plasma sintering (SPS) is effective way for densification at low sintering temperature and suppression of grain growth. Furthermore, colloidal processing is effective for controlling the pore size distribution in green compacts before sintering. The green compacts having small residual pores with a narrow size distribution is expected to enhance the densification at low sintering temperature during SPS. In this presentation, this processing was applied to fabrication of transparent alumina, AlN and AlON. Commercially available alumina and AlN powder was used as the starting materials. Slurries with 30 vol% solid were consolidated by slip casting. The green compacts before sintering were further densified by cold isostatic pressing at 392 MPa for 10 min. Final sintering was carried out by SPS. The transparency of alumina prepared by colloidal processing was higher than that densified by SPS from the as-received powder. In the case of AlON, after slip casting of the mixture of alumina and AlN, transparent AlON was obtained by reactive sintering during SPS.

10:10 AM

(ICACC-S14-025-2020) Mesoscale Modeling of Light Transmission Modulation in Ceramics (Invited)

L. Kuna*[1]; J. Mangeri[2]; J. Wollmershauser[3]; E. Gorzkowski[3]; S. Nakhmanson[4]

1. University of Connecticut, Physics, USA
2. Institute of Physics, Czech Academy of Sciences, Dielectrics, USA
3. U.S. Naval Research Laboratory, USA
4. University of Connecticut, Materials Science and Engineering, USA

This presentation introduces a novel modeling approach for simulating properties of polycrystalline ceramics with coupled optical, elastic and dielectric degrees of freedom. The approach is implemented in the Ferret application, developed at UConn and built upon the MOOSE framework, a part of the DoE initiative to create high-quality open-source software for scientific computing. Ferret computational capabilities allow for the determination of the dependency of optical properties on polycrystalline grain orientation, size, and shape, as well as temperature and the action of externally applied electric fields and elastic strains. In particular, we adopt a modification of the wave-retardation theory, developed by Raman and Viswanathan in 1955, for evaluation of optical transmission through a polycrystal, which produces excellent agreement with experimental results obtained for various ceramic materials in both visible and infrared parts of the EM spectrum. Furthermore, our computational approach can also predict the modulation of transmittance under a variety of applied mechanical, electrical and thermal conditions, showing changes from full transparency to opacity in some cases. The results of these investigations highlight a remarkable promise of functional nano- and micro ceramics for a range of advanced engineering applications, including transparent armor, multi-functional IR domes and metamaterials by design.

10:40 AM

(ICACC-S14-026-2020) Doping Effect on the Kinetics and Thermodynamics of Sintering of Yttrium Oxide

K. Nakajima*[1]; R. Castro[1]

1. University of California, Davis, Material Science & Engineering, USA

The sintering behavior of Y_2O_3 has been extensively studied as dense polycrystalline Y_2O_3 yields transparent ceramic with high thermal and chemical stability, suitable for applications such as lasers and refractories. A number of metal cations introduced in a small concentration as dopants have been shown to act as effective sintering aids, allowing production of fully dense Y_2O_3 via less energy intensive processes (i.e. lower temperature and ambient pressure) than undoped Y_2O_3. However, the mechanism of the dopant effect is not sufficiently understood as previous studies have solely focused on its kinetic aspect while the thermodynamic effects of dopants on interfaces of nanoparticles, and therefore, on the driving force of sintering is profound. In this study, undoped and doped Y_2O_3 nanoparticles were synthesized using coprecipitation method and were characterized to investigate the dopant effects on the thermodynamic driving force of sintering in Y_2O_3. The surface and grain boundary energies of undoped and doped samples were derived through direct measurement using differential scanning calorimetry (DSC). The DSC signals were further analyzed to obtain activation energies of sintering which provided important insights into the mechanisms of the sintering process as well as the dopant effect.

11:00 AM

(ICACC-S14-027-2020) Effect of Pressure During Hot-Pressing Transparent Alumina using Platelet-Morphology Powder

A. Schlup*[1]; W. Costakis[1]; R. Trice[2]; J. P. Youngblood[3]

1. Purdue University, Materials Engineering, USA
2. Purdue University, Department of Materials Engineering, USA
3. Purdue University, School of Materials Engineering, USA

Transparent polycrystalline alumina is a promising optical material, particularly in applications that require ballistic protection. However, the rhombohedral crystal structure of alumina limits its transparency due to birefengence effects. One method of reducing birefringence effects is to align the particles along the same crystallographic direction, minimizing the refractive index mismatch. Such alignment can be achieved using platelet-morphology alumina in shear/elongational flow. Sintering of this platelet alumina powder to high relative densities (>99.95%) is necessary to achieve transparency, but the high-aspect ratio of the platelets makes sintering difficult using conventional pressureless sintering methods. Hot-pressing of the platelet alumina powder has been studied at various pressures. An in-line transmission of 65% has been achieved despite a large grain size (>60 micron). Additionally, lower hot-pressing pressures are found to be optimal, with higher pressures resulting in a decrease in density and optical properties. The optical properties of this hot-pressed platelet alumina and how it relates to Rayleigh-Gans-Debye theory will be discussed, as well as the possible mechanisms that are responsible for the lower densities at higher pressures.

11:20 AM

(ICACC-S14-028-2020) Microstructures of Laser-sintered Alumina Body (Invited)

T. Kimura*[1]; S. Suehiro[1]

1. Japan Fine Ceramics Center, Japan

Sintering processes of ceramics usually require high-temperature heat-treatments for long time and consume the most energy in ceramics manufacturing processes. A direct heating process (such as microwave heating, direct resistance heating and laser heating) promises to reduce energy consumption because there is no energy used to heat the insulation walls in a furnace. We have developed a laser heating process for ceramics sintering using Nd:YAG laser, and microstructures of the laser-sintered alumina were investigated in this study. Microstructures of the laser-sintered alumina were strongly affected by the green (or initial) microstructures. When the laser was irradiated to a course pellet consisted of large alumina particles, a porous pellet with a superior bending strength was obtained. On the other hand, a highly packed green pellet results a transparent body. The formation mechanisms of these microstructures in the laser sintering process, including the distribution of heat generated under laser irradiation, will be discussed.

S15: 4th International Symposium on Additive Manufacturing and 3-D Printing Technologies

Direct Writing and Ink Jet Printing I

Room: Coquina Salon B

Session Chair: Paolo Colombo, University of Padova

8:30 AM

(ICACC-S15-028-2020) Rheological controls of slurry for direct-writing of 3D green structures (Invited)

H. Abe*[1]

1. Osaka University, Japan

Direct-ink-writing (DIW) offers the ability to produce three dimensional periodic green structures via a layer-by-layer deposition of filamentary-excluded gel inks from a micronozzle. The gel-inks exhibit a well-controlled viscoelastic response, i.e., they flow through the deposition nozzle and then set immediately to facilitate shape retention of the deposited features. These characteristics have been achieved with careful control of colloidal forces. Here, we present our gel- ink designs and their rheology which can apply to aqueous and non-aqueous slurry systems for DIW.

9:00 AM

(ICACC-S15-029-2020) Additive manufacturing using the direct ink writing technique of ceramic pastes typically used in traditional ceramics industry

E. Ordonez*[1]; H. Colorado[2]; J. M. Gallego[3]

1. Universidad de Antioquia, Mechanical Engineering, Colombia
2. Universidad de Antioquia, Colombia
3. SUMICOL, Colombia

This research presents an additive manufacturing study over a ceramic paste typically used at large scale in the ceramic industry from Colombia. The 3D printing technique used was the direct ink writing (DIW) technique. Different sample formulations were fabricated with different volumetric fractions of solids, between 50% and 56%. Two types of additives (sodium silicate and sodium polyacrylate) were used with concentrations between 0.2% and 0.8%. Samples were sintered after the printing process and subjected to different characterization: dimension stability, scanning electron microscopy, density, compression strength, x-ray diffraction, and rheology. Weibull analysis in the compression tests were conducted over the sintered printed samples.

9:20 AM

(ICACC-S15-030-2020) Direct Ink Writing of hierarchically porous geopolymeric structures for environmental applications

G. Franchin*[1]; R. Botti[1]; K. Goulart De Oliveira[1]; C. Bai[2]; M. L. D'Agostini[1]; G. Zangarini[1]; P. Colombo[1]

1. University of Padova, Industrial Engineering, Italy
2. Harbin Engineering University, College of Material Science and Chemical Engineering, China

Geopolymers are inorganic materials that form long-range, covalently bonded, amorphous networks; they are usually synthesized through the reaction of alumino-silicates in an alkaline medium.

They consolidate at low, even room temperature but can withstand temperatures above 1200°C. Their chemical composition is similar to zeolites, and they also possess intrinsic micro- and mesoporosity; for these reasons, they can be of interest for environmental applications as filters, adsorbers, catalysts and so on. Our group has been exploring Direct Ink Writing as a processing route for hierarchically porous components comprising geopolymers as active material and/or as a matrix for active fillers such as zeolites and activated carbons. Lattices with designed macroporosity above 50%vol were fabricated and characterized in terms of microstructure, mechanical properties, specific surface area and permeability. Specific compositions were developed for selected applications, including biodiesel production catalysis and water filtration from both cationic and anionic pollutants. Their performances will be presented and compared with more conventional materials and processing routes.

9:40 AM

(ICACC-S15-031-2020) Direct Write Additive Manufacturing and Characterization of Battery Electrodes with Engineered Architecture and Porosity

A. Gorven*[1]; A. S. Almansour[2]; A. Salem[4]; M. Singh[3]

1. Boise State University, Mechanical Engineering, USA
2. NASA Glenn Research Center, Mechanical Engineering, USA
3. Ohio Aerospace Institute, USA
4. Washington University, Mechanical Engineering, USA

The development of batteries with high specific power and energy densities will enable the next generation of smart devices, and allow for more efficient implementation of all-electric aircraft and urban air mobility (UAM) technologies. Additive manufacturing technologies can be leveraged to produce engineered 3-dimensional cell structures with increased electrolyte/electrode interfacial area, yielding increased power density and reducing weight through the implementation of ceramic electrodes. Direct-write additive manufacturing (DWAM) technology allows for the deposition of solid-bearing inks with a high degree of dimensional accuracy. Ink rheology was adjusted in order to optimize material characteristics of the final object. Engineered LiFePO4 (LFP) cell structures were manufactured, sintered, and characterized by leveraging the sub-micron accuracy of direct write printing. The sintered electrodes were shown to exhibit favorable porosity and dimensional tolerances. The direct write additive manufacturing process, ink rheology, and sintering properties will also be discussed.

Direct Writing and Ink Jet Printing II

Room: Coquina Salon B
Session Chair: Hiroya Abe, Osaka University

10:20 AM

(ICACC-S15-032-2020) Evaluating the Effect of Rheology and Printing Parameters on Direct Writing

S. Kondapalli*[1]; M. K. Alazzawi[2]; B. Beyoglu[1]; R. A. Haber[1]

1. Rutgers, The State University of New Jersey, Materials Science and Engineering, USA
2. Rutgers University, Materials Science and Engineering, USA

Direct Writing is an extrusion based additive manufacturing process by means of a moving print head and/or stage. Typically, nozzles of varying shapes and sizes can be affixed to the print head to form simple and complex objects. The variety of nozzles can induce different levels of shear stress which can alter the print resolution. In this study, the print resolutions of direct writing as a function of printing parameters were investigated. This can help create highly repeatable components that easily accommodate rapid prototyping. The materials system consisted of silver powder and an organic resin. Two essential aspects studied to improve the print resolutions were the rheology of the resin and the printing parameters. The line spacing was visualized and assessed as a function of nozzle size, and the height and shape of the prints were additionally assessed as functions of drying times. Rheological measurement was also applied to understand the relaxation behavior of the material system as a function of shear rates.

10:40 AM

(ICACC-S15-033-2020) Polymerizable ceramic ink system for thin inkjet-printed dielectric layers

T. Reinheimer*[1]; J. R. Binder[1]

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

Since printed electronics have become an important research area in the last few years, there is a high interest in printed dielectric layers. These layers are amongst others needed for producing capacitors for several applications. To attain high capacities, materials with large permittivities are required, which have to be printed as thinly as possible. Ferroelectric ceramics show high permittivities in general, but are challenging to print. Whenever diluted particulate ink systems are used, typical undesired drying effects can occur. To avoid these drying effects and to achieve thin layers (< 1 µm) a polymerizable ceramic ink system for inkjet printing was developed. This innovative system contains surface modified $Ba_{0.6}Sr_{0.4}TiO_3$ (BST) particles, a crosslinking agent, as well as a thermal radical initiator. The polymerization starts immediately after the ink drop contacts the heated substrate and therefore leads to very homogenous topographies. Since an organic/inorganic composite ink is used, no sintering is needed after printing and thus printing on flexible substrates is possible. Topographies of different printed structures are compared by white light interferometry, the occurring polymerization is confirmed by measurements with an oscillatory rheometer, layer thicknesses are determined by SEM images and the dielectric properties of printed capacitors are analyzed extensively.

11:00 AM

(ICACC-S15-034-2020) Preceramic polymer based additive manufacturing of ceramic composite structures with tailored microstructures

J. W. Kemp*[1]; L. Rueschhoff[2]; B. G. Compton[1]

1. University of Tennessee, Mechanical, Aerospace, and Biomedical Engineering, USA
2. Air Force Research Lab, Materials and Manufacturing Directorate, USA

Additive manufacturing (AM) via direct ink writing (DIW) allows for fabrication of ceramic composites with both complex geometries and high feature resolution. DIW utilizes shear-thinning, viscoelastic inks which can be comprised of preceramic polymer resin and ceramic filler materials. The alignment of high aspect-ratio filler material, a feature of DIW, improves mechanical properties along the print direction. DIW of preceramic polymer resin and high or ultra-high temperature ceramic filler inks present new opportunities in the processing and manufacturing of high-strength, high-temperature ceramic structures. This talk will discuss recent work investigating DIW preceramic polymer resins highly loaded with ceramic powders and fibers. Hexagonal boron nitride (hBN) and zirconium diboride (ZrB_2) powders are shown to impart shear-thinning behavior and enable DIW printing of SiC and Si_3N_4 ceramic precursor resins. This study includes an analysis of rheology and printability of inks, the effects of printing parameters on the polymer-to-ceramic conversion process, and mechanical properties of printed ceramic parts.

11:20 AM

(ICACC-S15-035-2020) 3D printing of sol-gel and melt-derived glass-ceramics for bone regeneration

E. Fiume*[1]; F. Baino[1]; J. Massera[3]; D. Massai[2]; C. Bignardi[2]; E. Verné[1]

1. Politecnico di Torino, Department of Applied Science and Technology, Italy
2. Politecnico di Torino, Department of Mechanical and Aerospace Engineering, Italy
3. Tampere University of Technology, Biomeditech Institute and Faculty of Biomedical Sciences and Engineering, Finland

Scaffolds for bone grafting procedures have to satisfy a series of minimum requirements to provide mechanical support while allowing cell migration and fluid exchange. However, traditional manufacturing techniques suffer from poor repeatability and low control on porosity, which may preclude a safe therapeutic use of the device. In this sense, additive manufacturing technologies represent a powerful platform to easily tailor all these parameters by simply acting on the design and process parameters. In this work, a glass-ceramic based on the system SiO_2-CaO–P_2O_5–Na_2O–MgO-K_2O was successfully synthetized for the first time by sol-gel route and compared to the correspondent melt-derived material. Thermal properties, crystallization and surface area were object of study for comparison. In a second part of the study, both the systems were used as basic materials for the production of highly controlled grid-like 3D scaffolds by robocasting technology. Bioactivity and ion release were assessed by soaking the scaffolds in Simulated Body Fluid (SBF), in both static and dynamic conditions. In the latter case, a customized perfusion system designed for continuous SBF recirculation through the scaffolds was used to mimic physiological conditions. Up to now, results are promising and future studies deserve to be carried out to investigate cellular response and mechanical properties of the developed scaffolds.

Direct Writing and Ink Jet Printing III

Room: Coquina Salon B

Session Chair: Michael Halbig, NASA Glenn Research Center

1:30 PM

(ICACC-S15-036-2020) Sol-Gel Derived Inks for 3d Printed Glass Optics (Invited)

R. J. Dylla-Spears*[1]; K. Sasan[1]; T. Fears[1]; N. Dudukovic[1]; M. Johnson[1]; D. Nguyen[1]; T. Yee[1]; O. Herrera[1]; C. Mah[1]; A. Lange[1]

1. Lawrence Livermore National Laboratory, Optics and Materials Science & Technology, USA

Techniques for three-dimensional (3d) printing of glass have opened the door to novel glass optics with both unconventional structures and tailored compositions. In the direct ink writing (DIW) approach, rheologically tuned silica-containing pastes are first extruded through a nozzle at room temperature and deposited in the geometry of interest, forming low density green bodies. The green bodies are then converted to full density, optically homogeneous glass by a series of heat treatments. The 3d-printed glass components have material and optical properties that rival conventionally prepared optical grade fused silica. However, to fully realize the potential of this technique for preparing novel optics requires the development of a library of ink formulations that lead to glasses with different properties. Several sol-gel approaches to ink preparation leading to refractive index control in 3d printed silicate glasses will be presented. In addition, efforts to blend and thermally process these inks into gradient composition optics will be described. This work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344 within the LDRD program 16-SI-003 and 19-ERD-020. LLNL-ABS-765937

2:00 PM

(ICACC-S15-037-2020) Quantifying the Link between Rheology and Printability for Ceramic On-Demand Extrusion

A. J. Martin*[1]; J. Watts[1]; G. Hilmas[1]; M. C. Leu[2]; T. Huang[3]

1. Missouri University of Science & Technology, Materials Science and Engineering, USA
2. Missouri University of Science & Technology, Mechanical & Aerospace Engineering, USA
3. Kansas City National Security Campus, USA

This study will continue research to link feedstock rheology to the 'printability' of ceramic components fabricated by Ceramic On-Demand Extrusion (CODE), a direct-write extrusion process which utilizes a rising oil bath to prevent excessive and non-uniform drying. Relevant background work assessing extrudability and printability will be discussed. Next, a comprehensive approach to couple rheology to object printing parameters will be described, and a wide range of zirconia pastes will be explored to establish process-property relationships. Paste microstructure will also be explored. From these investigations the constraints on ideal paste design will be discussed.

2:20 PM

(ICACC-S15-039-2020) Robocasting of reaction bonded silicon carbide structures

L. Wahl*[1]; N. Travitzky[1]

1. Friedrich-Alexander-University Erlangen-Nürnberg, Material Science and Engineering, Germany

Additive manufacturing of ceramics shows great potential to overcome the shape limitations of traditional fabrication methods due to the layerwise deposition of material. In this work, a novel shaping method for the manufacturing of reaction bonded silicon carbide (RBSC) structures was investigated. RBSC is attractive for high temperatures and extreme condition applications, such as turbines and bearings, due to its outstanding thermal and mechanical properties. To produce RBSC, a highly filled paste consisting of silicon carbide and carbon powder was developed and printed by robocasting technology using nozzles with diameter of 0.5 and 1.5 mm. To enable printing, the paste has to show suitable rheology, which means the presence of a yield point and a shear-thinning behavior. After heat treatment, the samples were further processed using the liquid silicon infiltration technique to obtain dense near-net shape RBSC. Different structures such as lattices, hollow cylinders, bending bars and gyroids were printed to show the variability of robocasting. The mechanical and physical properties of the robocasted samples were measured and are comparable with those of traditionally fabricated RBSC. These results open the field for a wide variety of materials, which can be processed in similar manner.

Fused Deposition Modeling

Room: Coquina Salon B

Session Chair: Rebecca Dylla-Spears, Lawrence Livermore National Laboratory

3:20 PM

(ICACC-S15-040-2020) Ceramic matrix composites fabricated by Fused Filament Fabrication (FFF)

H. Klemm*[1]; J. Abel[1]; A. Michaelis[2]; M. Singh[3]

1. FhG IKTS Dresden, Germany
2. Fraunhofer IKTS, Germany
3. Ohio Aerospace Institute, USA

Ceramic matrix composites with SiC short fibers have been fabricated by Additive Manufacturing (AM) using a thermoplastic approach. By means of Fused Filament Fabrication (FFF) or Fused Deposition Modelling (FDM) a ceramic part is shaped layer by layer using a thermoplastic filament which is extruded through a heated nozzle.

According to the degrees of freedom for movement of the printing head (and printing bed) green bodies with complex geometries can be fabricated. In this study a ceramic feedstock with SiC fibers (Hi Nicalon, UBE SA3), SiC powder with sintering additives and a thermoplastic binder system was used. Tailored filaments with approximately 1.8 mm in diameter were developed, fabricated by extrusion and printed by FFF to achieve CMC with short and long SiC-fibers. After binder burnout ceramic matrix composites were fabricated by polymer infiltration and pyrolysis (PIP) and liquid silicon infiltration (LSI). Various composites with unidirectional fiber orientation and different geometries were obtained. Finally, the materials fabricated were characterized regarding their microstructure and mechanical properties.

3:40 PM

(ICACC-S15-041-2020) Multi-Material Additive Manufacturing of High Temperature Polymers for Electric Aircraft Applications

H. Leonard*[1]; A. Salem[2]; M. C. Halbig[3]; M. Singh[4]

1. Rochester Institute of Technology, Mechanical Engineering, USA
2. University of Washington, USA
3. NASA Glenn Research Center, USA
4. Ohio Aerospace Institute, USA

The future of aviation lies in the use of electric aircraft with lightweight, efficient motors and energy storage systems. While motors currently reach into the 95% efficiency range, motors and batteries have thermal management issues coupled with the extra weight of heavy iron plates and safety systems. One can mitigate these problems by replacing metals with lightweight yet strong polymers that can be 3D printed to allow new shapes and designs that help move heat away from sources by active or passive cooling systems. New shapes from additive manufacturing will allow the use of water cooling or forced air cooling of motor stators and battery packs helping to keep systems lighter and running more efficiently when high up in the air. Advanced polymers like Ultem and TCPoly can be used to create strong, lightweight and efficient structures to improve the performance. In this paper, high temperature polymer materials with good mechanical and thermal properties will be discussed. Few high temperature filaments have also been printed together to create a multi material 'zebra' part with the characteristics of both materials in one part. A few different battery cooler designs have also been fabricated to characterize their feasibility as a battery cooling option for aerospace applications.

4:00 PM

(ICACC-S15-042-2020) Additive Manufacturing of Multi-Material Structures for Enhanced Multifunctional Performance

A. Salem*[1]; H. Leonard[2]; M. C. Halbig[3]; M. Singh[4]

1. Washington University in St. Louis, USA
2. Rochester Institute of Technology, Mechanical Engineering, USA
3. NASA Glenn Research Center, USA
4. Ohio Aerospace Institute, USA

Additive Manufacturing (AM) process is most beneficial for high-complexity and low production volume parts. AM has enabled the fabrication of incredibly complex geometries, as seen in generative design technologies. The majority of additively manufactured parts are made from a single type of material. Dual-extrusion printers have become increasingly popular, but the use of multi-material printing is usually only for aesthetic differences, not functional ones. Significant functional advantages can be achieved by tailoring a part's material composition to a specific application. Part composition can be locally defined in accordance with an application requirements, enabling even further benefits. In this research, we explore the benefits of multi-material, multifunctional, parts using a Hyrel Hydra Fused Deposition Modeling (FDM) printer. The materials being explored were ULTEM 9085, ULTEM with chopped carbon fiber, and Polyphenylsulfone (PPSU). Analysis was conducted through tensile testing, microscopy, thermogravimetric analysis, and differential scanning calorimetry. It was found that one can engineer the thermomechanical properties by changing the ratio and deposition order of multi-material parts. In this presentation, effect of layer thickness and composition on the microstructure and thermomechanical properties are discussed.

4:20 PM

(ICACC-S15-043-2020) Tribological Behavior of 3D Printed Multilayered Composites

S. Abu Aldam*[1]; M. C. Halbig[2]; M. Singh[3]; S. Gupta[1]

1. University of North Dakota, Mechanical Engineering, USA
2. NASA Glenn Research Center, USA
3. Ohio Aerospace Institute, USA

Fused Deposition Modeling (FDM) has emerged as important tool for fabricating 3D printed composites. In order to deploy and design novel materials by using FDM, it is critical to understand and explore the effect of microstructure on the mechanical and tribological behavior of 3D printed composites. In this presentation, we will report the design of 3D printed composites in different configurations. More particularly, ABS (Acrylonitrile butadiene styrene) and ABS reinforced with Carbon Fibers (ABS-CF) were selected as candidate materials. By using these materials, different composites with multilayered texture were 3D printed, for example, (a) bi-layered samples, and (b) alternating layers of ABS and ABS-CF. As a fundamental study, the effect of different orientations on the mechanical and tribological behavior of these composites will be reported.

S16: Geopolymers, Inorganic Polymers and Sustainable Materials

Synthesis and Processing

Room: Tomoka C

Session Chair: Waltraud Kriven, University of Illinois at Urbana-Champaign

1:30 PM

(ICACC-S16-001-2020) Synthesis of High-Surface Area Organo-Modified Aluminosilicates from Geopolymerization (Invited)

D. Seo*[1]; W. Zhang[1]; S. Chen[1]

1. Arizona State University, School of Molecular Sciences, USA

Organic/Inorganic hybrid materials are one of the emerging materials due to their potential multifunctionalities. However, possibility of such materials in geopolymer research has not been fully realized. Given the innate scalability of geopolymer production, the advent of such new geopolymer material type has a huge implication in novel large-scale applications of geopolymer. In this presentation, we report our newly syntheses of organo-modified aluminosilicate nanomaterials and conjecture their formation principles in terms of geopolymer chemistry in relation to sol-gel synthesis. We will also discuss the key features of the synthetic conditions and the relationship between the synthetic parameters (the concentrations of the precursors, reaction time and temperatures) and the morphologies of the geopolymeric nanomaterials in terms of precursor types/compositions, surface areas and porosity.

2:00 PM

(ICACC-S16-002-2020) Thermal behavior of several clay mixtures: Geopolymer synthesis (Invited)

S. Rossignol*[1]

1. IRCER, France

Over the last decades, geopolymers have gained tremendous interest as promising new binders, environmentally friendly and with good working properties. Metakaolin is one of the most used aluminosilicate precursor due to its high purity and reactivity. Nevertheless, research of low-cost and more available materials has encouraged many investigators to turn to the use of common clays and industrial

co-products. In this topic, the objective of this work is to evaluate the reactivity of various aluminosilicate sources at different temperatures. To achieve the objective the different raw materials such as various clays and its mix calcined at different temperatures were used. Moreover, the different type of mixtures based on kaolin and additive (calcite, dolomite…) were prepared. Structural characterization by XRD, NMR, and FTIR spectroscopies before and after calcination of the raw materials were carried out. The characterization of the different mixtures evidences structural differences depending on the type of the used raw material, additive and the calcination temperature. The results show the relationship between thermal behaviors of clays and their chemical and mineralogical composition. Moreover, the feasibility study evidences the suitability of the different sources after heat treatment to produce geopolymer materials with different mechanical properties.

2:30 PM

(ICACC-S16-003-2020) Ferrosilicates formation during the geopolymerization of Natural Fe-rich aluminosilicate precursors (Invited)

E. Kamseu*[1]; R. C. Kaze[4]; J. N. Nouping Fekoua[1]; U. C. Melo[4]; S. Rossignol[2]; C. Leonelli[3]

1. MIPROMALO, Research, Cameroon
2. Laboratoire SPCTS, France
3. University of Modena and Reggio Emilia, Department of Engineering "Enzo Ferrari", Italy
4. University of Yaoundé I, Laboratory of Applied Inorganic Chemistry, Cameroon

The progressive addition of the reactive silica from rice husk ash (10 to 50 wt%) changes the phases evolution and the microstructure of the laterites based geopolymer composites. It was found that the presence of soluble silica enhanced the formation of Si–(A)-OH–Fe bonds and changed considerably the FTIR and XRD patterns of the products: polysialates, ferrosialates and ferrisilicates were present into the matrices. The solid/liquid ratio and the curing cycle influenced the pore network, the bulk density, the total pore areas, and the final microstructure. The relative low viscous paste that is maintained at ambient temperature for 1-2 hours before curing at 90 °C gave better cohesion and low porosity resulting to the high concentration of strongest (Fe–O–Si) bonds compared to pastes cured directly after preparation or those with high viscosity. While polysialates and ferrosilicates are hindered in the case of quite treatment at 80°C due to the rapid evaporation of water, polysialates, ferrosialates and polynuclear ferrisilicates complexes are formed at room temperature and polynuclear complexes can growth to more crystalline phases with the increase of the temperature. Ferrisilicates integrated the interlayers of the matrix improving the geopolymerization with significant modification of the microstructure.

Mechanical Properties

Room: Tomoka C

Session Chair: Patrick Keane, University of South Australia

3:20 PM

(ICACC-S16-004-2020) Effect of porosity on the mechanical response of geopolymer composites (Invited)

A. Akono*[1]; W. M. Kriven[2]; S. Koric[2]

1. Northwestern University, Civil and Environmental Engineering, USA
2. University of Illinois at Urbana-Champaign, USA

Geopolymers are X-ray amorphous materials that are synthesized by mixing a source of aluminosilicates with an alkaline solution. Geopolymer hybrids are relevant in many applications such as membranes for water filtration, adsorbents for carbon capture, components for sound insulation systems, or thermal barrier coatings in thermal insulation systems. We study the effect of air voids on the mechanical properties. Porosity contributes to a low density, high permeability, and high selectivity. However, the role of porosity on the strength constants is not known. We employ a hybrid theoretical and experimental approach. We formulate a multiscale mechanistic model that connects the homogenized behavior to the chemistry, the microstructure, and the composition. We validate our analytical model against three-point bending tests conducted on metakaolin-based geopolymer composites. In parallel, we synthesize metakaolin-based geopolymer binders while varying the alkali activator solution, the processing, and the curing temperature. We probe the elastic and plastic behavior using indentation tests. Nanopores contribute to improved strength behavior through local rearrangements. In contrast, micropores are detrimental to strength improvement. The distribution of nanopores is controlled by the chemistry solely, whereas the distribution of micropores is controlled primarily by the processing, and curing temperature.

3:50 PM

(ICACC-S16-005-2020) Fracture Behavior of Metakaolin-based Geopolymers Reinforced with Carbon Nanofibers

A. Akono*[1]

1. Northwestern University, Civil and Environmental Engineering, USA

We investigate the fracture response of metakaolin geopolymer reinforced with carbon nanofibers. We synthesize the geopolymers by mixing carbon nanofibers, amorphous silica, an alkaline solution, and a synthetic source of aluminosilicate. We measure the fracture energy of the resulting hybrids using cutting edge microscopic fracture tests. In our tests, a sphero-conical diamond pushes across the surface of the specimen under a prescribed vertical force. We analyze the recorded penetration depth and horizontal force through the lenses of nonlinear fracture mechanics and extract the fracture parameters. After testing, we apply scanning electron microscopy to observe the fracture surfaces. We observe that the presence of carbon nanofibers contributes to an improvement in fracture behavior. We explore the influence of curing temperature, processing, and mix design on the final fracture parameters. This study is important to pave the way for novel enhanced-performance and multifunctional structural materials.

4:10 PM

(ICACC-S16-006-2020) Basalt Minirod-Reinforced Geopolymer Composites

V. Chadha*[1]; W. M. Kriven[2]

1. University of Illinois at Urbana-Champaign, Materials Sciences, USA
2. University of Illinois at Urbana-Champaign, USA

Basalt is the most common volcanic rock-type on earth. The sheer abundance of basalt has been a reason it is extensively being researched as an alternative reinforcement. Composite basalt fiber minirods, obtained from Galen LLC, Florida, were fully coated with adherent sand and had a diameter of 2.2 mm by an approximate length of 20 mm. They were designed to prevent cracking and chipping at the edges of concrete joints and pre-fabricated structures. Sodium geopolymer of composition Na2O.Al2O3.4SiO2.11H2O was produced from fumed silica, deionized water, sodium hydroxide, (i.e. water glass) and BASF Metamax® metakaolin. The geopolymer matrix was fabricated in an IKA® high shear mixer (Model RW20DZM, IKA, Germany) then vibrated on an FMC Syntron vibrating table (FMC Technologies, Houston, Texas) to remove trapped air bubbles introduced during the high shear mixing. With the optimized addition of 50 micron size chamotte powder to increase the viscosity of the matrix to support better, the distribution of minirods, the weight percentage of minirods was increased to ≥ 10 wt %. The basalt minirods and chamotte powder were then dispersed in the NaGP using a low torque mixer and the samples were allowed to set under applied pressure at ambient temperature for 1 day, followed by 1 day at 50C to complete the reaction. Weibull summary statistics of material properties were determined by mechanical characterization testing.

4:30 PM

(ICACC-S16-007-2020) High strength geopolymers using feldspathic solid solutions: Mechanical properties and Microstructure

A. Nana[3]; R. C. Kaze[4]; H. Tchakoute Kouamo[4]; M. C. Bignozzi[5]; E. Kamseu*[1]; C. Leonelli[2]

1. MIPROMALO, Research, Cameroon
2. University of Modena and Reggio Emilia, Department of Engineering "Enzo Ferrari", Italy
3. University of Dschang, Research Unit of Noxious Chemistry and Environmental Engineering, Cameroon
4. University of Yaoundé I, Laboratory of Applied Inorganic Chemistry, Cameroon
5. University of Bologna, DICAM, Italy

Amorphous fraction, grains defects and the incongruent dissolution of solid solutions (pegmatite, and granite) were exploited to design high strength geopolymer composites with crystalline content in the range of ~ 70-85%. The solid solutions were altered by 15, 20, 25 and 30% of metakaolin and activated with an alkaline solution. Three-point flexural strength, Hardness Modulus, Impact Testing and microstructure are combined to the mercury intrusion porosimetry and water absorption to assess the potential of the new matrices for engineering applications. The amorphous/crystalline ratio of the solid precursors were used to understand the role of dissolved and undissolved fraction into the strength development of geopolymer composites. It was concluded that high strength

4:50 PM

(ICACC-S16-008-2020) Mechanical Properties of Flax and Hemp Felt Geopolymer Composites

P. F. Keane*[1]; W. M. Kriven[1]

1. University of Illinois at Urbana-Champaign, USA

Two types of felt samples were received from Fibres Recherche Development® in France, via Mr. Ralph Davidovits of www.geopolymer.org. Continuous, non-woven mats of flax and hemp of dimensions 3 m^2 wide and weighing 300 g/m^2 were received. The flax mat was 100% flax while the hemp was composed of 80% hemp and 20% polyester threads in the form of very fine, white threads. The organic polyester was not expected to react with the geopolymer. The hemp and fiber felt were dispersed in potassium metakaolin-based, geopolymer reinforced with 50 mm size, mullite particulate reinforcements in the form of chamotte. The low viscosity of the KGP liquid was able to penetrate the felt upon the application of some pressure. Upon curing at ambient temperatures, for each type of reinforcement, ten bend bars of dimensions 1′″ x 1″ x 6″ were broken in 4-point flexure, and the data was analyzed by Weibull statistics. The microstructure and microchemistry of the fracture surfaces were examined by SEM and EDS.

S17: Advanced Ceramic Materials and Processing for Photonics and Energy

Multifunctional Materials II

Room: Tomoka C

Session Chairs: Elisa Moretti, Ca' Foscari University of Venice; Clara Santato, Ecole Polytechnque de Montreal

8:30 AM

(ICACC-S17-024-2020) Towards the creation of catalytic structures using liquid metal processes (Invited)

K. Kalantar-zadeh*[1]

1. UNSW, School of Chemical Engineering, Australia

Exploring the concept of liquid phase processing of low melting point alloys can provide new routes for the creation of catalytic micro and nanostructures that can be readily engineered. Here we show that it is possible to produce particles of liquid metals, based on examples of Field's metal (eutectic alloy of bismuth, tin, and indium) and eutectic alloy of bismuth and tin, in a one-step ultrasonication procedure. The synthesis takes place just above the melting point of these eutectic alloys, and results in the formation of low dimensional particles after solidification. It is demonstrated that sonication produces near spherical core-shell metal-metal oxide heterostructures. Classical alloy related defects, in the form of grain boundaries and edge locations appear after solidification. We show the effect of a blowing aganet to produce catalytic foams from these nanopartcles. We also demonstrate that Ag can be incorporated by either using direct alloying or an Ag precursor during the process, leading to the formation of an Ag metal enriched core with tailored composition. Interestingly, the modification of particles with Ag gives specific selectivity for the degradation of two commonly encountered azo dyes. The one-step low-temperature synthesis and solidification procedure and also the creation of catalytic foams can be readily extended for designing catalysts with desired specificity and selectivity.

9:00 AM

(ICACC-S17-025-2020) $Zn_{0.35}Co_{0.65}O$: A Stable and Highly Active Oxygen Evolution Catalyst Formed by Zinc Leaching and Tetrahedral Coordinated Cobalt in Wurtzite Structure (Invited)

N. Pinna*[1]

1. Humboldt-Universität zu Berlin, Department of Chemistry, Germany

To arrive to sustainable hydrogen-based energy solutions, the understanding of water-splitting catalysts plays the most crucial role. Herein, we combined state-of-the-art hypotheses on electrocatalytic active metal sites towards the oxygen evolution reaction (OER) to develop a highly efficient catalyst based on earth-abundant cobalt and zinc oxides. The precursor catalyst $Zn_{0.35}Co_{0.65}O$ was synthesized via a fast microwave-assisted approach at low temperatures. Subsequent, it transformed in situ from the Wurtzite structure to the layered γ-Co(O)OH, while most of its zinc leaches out. This material shows outstanding catalytic performance and stability towards the OER in 1 M KOH (overpotential at 10 mA cm^{-2} $\eta_{initial}$ = 306 mV, $\eta_{98\ h}$ = 318 mV). By comparing the electrochemical results and ex situ analyses to today's literature, we were able to identify clear structure-activity correlations. Our findings suggest that coordinately unsaturated cobalt octahedra on the surface are indeed the active centers for the OER.

9:30 AM

(ICACC-S17-026-2020) Multi-Functional Lanthanide-Doped Nanoparticles and their Applications (Invited)

F. Vetrone*[1]

1. Institut National de la Recherche Scientifique, Université du Québec, Centre Énergie, Matériaux et Télécommunications, Canada

The ability to stimulate luminescent inorganic nanoparticles with near-infrared (NIR) light has made possible their use in a plethora of biological and medical 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. The use of NIR light for excitation mitigates some of the drawbacks associated with high-energy light (UV or blue) excitation, for example, little to no background autofluorescence from the specimen under investigation as well as no incurred photodamage. Moreover, one of the biggest limitations is of course, that of penetration. As such, NIR light can penetrate tissues much better than high-energy light especially when these wavelengths lie within the three so-called biological windows. Thus, significant strides have been made in the synthesis of inorganic nanomaterials whose excitation as well as emission bands lie within one of these three optically transparent biological windows. Here, we present the synthesis of various NIR excited (and emitting) inorganic core/shell (and hybrid) nanostructures and demonstrate their potential use in nanomedicine. Furthermore, we will show how such nanoparticles can be used as building blocks towards developing multifunctional nanoplatforms for simultaneous detection and therapy of disease.

Multifunctional Materials III

Room: Tomoka C

Session Chair: Nicola Pinna, Humboldt-Universität zu Berlin

10:20 AM

(ICACC-S17-027-2020) Towards ion-gated phototransistors as photocatalytic sensors (Invited)

C. Santato*[1]

1. Ecole Polytechnque de Montreal, Canada

Ion-gated transistors make use of ionic gating media to modulate the current in the transistor channel. They operate at low voltages (ca 0.5) such that they can be easily integrated with a number of chemo- and bio-sensing platforms. We report on ion-gated tungsten oxide phototransistors operated in different ionic media, fabricated both on rigid and flexible substrates. Ion-gated tungsten oxide phototransistors operating in aqueous media can be used as photocatalytic sensors in portable applications.

10:50 AM

(ICACC-S17-028-2020) Flower-like Ce-Ti oxide systems for the CO preferential oxidation in H_2-rich stream under simulated solar light irradiation (Invited)

E. Moretti*[1]; A. Infantes-Molina[2]; E. Rodriguez-Castellon[2]; A. Talon[1]; A. Vomiero[3]

1. Ca' Foscari University of Venice, Department of Molecular Sciences and Nanosystems, Italy
2. University of Malaga, Department of Inorganic Chemistry, Crystallography and Mineralogy, Spain
3. Lulea University of Technology, Engineering Sciences & Mathematics, Sweden

The investigation of CeO_2-based materials is a research hotspot for environment- and energy-related applications. In particular, tuning the morphological features of a catalyst has emerged as an important strategy to improve catalytic activity and there has been extensive research to develop highly active ceria-based systems rationally designed with a controlled morphology at the nano/microscale. The present work aims to investigate the photocatalytic behavior of Au nanoparticles (1.0 wt% nominal loading) supported on CeO_2-TiO_2 matrices with a flower-like morphology in the CO preferential oxidation (photo CO-PROX) in excess of H_2, assessing not only the role of each component in the system and on the catalytic response, but also how a peculiar morphology can affect the photocatalysis. CeO_2 samples containing different TiO_2 loadings were synthesized by a surfactant-free and environmentally friendly slow co-precipitation method. As shown by SEM microscopy, the samples appeared organized in a hierarchical structure comparable to a microscale size flower. HRTEM revealed the presence of 2-4 nm size Au NPs, homogenously distributed on the support surface. The Au/CeO_2-TiO_2 systems showed a morphology dependent behavior in the photo CO-PROX under simulated solar light irradiation at R.T., resulting much more active than samples with a non-organized structure.

11:20 AM

(ICACC-S17-029-2020) Silk-titanate nanosheets composites for biophotonic and plasmonic devices (Invited)

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2. Tufts University, USA
3. IIT Genova, Italy

In the last decade silk fibroin, the protein extracted from the silk fibers, emerged as an attractive material for biophotonic applications due to its biocompatibility combined with unique mechanical and optical properties. A strategy to enhance the performance of silk as optical material and to introduce specific functionality, is to combine silk with inorganic nanoparticles (NPs). In this paper we combine silk with titanate nanosheets (TNTs), a 2D precursor of TiO_2, in order to increase the refractive index of silk while preserving all its specific properties. The structural and functional characterizations of the Silk-TNT composites were performed to correlate the material structure with its properties. The different fabrication techniques already developed for pure silk were applied on the high refractive index nanocomposite. Thanks to the high refractive index achievable by using TNTs, a multilayer structure was fabricated, that exhibits an interference peak in the transmittance spectrum centered at 400 nm, whose position responds to changes in the relative humidity of the environment. The stimuli-responsive properties of the film were characterized and a simple optical model for the sensing mechanism was proposed. Au NPs were also introduced in order to have plasmonic functionality for laser plasmon resonance induced heating.

11:50 AM

(ICACC-S17-030-2020) Complex nano-structured sponges for sensing- and energy-applications (Invited)

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There is a strong demand for tailored materials for sensors, catalysts, solar-cells and energy storage for renewable energy areas. Typical features of these materials are; a large interface to the surrounding liquid electrolyte, reaction liquid, or gas phase, surface modification for e.g. catalysis, band structure and corrosion control. Typically, a high conductivity is required to get generated electrons out to the surface for electro-catalysts, and in for sensors and solar cells, which implies sponges of high crystalline quality and good connectivity though the structure. Here we present salt-based synthesis routes yielding oxide sponges of various contents at temperatures from 200 ºC, 3 min. Systems such as doped and non-doped ZnO, MgO and transition metal oxides are discussed in detail from the solution to the final products, along with some properties. The sponges obtained at 200 ºC typically consisted of highly porous structures built from well-connected 8-10 nm crystallites. The sponges show remarkable resistance to sintering their overall porous structure at elevated temperatures, but from ca 500ºC, a reproducible grain growth took place within the sponge structures, allowing for tuning of crystal sizes. The processes and products were studied with; TG/DT/DSC, XRD, SEM-EDS, TEM-EDS/ED, XPS, IR- and Raman spectroscopy.

Poster Session B

Room: Ocean Center Arena

5:00 PM

(ICACC-S1-P067-2020) Development of Artificial Pedra Using Granite and Epoxy

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The extracting process of ornamental stone generate residues, usually blocks of differents sizes that can not be used. The Brazilian industry has high potential and many reserves, however the waste of stones causes environmental impact, because it do not have the proper elimination causing a worldwide problem. This research aims to evaluate the production of artificial stone using 85 wt% of white granite agglutinated by 15 wt% of epoxy resin to be used in the civil construction sector. The plates were produced with the use of vibration, vacuum and compression. The residues were sieved and classified in three granulometry: coarse (from 2 - 0,42 mm), medium (from 0,42 - 0,075 mm), and fine (grains inferior to 0,075 mm).

The test for determination of the highest dry bulk density mixture was determinated to find the best composition to produce the plates. To characterize the artificial plates was made water absorption, density and apparent porosity, and bending tests on three points to mechanical characterization. Also, the resistance chemical was performed. The artificial stone presented values of phisycal proprieties considered adequate when compared to other artificial stones, the material showed good resistance to bending, exceeding recommended values for coating materials, it was resistant to chemical attack.

(ICACC-S1-P068-2020) Electrical properties of 8YSZ – ScCeSZ composite

T. G. Fujimoto*[1]; E. N. Muccillo[1]

1. IPEN, Brazil

Ceramics Composites Materials has been widely studied in order to enhance the properties of the individual components when two or more materials are combined. Yttrium stabilized zirconia (8YSZ) has been applied as a solid electrolyte in solid oxide fuel cells (SOFCs) due to good thermal and chemical stability, mainly its high ionic conductivity. However, 8YSZ works at high temperature (800-1000 °C); there is a great interest in to reduce this temperature aiming to optimizing the devices operation. Another candidate for application in SOFCs with higher ionic conductivity at intermediate operating temperature (600-800 °C) is Zirconia-scandia-ceria (ScCeSZ). At high temperature the ions Ce^{4+} present in ScCeSZ can reduce to Ce^{3+} after a long time of cells operation. In this work, differents amounts of ScCeSZ (1, 2.5, 5, 7.5, 10 wt.%) were add to 8YSZ to study the influence of ScCeSZ in 8YSZ conductivity and the ScCeSZ stability. The samples were prepared by conventional mixed oxide route and sintered at 1450 °C for 4 h. The density, microstructure and ionic conductivity of the sintered samples were investigated. The relative densities are higher than 95wt.%. X-ray diffraction patterns exhibit the characteristic cubic structure of 8YSZ. Was obtained that composite containing 5wt.% of ScCeSZ presents higher values of ionic conductivity if compared with 8YSZ.

(ICACC-S1-P069-2020) ASTM International Standards for Properties & Performance of Advanced Ceramics – High-Quality, Technical Rigor for Academe, Government and Industry

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2. NASA Glenn Research Center, Materials and Structures, USA
3. Gateway Materials Technology, USA
4. American Dental Association Foundation, Paffenbarger Research Center, USA
5. Corning Incorporated, Consultant, USA

Mechanical and physical properties/performance of brittle bodies (e.g., advanced ceramics and glasses) are challenging to measure accurately and precisely unless the proper techniques are used. Now entering a fourth decade of effort, ASTM Committee C28 on Advanced Ceramics, has developed many full-consensus standards (e.g., test methods, practices, guides, terminology) to measure various properties and performance of monolithic and composite ceramics as well as coatings and consitutents that may apply to some glasses. These standards provide big and little picture details for determining mechanical, physical, and thermal properties and performance, as well as characteristics for processing, thereby providing accurate, reliable, repeatable and complete data. Users, producers, researchers, designers, and academicians who are involved in ASTM Committee C28 write, update, and validate through round robin test programmes the over 50 standards under the jurisdiction of the Committee since its inception in 1986. This poster includes a pictogram of Committee C28 standards and how to obtain them as either individual or collection of standards. Also included is a listing of other related ASTM committees. Finally, some examples of the tangible benefits of standards for advanced ceramics demonstrating their practical application are provided.

(ICACC-S1-P070-2020) Mechanical Properties of Spark Plasma Sintered B_4C

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The proposed research work is on the assembly of the mechanical behavior of boron carbide ceramics prepared by Spark Plasma Sintering as well as its characterization of thermal and electrical behavior. After sintering of almost dense B_4C, its crystal structure and vibrational properties were characterized by XRD and micro-Raman spectroscopy. Such properties as Young's and shear moduli, flexure and biaxial strength, fracture toughness, hardness, along with thermal expansion and electrical conductivity were measured. Impulse Excitation technique, Resonant Ultrasound spectroscopy, 4 point bending, ring-on-ring testing, Single Edge V Notch Beam bending, Vickers hardness and nanoindentation were used for study of mechanical behavior of B_4C. A high temperature dilatometry was used to measure thermal expansion and 4 point probe technique was used to measure the electrical conductivity. In addition, the Finite Elements Analysis has been used to demonstrate the stresses appeared during biaxial ring-on-ring loading along the surface of the B_4C samples. The performed research contributes to the collection of the properties of B_4C ceramics, an important material in many industrial and military applications.

(ICACC-S1-P071-2020) Metal matrix composite for electronic packaging

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Electronic packaging of level 1 requires low value of coefficient of thermal expansion and high thermal conductivity. The properties like thermal conductivity (TC) and coefficient of thermal expansion (CTE) of metal matrix composites (MMC) can be optimized effectively through adjusting the type or the volume fraction of reinforcement. Metals and ceramics cannot be used for electronic packaging because of high CTE and low TC respectively. In this study Barium strontium titanate (BST) ceramic powder is synthesized by citric-acid gel method and it is taken for reinforcement into copper metal matrix because it shows negative coefficient of thermal expansion during the phase transformation from tetragonal to cubic phase.

(ICACC-S1-P072-2020) Electric potential change of glasses by polishing with thermally oxide silicon

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Chemical mechanical polishing (CMP) realizes ultra-smooth surface of materials that is indispensable to optical devices as well as semiconductor devices. Cerium oxide (ceria) shows low mechanical strength but distinguished polishing properties as compared to zirconia and alumina abrasives. Such high properties would be derived from some chemical reactions that are so-called CMP. Previously we proposed chemically polishing mechanism based on electrochemical charge transfer. Charge transfer on the surface of both ceria abrasives and glasses would lead to form hydration layer on the glasses. Polishing properties would much depend on formation rate of hydration layers on glasses. We thus investigate the effects of charge transfer in the vicinity of glasses on formation of hydration layer by measuring electric properties of glasses using thermally oxide silicon as abrasives. Electric potential change between silicon dioxide obtained by oxidizing silicon wafer thermally and water solution during polishing. The potential was decreased by inducing shear stress and it was back to initial value after the rotation was stopped. The rate of potential change was altered by charge carriers of silicon wafers. Charge transfer rate of abrasives would much depend on hydration layer formation on glasses.

(ICACC-S1-P073-2020) Characterization of Properties of Ceramic Mass for Production of Pressed and Burned Blocks for Structural Masonry

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3. State University of North Fluminense, Civil Engineering, Brazil
4. State University of the North Fluminense, Advanced Materials Laboratory, Brazil

This paper deals with the evaluation of the potential of ceramic soil mass of Miracema/RJ, Brazil, for the production of pressed ceramic blocks, based on the physical, chemical, mineralogical and thermal properties of the soil. The physical analysis indicated that the ceramic mass presents 35.7% of clay and density of 2.59 g/cm^3. The chemical analysis was done using dispersive energy equipment, presenting high levels of silicon dioxide (SiO_2) and aluminum oxide (Al_2O_3), these results indicate that the soil has refractory properties. The mineral analysis, performed by means of an X-ray diffractometer, showed the presence of Caulinite, Quartz, Mica Muscovite and Gibsta, and the first showed higher levels of indication of concentration, plastic characteristic of the sample tested. The ATD and TG curves in the temperature at the firing temperature of most ceramics of the Campos dos Goytacazes / RJ Ceramics Pole, RJ, Brazil, 889 °C, do not identify the presence of thermal transformation peaks. It was concluded that the analyzed ceramic mass presents viability of use in the production of ceramic blocks.

(ICACC-S1-P074-2020) Experimental Analysis and Numerical Simulation of Mechanical Behavior In Breaking Pressed and Burned Blocks of Red Ceramic

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The objective of the present work was to study the mechanical behavior in the rupture of non-grouted pressed and burned ceramic blocks (BPQ) with ungrouted joints through the mechanical characterization tests of isolated blocks and prisms with the proposition of a simulation model in the study of BPQ wall behavior. The properties evaluated were compressive strength, modulus of elasticity, the block and prism Poisson's ratio, as well as the definition of the rupture mode and the prism / block efficiency. Applying the methodology of the Brazilian standards, the characteristic compressive strength of the blocks at 3.62 N/mm^2 was determined, and the longitudinal, transverse deformation modules and Poisson's ratio for the BPQ element at 2,801 N/mm^2, 1,185 N/mm^2 and 0.182, respectively. The prisms showed efficiency in the range of 0.45 and 0.55. The rupture mode of the BPQ was fragile, with a tendency to block failure. The proposed model for the numerical analysis was the micromodeling simulation using Solidworks 16.0 software. Although in the modeling a linear behavior criterion was adopted for the blocks, there is an adequacy of the model, with differences around 10% only in the tests. The results confirm the quality of the BPQ, indicating its applicability for the execution of small works.

(ICACC-S1-P075-2020) Improve Sensitivity of Electrical Resistance In High Temperature CMC Using Carbon Monofilament SiC Fibers

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1. University of Akron, Mechanical Engineering Dept., USA

Due to their low density and high thermal capability, continuous fiber reinforced ceramic matrix composites are leading their way into aerospace and nuclear applications. To fully understand the damage mechanism of this material, an improvement of in-situ monitoring techniques needs to be studied. Most non-destructive evaluation (NDE) techniques such as X-ray, C-Scan etc., have limited capabilities to damage inspection of components at extreme temperatures. Most likely, structural removal is required to examine the constituent. Therefore, a continuous health monitoring NDE techniques are necessary to detect and alert as damage initiates and propagates. Electrical resistance (ER) and acoustic emission (AE) are promising approaches that allow in-situ health monitoring from crack initiation up to failure due to thermal and mechanical loading. In this study, embedded high conductive silicon carbide fibers (SCS-6) with carbon core is used in a deliberate arrangement. The principle behind this strategy is to use the carbon in SCS-6 fibers as the main conducting material in two different non-oxide composites. Utilizing the carbon rods as implemented ER sensors will continuously intensify sensitivity to damage detection. SCS will improve ER health monitoring in complex structures and components located in the hot zone and operating under harsh conditions.

(ICACC-S1-P076-2020) Corrosion Resistance of 2D Nanomaterial-based Coatings on Stainless Steel Substrates

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Two dimensional (2D) materials have elicited considerable interest in the past decade due to a diverse array of novel properties ranging from high surface to mass ratios, a wide range of band gaps (insulating boron nitride to semiconducting transition metal dichalcogenides), high mechanical strength and chemical stability. Given the superior chemo-thermo-mechanical properties, 2D materials may provide transformative solution to a common yet persistent problem of significant socio-economic burden: the corrosion of stainless steel. With this broader perspective, we investigate corrosion resistance properties of stainless steel coated with 2D nanomaterials; molybdenum disulfide (MoS2), boron nitride (BN), bulk graphite in 3.5 wt. % aqueous NaCl solution. The nanosheets were prepared by a novel liquid phase exfoliation technique and the coatings were made by a paint brush to achieve uniformity. Open circuit potential (OCP) and potentiodynamic plots indicate the best corrosion resistance is provided by the MoS2 and the BN coatings. Superior performance of the coating is attributed low electronic conductivity, large flake size, and uniform coverage of SS substrate, which likely impeded the corrosive ions from the solution to diffuse through the coating. A linear relationship is observed between the corrosion current and the amount of corrosion suffered.

(ICACC-S1-P077-2020) Effect of Alkali-Silane Treatment on the Water Absorption of Kenaf Fibre Reinforced Polypropylene Composites

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In spite of the excellent specific properties of natural fibre reinforced composites, applications of them have been limited due to poor interfacial bonding and excessive water absorption. Various surface modification techniques exist in the literature to address poor interfacial bonding. Excessive water absorption is caused by the hydrophilic nature of the kenaf fibre. The present paper deals with some aspects related to this problem. Specifically, the effects of a combined alkali-silane treatment on the moisture diffusion resistance of kenaf fibre reinforced polypropylene composites are investigated. Composite plates fabricated by compression moulding using the film-stacking technique were subjected to long-term moisture diffusion tests. Untreated, alkali treated and alkali-silane treated kenaf fibres were considered. Scanning electron microscopy was used to analyse changes to the surface morphology of kenaf fibres caused by chemical treatments and moisture absorption. Results show that composite plates with fibre content less than 35% by mass exhibit Fickian-behaviour. Results also indicate that the alkali-silane

treatment reduces the saturation water content in the composites by as much as 45%. This reduction is the consequence of improved interfacial adhesion between the fibre and matrix.

(ICACC-S1-P078-2020) Nondestructive ultrasonic elastic modulus measurement of high-temperature ceramic electrical conductor and insulators

M. Fein[1]; P. Hsieh*[1]; R. Woodside[1]

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Magnetohydrodynamic (MHD) power generators can be operated at extreme temperatures that are beyond the service temperatures of most materials. Thus, ceramic electrodes and insulators are being considered for use in MHD generators. Due to elevated pressures and velocities within the generator and the potential for the development of large thermal gradients, the mechanical properties of the plasma facing materials within MHD generators are an important design consideration. We determined the Young's modulus, Poisson's ratio, and shear modulus of gadolinium-doped ceria (GDC), magnesium oxide (MgO) and aluminum oxide (Al_2O_3) samples using a non-destructive ultrasonic method. The microstructure of the GDC, MgO, and Al_2O_3 samples were further characterized using scanning electron microscopy. Theories for predicting Young's modulus and ultimate strength were evaluated for suitability in predicting the mechanical elastic properties of ceramic electrodes and insulators under MHD relevant conditions.

(ICACC-S1-P138-2020) Mechanical Properties Characterization of C/C Composite through Experiment and Simulation

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In this study mechanical properties of C/C composite plate specimen were evaluated through mechanical testing and simulation. The Young's modulus and hardness of individual fibers were measured through nano-indentation method. The mean values (and standard deviations) for Young's modulus and hardness were 4.388 GPa (1.007 GPa) and 0.33 GPa (0.189 GPa), respectively. Using SEM imaging, fiber diameter, the cross-sectional area of each tow were calculated, followed by calculation of fiber volume fraction in each tow. The Young's moduli of the tow were calculated using the rule of mixture and were found to be 193 GPa in axial direction and 15.6 GPa in transverse direction. A finite element (FE) model for a C/C woven composite was developed using micro-computed tomography. Using the calculated Young's modulus for a single tow and assuming orthotropic elasticity model, the finite element analysis was performed to predict the bulk Young's modulus of the composite plate.

(ICACC-S3-P079-2020) Characteristics of $Sr_{0.92}Y_{0.08}Ti_{1-x}V_xO_{3-\delta}$ anode using CH_4 fuel in solid oxide fuel cells

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Solid oxide fuel cells (SOFCs) are a promising alternative energy conversion device due to their high energy efficiency and fuel flexibility. Nickel/yttria-stabilized zirconia (Ni/YSZ) cermet has been widely used as an anode material in SOFCs. Ni/YSZ anode, however, permits only a few hundred ppm levels of sulfur compounds contained in commercial hydrocarbon fuels. In addition, the Ni phase is easily deactivated by carbon deposition in hydrocarbon fuels resulting to severe degradation of the cell performance. In this study, yttrium and vanadium co-doped strontium titanate ($Sr_{0.92}Y_{0.08}T_{1-x}V_xO_{3-\delta}$, SYTV) is investigated as an alternative anode materials for SOFCs for direct utilization of hydrocarbon fuels. Aliovalent cation substitutions of yttrium and vanadium may introduce electronic and ionic conductivity in the SYTV anode. The SYTV was synthesized by Pechini method, and the formation of perovskite phase were confirmed by TGA/DTA and XRD analysis. The SYTV exhibits excellent phase stability during the cell fabrication process and the cell operation. Due to the mixed ionic and electronic conductive (MIEC) property showed in the SYTV anode, the cell performance was improved comparing to Ni/YSZ anode in CH_4 fuel condition. In addition, long-term performance of the cell was excellent, and the carbon deposition was reduced in CH_4 fuel.

(ICACC-S3-P080-2020) Investigating Sr Vapor Phase Evolution from LSM/YSZ and LSCF Cathodes During Sintering

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After sintering LSCF cathodes, EDS maps of Sr typically show large amounts of Sr at the surface of the YSZ electrolyte layer (in the form of $SrZrO_3$) with virtually no Sr detected in the ceria barrier layer separating the cathode from the electrolyte. Normally, a concentration gradient of Sr across this ceria interlayer would be expected if solid state diffusion were the transport mechanism. However, consistent observations of the absence of Sr in the ceria interlayer between the cathode and the YSZ interface gives rise to the hypothesis that perhaps vapor phase transport plays a role. Thermodynamic calculations lend credence to this hypothesis, indicating that when SrO is heated to typical LSCF sintering temperatures of 900-1100°C in air containing 1% water (which is equivalent to ~33% relative humidity at room temperature), vapor phase $Sr(OH)_2$ concentrations are predicted to reach 1.1×10^{-8} which is of the same order of magnitude as the concentration of Cr vapor generated over the Cr_2O_3 scale on a metallic SOFC interconnect at 750°C on a day when the relative humidity is 20%. Experimental results will be presented that suggest that vapor phase transport plays a role in Sr migration from LSCF cathodes when the sintering temperature is greater than 1000°C, while no significant Sr vapor phase transport from LSM/YSZ cathodes was observed in the temperature range investigated.

(ICACC-S3-P081-2020) Microstructure of Anode-Supported SOFCs Fabricated by Low-Energy Microwave Sintering

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The electrode-supported SOFCs require co-firing between electrodes and an electrolyte, but it is difficult to obtain adequate electrode-electrolyte interfaces by co-firing that show high SOFC performance as expected due to the difference in sintering temperature between electrode and electrolyte. Co-sintering of 8 mol% yttria-stabilized zirconia (8YSZ) electrolyte and NiO/8YSZ anode would easily bring about high anodic polarization by over-sintering of NiO or delamination of anode/electrolyte interface by insufficient sintering. Materials can be selectively fired by microwave depending on their dielectric loss. In general, the loss of SOFC electrolyte is larger than that of electrode. Therefore, the microwave co-firing would result in high-performance cells. We then investigated preparation of anode-supported cells by microwave sintering. The heating selectivity was improved by decreasing incident power. Microwave adsorption of 8YSZ was also much depended by particles size or density of ceramics. The microwave adsorption and selectivity of 8YSZ much improved by increasing calcination temperature. Microwave co-sintering after controlling microstructure of calcined compacts resulted in delamination-free dense interface between dense electrolyte and porous electrode. Optimized microwave-sintering would be one of the most effective co-sintering processes for anode-supported SOFCs.

(ICACC-S3-P082-2020) Numerical Investigation of the Optimum Design of Solid Oxide Fuel Cell Electrodes

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Ideally, the performance of solid oxide fuel cells (SOFCs) could be unlimitedly improved by using materials with better electrochemical activity and creating microstructures with more active sites. In reality, there is an optimum combination of material and microstructure parameters for the cell performance under given operating condition, especially from the cost/benefits perspective. The present optimization study focuses on the numerical investigation of SOFC electrodes via multiphysics simulations. The performance of SOFC under different operating conditions was simulated via parametric study. The kinetic parameters for electrode reactions under different operating conditions are pushed until the specific electrode reactions are no longer rate-determining with respect to total cell performance. The changes of resistance and losses (e.g., ohmic, concentration, and activation losses) under different working conditions are also analyzed. Then an optimum activity term is proposed based on the asymptotic changes of resistances and losses. Further, these studies are also carried out for different microstructures and temperatures. The ability to improve SOFC performance even further is discussed with respect to electrode infiltration. This study provides quantitative analysis of the resistance/losses from different materials and microstructures, hence guiding future cell performance improvements.

(ICACC-S3-P083-2020) Microstructure and electrical properties of 8YSZ-LSGM composite electrolytes

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Over the last years great attention has been devoted to yttria-stabilized zirconia (YSZ), due to its potential application in different types of devices such as sensors and solid electrolytes and electrodes in clean-energy production devices, e.g. solid oxide fuel cells. Ceramic materials based on strontium- and magnesium-doped lanthanum gallate (LSGM) have been proposed as alternative solid electrolytes for device operation at lower temperatures than that of YSZ, due to their higher ionic conductivity. Recently, novel solid electrolytes consisting of a combination of two ceramic materials have been studied, envisaging advantages of the individual properties. In this work, the microstructure and the electrical properties of 8YSZ - x wt.% LSGM composites were investigated. The preparation of composites (x = 1, 10 and 20) was performed by the conventional mixing of oxides, followed by sintering at 1450 °C for 4 h. The relative density of composites decreases with increasing LSGM content; X-ray diffraction patterns show the predominant fluorite cubic phase of 8YSZ. For higher LSGM contents, the $La_2Zr_2O_7$ phase was detected. The average grain size increases with increasing LSGM content, although fine grained LSGM was found at the grain boundaries and triple junctions. The highest ionic conductivity was found for x = 1.

(ICACC-S3-P084-2020) Influence of dysprosium co-doping on enhancement of electrical conductivity of samarium-doped ceria electrolyte

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Ceria-based solid solutions have been considered as promising electrolytes for intermediate temperature (600–800°C) solid oxide fuel cells (IT-SOFCs) due to their excellent oxygen-ion conductivity compared to yttria-stabilized zirconia (YSZ). It is well known that enhanced ionic conductivity and other properties may be accomplished by introducing a second dopant. In this work, dysprosium ion was chosen as the second additive and its effect on densification and ionic conductivity was investigated. $Ce_{0.8}Sm_{0.2-x}M_xO_{1.9-x/2}$ compounds, M = Dy and x = 0, 0.025, 0.05, 0.10, 0.15 and 0.20, were prepared by solid state reaction followed by sintering. All compounds were found to possess cubic fluorite-type structure. The sintered solid electrolytes achieved densities higher than 92% of the theoretical value after sintering at 1500°C/3 h and higher ionic conductivity than that of the $Sm_{0.2}Ce_{0.8}O_{1.9}$ parent electrolyte.

(ICACC-S3-P085-2020) Modification of $(Mn,Cu)_3O_4$ spinels by iron substitution for potential material for interconnect protective coating

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$(Mn,Cu)_3O_4$ is a promising material for protective coatings for solid oxide cell interconnects. In comparison to the widely used $(Mn,Co)_3O_4$ spinel, it has no carcinogenic element and copper is more readily available. One of the drawbacks of this material is a relatively high thermal expansion coefficient and visible grain growth/coarsening throughout the high-temperature exposure. Partial substitution of copper by iron results in lowered thermal expansion, decreased grain coarsening, maintaining good sinterability and high electrical conductivity. Overall, the modified $(Mn,Cu,Fe)_3O_4$ spinels are interesting materials for replacing the current state of the art $(Mn,Co)_3O_4$ spinels.

(ICACC-S3-P086-2020) Reliability of the Electrical Conductivity Relaxation (ECR) technique: An Experimental Strategy to Enhance Accuracy of the Kinetic Parameters

B. Na*[1]; T. Yang[1]; S. Lee[1]; J. Liu[1]; H. Abernathy[1]; T. Kalapos[1]; G. Hackett[1]

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The electrical conductivity relaxation (ECR) technique is one of the most popular methods to evaluate the electrochemical properties such as the surface exchange coefficient (k_{chem}) and bulk diffusion coefficient (D_{chem}) of mixed ionic and electronic conductors (MIECs). Although ECR experiment is simple and intuitive, the fitted result of either k_{chem} or D_{chem} always carries some degree of uncertainty when they are simultaneously obtained from one relaxation curve fitting. In this study, the group attempts to shed some light on the uncertainty of k_{chem} or D_{chem} values obtained from ECR experiment via the statistical analysis tool we have developed. Specifically, we investigated the impact of Biot number, the ratio of the relaxation time of surface exchange and bulk diffusion, on the reliability of k_{chem} and D_{chem} under various temperature and oxygen partial pressure. LSCF was chosen as a sample material and its k_{chem} and D_{chem} measured from three bulk samples with distinct Biot number were evaluated, demonstrating the direct relationship between Biot number and the degree of uncertainty of k_{chem} and D_{chem}. Based on the results, a comprehensive experimental strategy for ECR measurement is developed to improve the reliability of k_{chem} and D_{chem}.

(ICACC-S3-P133-2020) Innovative approach of Anode Functional Layer (AFL) design for Solid Oxide Fuel Cell system

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Application of Anode Functional Layer(AFL) to Solid Oxide Fuel Cell system is always at the center of attention for their high performance and structural stability. Ni-YSZ/YSZ composite AFL has been widely used for conventional anode supported SOFC. However, their severe operating temperature (up to 900°C) and redox cycle caused agglomeration of Ni metal and delamination or mechanical failure of AFL layer, which have been the main offenders of cell degradation. Here we introduce an innovative AFL material, where nano-sized catalytic Ni particles were doped inside $La_{0.43}Ca_{0.37}Ni_{0.06}Ti_{0.94}O_{3-d}$ perovskite substrate and spontaneously exsolved on its surface

under SOFC operating condition (low pO_2). Electrochemical performance tests for both fuel cell and electrolysis mode were conducted to identify characterize degradation mechanism of AFL layer. This nano structured AFL design prepared by exsolution method showed superior stability even under direct utilization of hydrocarbon fuel and the homogenously exsolved nano Ni particles on AFL backbone were successfully maximized Three Phase Boundaries (TPBs), which is the critical requirement of SOFC electrode design.

(ICACC-S3-P134-2020) In-situ nano structured electrode design for symmetric Solid Oxide Fuel Cell application

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Symmetrical Solid Oxide Fuel cells has been studied for its higher thermo-mechanical stability with simple and low fabrication cost. As a competitive candidate, SFM($Sr_2Fe_{1.5}Mo_{0.5-x}O_{6-d}$) has been widely adopted as symmetric electrode material to accomplish its requirements under various pO_2 range. However, their insufficient catalytic activity than conventional anode or cathode has hindered its real application. Herein we demonstrate an innovative electrode design for symmetric SOFC, where state of the art nano exsolved catalytic metals were homogenously socketed on the surface of SFNM($Sr_2Fe_{1.5}Ni_xMo_{0.5-x}O_{6-d}$) perovskite backbone. We doped Ni inside the perovskite lattice structure and activated its in-situ growth by simply manipulating pO_2 environment. Electrochemical Impedance Spectroscopy (EIS) and Leaner Sweep Voltammetry (LSV) test have been conducted to identify characteristic impedance of each material and cell performances, respectively. This nano-structured electrode design prepared by in-situ exsolution method successfully maximized catalytically active region, while optimizing their structural stability for next generation of symmetric SOFC application.

(ICACC-S6-P087-2020) Crystallization of the $Na_2Fe_xNi_{1-x}P_2O_7$ glass and ability of cathode for sodium ion batteries

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$Na_2FeP_2O_7$ crystallized glass has been acknowledged that it could be the cathode candidate for the sodium ion batteries (SIBs). In this research, in expectation of further improving discharge potential, $Na_2NiP_2O_7$ which has high potential of pyrophosphate $Na_2MP_2O_7$ system, tried to be practiced as the positive electrode of SIBs. The method of activating $Na_2NiP_2O_7$ would be to replace it with active Fe^{2+}. Furthermore, the dQ/dV plot showed two weak $Ni^{3+/2+}$ reduction peaks at 4.3 V and 4.4 V in the discharge curves of composition x = 0.25. As a result, a little Fe substitution activated $Ni^{3+/2+}$ in the $Na_2Fe_xNi_{1-x}P_2O_7$ composition. Since the redox potential of Ni^{2+} already exceeded the decomposition potential of the electrolyte $NaPF_6$, a solid electrolyte was used to assemble an all-solid-state sodium ion battery. In order to reduce the interface resistance, $NaPO_3$ glass solid electrolyte with the feature of low temperature softening flow was used to form a dense sintered body. The prepared sodium all-solid-state battery was able to obtain three $Ni^{2+/3+}$ derived oxidation peaks at about 4.2 V, 4.6 V and 4.8 V during charging. From the above, it has been shown that the $Na_2Fe_xNi_{1-x}P_2O_7$ crystallized glass functions as a positive electrode of a sodium ion secondary battery. In addition, the activatable $Na_2NiP_2O_7$ crystallized glass can be applied to all-solid-state sodium ion battery.

(ICACC-S6-P088-2020) Two dimensional nanomaterials functionalized by polymer-derived ceramic as stable battery electrodes

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We studied synthesis of layered polymer-derived ceramic (PDC) - transition metal dichalcogenides (TMD) structure via pyrolysis of polysilazane or polysiloxane functionalized TMD flakes. The layered morphology and polymer to ceramic transformation on TMD surfaces was confirmed by use of electron microscopy and spectroscopic techniques. Tested as thick film electrode in a Alkali-metal-ion battery half-cell, PDC-TMD showed the classical three-stage reaction with improved cycling stability and capacity retention than neat MoS2. Contribution of conversion reaction of Alkali-metal ion/TMD system on overall capacity was marginally affected by the presence of SiCN or SiOC while reaction-irreversibility arising from electrolyte decomposition was greatly suppressed. This is understood as one of the reasons for decreased first cycle loss and increased capacity retention. Preliminary tests suggest that molecular level interfacing with precursor–derived ceramic is an effective strategy for suppressing the metal-sulfide/electrolyte degradation reaction at low discharge potentials.

(ICACC-S6-P089-2020) Rechargeable Li-O_2 Battery Architectures Based on Multilayered Li^+-Selective Solid Membranes

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With the increase in electric vehicles, rechargeable Li-O_2 batteries (LOBs) are attracting considerable attention, as their theoretical energy density is several times higher than that of current Li-ion batteries. Recent studies have suggested that the use of Li^+-selective solid membranes (LSSMs) between the anode and cathode is an effective approach to address the technical issues of LOBs, such as the low round-trip (energy) efficiency and poor cycle life. Herein, an LOB architecture based on a triplex-LSSM is proposed. A facile tape-casting process was used to fabricate triplex-LSSMs with controlled microstructures: a thin, dense layer acted as a separator between the anode and cathode, while two porous layers provided mechanical support for the thin layer and served as a reservoir for liquid electrolytes essential for LOB operations. The LOB cell assembled with the triplex-LSSM showed almost the same discharge behavior as the conventional cell without the triplex-LSSM, indicating its successful operation. Furthermore, the LOB cell with the triplex-LSSM exhibited an improved rate-capability compared to that of the cell with the commercial membrane and showed reversible redox behavior during cycling. The triplex architecture can be easily scaled up in size without significant loss of dimensional and mechanical stabilities.

(ICACC-S6-P090-2020) Biomass derived carbons and PDC functionalized carbon composite for electrochemical energy storage

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Biomass derived carbons are among the most promising candidates for large-scale electrical energy storage devices owing to the low cost, sustainability, and widespread of resources and molecular precursor-derived Si-based ceramics show high chemical resistance and thermal stability. The electrochemical performances of the prepared biomass carbons were investigated in supercapacitors. Both the cassava

and bamboo samples possessed high graphitization degree and good surface wettability demonstrated by X-Ray Diffraction (XRD), Raman spectroscopy and Fourier-transform infrared spectroscopy (FTIR). As a supercapacitor electrode, the cassava derived carbons showed the best performance with maximum specific capacitance of 212 F g-1 and a capacity retention of 95% after 2000 cycles. Besides, as an electrode material for SIB, PDC-biomass carbon composite demonstrated the highest charge capacity of 110 mAhg-1 (at 25 mAg-1) with stability in various current densities. This approach shows great potential to achieve advanced electrode materials, from low-cost, green and industrial-grade production of biomass-derived carbon materials and PDC by a simple synthesis for advanced energy storage applications in the future compared to the conventional approaches.

(ICACC-S6-P091-2020) Electrospun SiOC Ceramic Fiber Mats as Freestanding Electrodes for Electrochemical Energy Storage Applications

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Polymer-derived-ceramics (PDCs) and PDC-based fibers are being studied for electrochemical energy storage applications. Among these, SiOC and SiCN have shown most promise, especially as electrodes in lithium ion batteries (LIB). The study of PDCs for supercapacitor electrodes is, however, very limited. This work investigates the fabrication of SiOC based PDC fiber mats via electrospinning of three pre-ceramic polymers (MK, H44, RSN) of varying composition, and their applications in supercapacitors and lithium ion battery (LIB). Characterization including electron microscopy, Fourier transform infrared spectroscopy (FTIR) and Raman spectra was performed to study the effect of precursor chemical composition, pyrolysis time and temperature on the morphology, polymer to ceramic transformation and free carbon content. As supercapacitor electrodes, the H44 derived SiOC showed the best performance with maximum specific capacitance of 50 F g^{-1} with capacity retention of ~100% after 2000 cycles. Similarly, as LIB electrodes, H44 derived SiOC fiber mat showed highest reversible capacity of 578 mAh g^{-1} at a current density of 50 mA g^{-1} Results indicate that higher pyrolysis temperature and longer pyrolysis times facilitate enhanced charge storage in SiOC materials.

(ICACC-S6-P092-2020) Cerium Oxide/Graphene Aerogels for Electrochemical Energy Storage Applications

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A breakthrough in energy storage materials is required to cater to the ever-growing energy needs of modern society. Nanotechnology could hold key to this new era of materials. Most of the currently used active materials in electrochemical energy storage devices (EEDs), such as cobalt-based oxides, have major shortcomings such as toxicity, high cost, low suitability, and dubious ethical origins. Thus, there is a dire need to develop environmental friendly low-cost energy storage material. In this regard, CeO2/graphene aerogel is obtained through a simple hydrothermal technique as an active material for supercapacitors - a type of EEDs which acts as a bridge between batteries and traditional capacitors. Herein, we formulate a binary composite using two types of supercapacitor materials such that the advantage of one material effectively suppresses the drawback of the other. The synthesized CeO2/graphene aerogel was physiochemically characterized using XRD, FTIR, SEM, XPS, and gas adsorption surface analysis. Electrochemical analysis revealed that the optimized composite exhibit high specific, good rate performance and excellent cycle life. An asymmetric device constructed using the active material revealed high specific energy and power densities with a very stable cyclic performance. Thus, we demonstrate a strategy to produce CeO2/graphene aerogel for high-performance supercapacitor applications.

(ICACC-S9-P093-2020) Titania microspheres obtained by internal gelation method and its photocatalytic activity

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Titanium dioxide (TiO_2) it's a particular semiconductor which have an interesting set of characteristics and properties that allow its application in a wide range of areas, including pigments, biomaterials, catalysis, sensors, ion exchange, etc. Due its thermal/chemical stability and low cost, TiO_2 has been intensively studied and applied as a photocatalyst for the treatment of effluents containing organic pollutants. The aim of this work focused to obtain porous TiO_2 microspheres from $TiCl_4$ by internal gelation method through microfluidic glass capillary device, in order to explore its diameter and porosity toward optimizating photocatalyst reactors, such as column reactors or fluidized bed reactors (for aqueous or gaseous effluents), as well as easily separating TiO_2 microspheres from the reaction middle. The obtained microspheres were heat treated at temperature of 550°C for an hour and characterized concerning their size and sphericity (SEM), crystalline phases (XRD), specific surface area and porosity (BET/BJH). The microfluidic device allowed to obtain microspheres with regular size and sphericity. The heat-treated microspheres showed only anatase phase. Were carried out phocatalysis tests for the degradation of Amoxicillin in water and its degradation was measured by spectrophotometer and HPLC. After 60 minutes of reaction, most of 50% of amoxicillin had been degraded.

(ICACC-S9-P094-2020) Robust porous ceramics via freeze casting of viscous solutions

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Porous ceramics with desirable transport and mechanical properties are of interest for applications such as filtration, bioscaffolds and catalyst supports. Freeze casting has shown promise since it provides tailorability in porosity, pore size, morphology, and directionality, which affect both transport and mechanical properties. Moreover, in suspension-based freeze casting, several studies have demonstrated that changing suspension properties (viscosity, pH and additives), incorporating anisotropic fillers, and large-scale pore alignment improve mechanical properties. While suspension-based freeze casting offers numerous options for engineered porosity, there are only a few such strategies in solution-based freeze casting of preceramic polymer. In this study, the rheological effect on solidification and freeze-cast porous structure is investigated to create porous ceramics with both high permeability and compressive strength. The viscosity of preceramic polymer solutions is varied by changing the molecular weight of preceramic polymer. It is shown that high viscosity in polymer solution results in bridge formation between lamellar walls which improves compressive strength. To show the efficacy of this method, both the permeability and compressive strength of porous SiOC ceramics are measured.

(ICACC-S9-P095-2020) Fabrication of Ceramic Diffuser for Subsurface Irrigation Application

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Technologies that ensure the availability of water requirements of a crop is to be developed in order for agriculture to be sustainable in the face of climate change. Technologies related to irrigation are needed to be improved with the aim of reducing associated labor cost. The common system developed which is a water-saving system used to irrigate upland crops by soil capillarity is a subsurface irrigation system. In this study, the suitability of subsurface irrigation with the use of a ceramic diffuser was evaluated by using an iron rich

material, clay and silica to achieve a porous diffuser. The samples were analysed using a TGA-DTA and it showed a sharp endothermic peak at 907.67 °C which indicates the oxidation of iron particles by process of diffusion of oxygen. Iron rich material when exposed to higher temperature oxidized and its reaction leaves the body in a form of gas resulting to a porous body. Furthermore, TGA-DTA analysis proved that the increase ratios of iron rich material had led to an increase in the porosity and water absorption. Thus, as the porosity of the formulation increases flow rate also increases from 80 ml/h to 500 ml/h. An actual testing of a plant was carried out to assess the performance of the subsurface irrigation system. It was evaluated by recording the plant growth in terms of its height and the number of leaves growing per week.

(ICACC-S9-P096-2020) A new approach to develop high porosity ceramic foam

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Porous ceramics are of great interest due to their numerous potential applications in the catalysis, adsorption, separation and filtration of molten metal or hot gases and the refractory insulation of furnaces, as well as other trends involved in engineering. In view of the demand of porous ceramic the present work was focused on the development of highly porous light weight ceramic foam using sucrose as a foaming agent. Oil of vitriol (H_2SO_4) was used to create the pores by reacting with sucrose and also generating carbon template. This carbon template was removed after sufficient heating which further helps in generating pores. Sintering of porous ceramic obtained was done to get sufficient strength. Sucrose and oil of vitriol amount, ceramic compositions, mixing and drying time, pre-sintering and sintering temperatures were investigated and optimized to get the highly porous ceramic foam with sufficient mechanical strength. Highly porous light weight ceramic foam was obtained with porosity ranging from 75-90% and compressive strength from 0.8-1.5 MPa.

(ICACC-S9-P097-2020) Theoretical and Experimental Investigations of Particle Engulfment of High-aspect Ratio Particles in Freeze Casting

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Strength and permeability are two critical characteristics for porous ceramics that are used as filters or supports for catalyst. The ideal filters and supports must be sufficiently durable to withstand the fluid pressure while allowing for rapid fluid transport. However, it is difficult to achieve the combination of strength and high permeability in the same system. One approach to optimize the pair is by reinforcing a highly permeable structure, in this case, a lamellar pore structure, with inter-lamellar wall bridges produced by dispersing high-aspect ratio particles in a freeze-casting solution. According to particle engulfment theories, particles sizes larger than a critical radius will be engulfed instead of being pushed away by the freezing front to the pore wall. However, most particle engulfment theories fail to explain the partial engulfment and the alignment of high-aspect ratio particles. To remedy these deficiencies, we propose a modified particle engulfment model. The model takes into the account angles and positions of the high-aspect ratio particles with respect to the freezing dendrites. Theoretical predictions from the modified model are compared with the experimental results of SiC whisker-reinforced SiOC produced via freeze casting of polysiloxane preceramic polymer solutions.

(ICACC-S9-P098-2020) Gel-casting of porous alumina supports with platinum nanoparticles as catalyst

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Porous alumina ceramics were prepared by gel-casting using hollow preexpanded polymer spheres as sacrificial template. The platinum nanoparticles were introduced I) during the gel-casting of alumina foams by coating the expandable microspheres with platinum (Pt) nanoparticles, and II) by impregnation of pre-sintered alumina foams with Pt nanoparticles. The Pt nanoparticles were coated on the surface of the polymer spheres by a polyelectrolyte-assisted process having three polyelectrolyte layers with alternate charges. The Energy-dispersive X-ray spectroscopy (EDS) and Ultraviolet-visible spectroscopy (UV-Vis) confirmed the successful adsorption of Pt on the polymer microspheres. The prepared alumina foam was dried, debinded and sintered and characterized regarding its porosity, morphology, elemental analysis and catalytic performance. The reconstructed 3D structure of the foam using X-ray microtomography showed a homogeneous 3D microstructure, suggesting well dispersion of the EPS in the ceramic slurry. The image analysis of the 3D structures showed a 58 vol% porosity consisting of 99.7 % open pores. The EDS confirmed the successful transfer of the Pt nanoparticles to the pore walls of the alumina foams in both processing routes and the electrocatalytic performance was evaluated and compared. The results showed a promising way to design porous ceramics with catalysts.

(ICACC-S10-P099-2020) Effects of Oxide Layer at Al/Fe Sliding Interface on Tribological Behavior: Molecular Dynamics Simulation

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In the automobile industry, it is important to reduce the friction in the engine for the improvement of fuel efficiency. Cast iron and aluminum are generally used as sliding components in engines. Previous experiment reported that oxidation of sliding surfaces of Fe/Al in engine oil contributes to low friction. However, the mechanism of friction reduction by oxidation is still unclear, because the dynamics of interfacial chemical reactions are difficult to be studied. Thus, the effect of oxide layer at Al/Fe interface on the tribological behaviors was investigated by the molecular dynamics simulation method using the reactive force field. To understand the effect of the oxide layer, we construct two sliding models of (i) Fe/Al as the non-oxidized surfaces and (ii) Fe_2O_3/Al_2O_3 with OH terminations as the oxidized surfaces. All simulations were performed for 25 ps with a load of 1 GPa and a sliding speed of 100 m/s. We found that Al surface layer adheres to Fe surface at Al/Fe interface, however, adhesion was not observed at Fe_2O_3/Al_2O_3 interface. Friction coefficients at Fe_2O_3/Al_2O_3 was lower than that at Al/Fe. These results suggests that surface oxidation layer suppresses surface adhesion and contributes to low friction.

(ICACC-S10-P100-2020) Formation, Wear, and Regeneration Mechanism of Tribofilm of Silicon Carbide in Water Lubrication: Molecular Dynamics Simulation

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Water lubrication does not use lubricating oil, and thus it is expected to be applied to hydroelectric facilities and so on. Silicon carbide (SiC) is proposed as sliding material for water lubrication because of

its super-low friction in water. The super-low friction of SiC has been attributed to a tribofilm formed at the sliding interface by mechano-chemical reaction. On the other hand, it was reported that the thin tribofilm is maintained at the sliding interface, though SiC worn out during the sliding. This result indicates that the tribofilm is worn out but regenerated. In order to design low friction sliding materials, the formation, wear and regeneration processes of the tribofilm should be clarified. In this study, we performed sliding simulation of SiC in water by molecular dynamics method. We found that oxygen atoms in water reacted with SiC surface and SiO_2 were formed. On the other hand, hydrogen atoms diffused to SiC bulk region. We also found that the SiO_2 layer worn out and SiO_2 particles dissolved in water. This result indicates that the high viscosity colloidal silica layer is formed at the sliding interface and the friction changes from a boundary lubrication to a hydrodynamic lubrication, leading to the low friction. The detailed formation, wear, and regeneration processes of the tribofilm will be discussed in the conference.

(ICACC-S10-P101-2020) Drying and analysis of mechanical and thermal behaviour of kaolin as ceramic material

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After shaping the water must be removed so that the shape becomes sufficiently rigid to withstand handling and setting in the kiln. Drying is a critical stage in ceramic manufacturing. If a clay piece is not completely dry before the first firing, it will crack, warp or even explode. An analytical study of fluedized bed drying process considering cross flow has been done. Diffusion in material has been considered. The modeling of heat and mass transfer between air and material in moving bed dryers is based on the application of enthalpy balance, mass balance, heat transfer rate, mass transfer rate and diffusion equation for a Specified size material. These equations obtained are highly implicit in nature and need to be solved simultaneously. Equations for mass balance of moisture between material and air, enthalpy balance, heat transfer rate and mass transfer rate at the outer surface of the material and diffusion of moisture inside a considered size of material, have been solved simultaneously through an implicit scheme using TDMA. A mathematical model and a simulation program in C++ have been developed for steady state cross flow deep bed drying process of spherical particles The input parameters include the temperature, moisture content, mass flow rate of material at one side of dryer and the temperature, humidity and mass flow rate of air at bottom of the dryer.

(ICACC-S10-P102-2020) Molecular Dynamics Simulation of Pt Nano-Particle Sintering on Porous Carbon for Polymer Electrolyte Fuel Cell

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Next-generation polymer electrolyte fuel cell (PEFC) requires higher power generation efficiency. However, sintering of Pt nano-particles in electrode catalyst causes a loss of active surface area that results in degradation of the catalyst performance. In order to improve the power generation efficiency of the PEFC, the sintering process of the Pt nano-particles should be elucidated and the simulation is effective for elucidation of the sintering process. However, the conventional sintering simulation was only performed by the models of two or three particles on a surface, and the sintering process of the Pt nano-particles on the porous carbon which is a realistic electrode catalyst material have not been elucidated. In this study, sintering of Pt nanoparticles on a porous carbon was investigated by reactive molecular dynamics simulation. When two adjacent Pt nano-particles are on the same carbon nano-particle, the sintering was observed. On the other hand, when two adjacent Pt nano-particles were at the interface consisting of two carbon nano-particles, the sintering was not observed. This is because both Pt nano-particles are adsorbed on the two carbon nano-particles consisting the interface and their movement is restricted. This result is not seen in the conventional sintering simulation where two or three particles are put on a surface model.

(ICACC-S10-P103-2020) DFT Study of the Effect of Rare Earth Dopants on the Cohesive Energy of Amorphous Si - B_6O Interface

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Boron suboxide is a promising material system for light-weight armor applications. It has an ultra-high hardness ranging from 24 to 45 GPa, a low density (~2.56 g/cc), high yield strength (26 - 30 GPa) at hydrostatic pressures above 50 GPa, high oxidation resistance at operating temperatures, and is chemically inert. However, it suffers from a low fracture toughness and stress induced amorphization which reduces the shear strength under extreme environmental conditions. To improve its performance, researchers have investigated the effects of rare earth (RE) additives on densification and microstructure to induce the formation of nanolayer grain boundary films (typically < 2 nm thick). Recent experimental studies have shown that Yb promotes abnormal grain growth whereas Y and La suppress it. In this poster, we develop a simple DFT model for an amorphous Si film at a (101) boron suboxide twin boundary based on AC-STEM/EDS data and calculate the effects of silicon concentration and RE dopants (Y, La, Lu, and Eu) on the cohesive energy and thereby the relative stability of the grain boundary interface.

(ICACC-S10-P104-2020) DFT Study of the Impact of Impurities in SiC Bulk and Grain Boundaries

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Density Functional Theory (DFT) was performed to determine the favorability of defects in silicon carbide (SiC) systems. Vacancy, substitutional, and interstitial defects were developed for bulk crystalline SiC and $\Sigma 9$ {122} SiC tilt grain boundaries. Ten elemental dopants were tested in varying configurations to understand their influence on the SiC system. Impurities of carbon, silicon, and silver were examined before other dopants in order to compare experimental values to literature values, and validate parameters used in calculations. All systems were relaxed and the resulting energy difference was related to non-defected structures to determine the formation energy of the impurity. Lower values of formation energy determined stable, and therefore favorable systems. Carbon vacancies in bulk were found to be more stable than silicon vacancies. Interstitial formation energies in bulk were found to be the highest of the recorded energies. This project was performed with the intention of predicting situations that are likely to be encountered during processing of SiC. This will allow probable cases to be further examined and classified as being beneficial or hazardous to the mechanical properties of the material. Then, processing procedures can be developed with this knowledge in mind.

(ICACC-S16-P105-2020) Modeling heat of reaction and compressive strength of geopolymers

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A geopolymer is an alkali-activated alumino-silicate material that can be used to make mortar or concrete. Typical starting materials are metakaolin, slag, and fly ash. The process begins with dissolution of the ingredients to yield silicate and aluminate species, which polymerize into an amorphous or partially microcrystalline gel. The reaction steps are exothermic. Heat flow measurements of individual

steps are often limited by poor resolution. However, the cumulative heat yields good guidance of the overall reaction progress and is modeled frequently to estimate the total heat of reaction, q_∞. Knowledge of q_∞ allows determination of the time for materials properties to reach their ultimate values. Also, materials properties have been modeled directly. We have prepared nine geopolymers and have measured the cumulative heat release for 21 days and compressive strength up to 28 days. We have used the results to evaluate the power of the often used Knudsen linear-hyperbolic and parabolic-hyperbolic models as well as the so-called exponential model. We will show and discuss where and, in some cases, why a model deviates from the experimental data and that calculation of q_∞ and ultimate strength may be associated with large uncertainties.

(ICACC-S16-P106-2020) Development of roof tiles based on alkali activated-materials

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Geolymers are obtained from the alkali-activation of aluminosilicate precursor materials, such as the metakaolin, with the addition of user-friendly alkaline reagents, such as sodium or potassium soluble silicates or hydroxides, in the presence of water. The geopolymers hardening process and strength gain occur at room temperature, dispensing the application of thermal treatments in ovens. Therefore, these materials show great potential to replace the ceramic ones, since the elimination of the firing step generates savings and environmental advantages. This work aims to verify the feasibility of using geopolymer materials based on metakaolin, activated by two distinct alkaline solutions, in place of traditional ceramic roof tiles. Geopolymeric compositions were performed with proportion 1.0000:1.0000:0.3500:0.4725 (metakaolin: fine aggregate: alkali-activator: water). It was used natural river sand as the fine aggregate, and sodium hydroxide and sodium silicate (water glass) solutions as activators. Eight samples were produced for each composition, being four pieces subjected to three-point flexural strength test and four pieces subjected to the water absorption test. The results demonstrated the technical viability of using geopolymer materials for roof tiles production, since the obtained parameters met the Brazilian and international standards.

(ICACC-S16-P107-2020) Investigating Chemical, Physical and Mechanical Properties of Eco-cement Produced Using Mineral Sources from Pulp and Paper Mills and Granite Waste

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The concern about climate changes and the pollution increase necessarily led to the creation of green policies for sustainable development. Thus, the incorporation of industrial wastes in building materials is one of the most efficient ways for the civil construction to become a sustainable activity. This work aimed to the production of eco-cement from grits waste, generated in pulp mills, and granite waste, generated in the granite rock processing. In order to obtain the wastes amounts for the composition of eco-cement, the parameters of lime saturation factor (LSF), alumina ratio (AR), silica ratio (SR) and Bogue equations were applied. The three mixtures obtained (M1-M3) were decarbonated at 900°C for 3 hours and sintered at 1450°C for 15 minutes. The mixtures were characterized by means of X-ray diffratometry (XRD) for the identification of crystalline phases and X-ray fluorescence (XRF) for quantification of oxides in each sample. The results showed that the crystalline phases identified in the mixtures were similar to those found in conventional clinkers and cements, confirming the potential use of wastes as an alternative for eco-cements production.

(ICACC-S16-P108-2020) Alternative geomaterials using laterites: Reactivity and properties of use

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Lateritic soils resulted from an alteration of clay via accumulation of iron, Fe3+, or aluminium, Al3+, species. The lateritic soils constituted of concentrated ions oxide of iron, silicon and/or aluminium, with kaolinite, halloysite, montmorillonite as predominant minerals clay. Nowadays various stabilization techniques have been often used to improve the structural, microstructural properties of laterites such as chemical and mechanical stabilization. Numerous studies have been conducted on stabilization of raw or calcined laterites using the aforementioned stabilizers above. The aim of the present work was to investigate the behavior of the iron minerals in two lateritic soils (different in iron content) during the consolidation process with acid or alkaline solutions. 57Fe Mössbauer spectroscopy was used to monitor any changes in the iron valence and coordination number occurring during the geomaterials process and the consolidated products were also characterized by electron paramagnetic resonance (EPR), X-ray diffraction (XRD), scanning electron microscopy (SEM), and compressive strength measurements. The state of consolidation will be determined through the measurements of the properties of use and correlated with the microstructure.

(ICACC-S16-P136-2020) Effect of the use of a fruit waste (Musa paradisiaca) onto the microstructure and the densification of a kaolino-illitic clay from Central African Republic (RCA)

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The main target is to promote the use of local clays in RCA for the production of environmentally friendly ceramics exhibiting various microstructures. Therefore, we aim at studying the sintering of the selected clay and the effect of using the peel waste of Musa paradisiaca (readily available in RCA) fruit on the final microstructure and properties of use of this clay. The deposit of Nzila (noted (NZ2) has been selected regarding the geographical location, the available amount and the previous use for the production of local handmade pottery. A commercial kaolin (BIP kaolin, Imerys Ceramics) serves as the control or reference raw clay to investigate the Musa paradisiaca fruit waste influence over the sintering behavior. The results obtained were correlated to the compressive mechanical strengths obtained for the studied samples. For the kaolin BIP, the increase of Musa paradisiaca fruit waste from 0 to 30 mass% leads to an increase of the total porosity from 18% to 24% r after sintering at 1000°C and 1200C respectively. When using NZ2, a kaolino-illitic clay, the increase of the fruit waste tends to increase the total porosity up to 13% while the compressive strength is decreased to 63% of the initial values without waste.

(ICACC-S16-P137-2020) Production of Ceramic Wall Tiles Utilizing Siliceous Iron-rich Material from Northern Mindanao, Philippines

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The properties of ceramic wall tiles can be tailored depending on the composition of the main ingredients and these materials are commonly used in houses and buildings as decorative or finishing surface. They have high water resistant and durable. In this study, a locally sourced siliceous iron-rich material from Santiago, Iligan City, Philippines was utilized, along with commercially available clay and feldspar to produce the ternary mixture of ceramic wall tiles. Based on the chemical compositions analysis of the siliceous iron-rich material (IRM), it showed 37.95% Fe_2O_3 and 32.74% SiO_2. The physical properties revealed zero percent fired shrinkage and 12.26 % water absorption. The formulation with 10% IRM sintered at 1235 °C produced a ceramic wall tile with low absorption of 0.14% and a high Modulus of Rupture (MOR) at 8.14 MPa. Surface morphology showed formation of elongated grains and other crystalline phases in the glassy matrix. Such morphology is responsible for the good strength of the fired product. X-ray diffraction revealed that quartz, mullite and hematite are the major phases formed in the sintered body. Comparison of the fabricated ceramic wall tiles from commercially available was also made in this study.

(ICACC-S18-P109-2020) Structural modification on EB-PVD thermal barrier coatings subjected to advanced cryogenic gradients, oxidation and thermal shock

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The primary task of thermal barrier coatings is to protect the underlying material from hot corrosion and high-temperature oxidation. In this paper we present the morphological and structural effects observed TBC's subjected to cryogenic gradients, oxidation and thermal shock of EB-PVD coatings. For this studies the substrates used were NIMONIC 80 alloy and high density izomolded graphite. There were used three layers for the coating of substrate: NiCrAlY alloy as binding layer, ZrO_2 substituted with 8 mol% rare earths mixtures and ZrB_2. After 10 hours' deposition time the coatings were evaluated by means of XRD and high resolution SEM which showed a good adherence and a continuous film along the substrate. The morphological characteristics observed were of polycrystalline nature with polyhedral shaped particles with rounded edges having an average grain size of 16,01 ± 6,19 μm. Morphological studies on induced cryogenic gradients showed that the barrier coating thickness of 5-7 μm improved the depth of the affected depth of the substrate from 15-17 μm to 5-7 μm. XRD and EDS analysis showed the formation of oxides Cr_2O_3 and Cr_2NiO_4 on the surface of the sample. After thermal shock at 1200°C for 5 hours the result is the detachment of the coating and the oxidation of the substrate for a depth of 15 μm and 30 μm respectively.

(ICACC-S18-P110-2020) Fabrication of Reaction Bonded TiB_2/Si Composites and Property Study

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Titanium diboride (TiB2) is an extremely hard ceramic which has excellent heat conductivity, oxidation stability and resistance to mechanical erosion and are used as impact resistant armor, cutting tools, crucibles, and wear resistant coatings. Broader application of this material is inhibited by economic factors, particularly the cost of densifying a material with a high melting point. In this study, reaction bonded TiB2 (RBTB) composites are fabricated by the reactive infiltration of molten Si into preforms of TiB2 plus carbon. The final TiB2 content in the composite is controlled by the starting TiB2 powder size, slip preparation and preforming process, initial carbon content and infiltration parameters. Microstructure analysis indicates uniform distribution of TiB2 particles in the Si matrix. The thermal conductivity of the RBTB ranges from 129 W/m-K at -55C to 73 W/m-K at 500C. The coefficient of thermal expansion (CTE) of RBTB ranges from 4.4 ppm/K at room temperature to 5.3 ppm/K at 200C, which is close to the CTE of Al2O3 in the same range (NIST data). RBTB is a potential material to replace Al2O3 in thermal management applications. The mechanical properties (hardness, fracture strength and toughness) of the RBTB composites are also measured.

(ICACC-S18-P111-2020) Atomic Layer Deposition of Ultra-High Temperature Ceramics as Hydrogen Environmental Barrier Coatings for Nuclear Thermal Propulsion

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Nuclear thermal propulsion is an attractive in-space propulsion technology due to its large specific impulse. This specific impulse is achieved by using energy released from uranium fission to heat hydrogen gas to over 2500°C before expelling it from a supersonic nozzle to generate thrust. However, hydrogen diffuses into the nuclear fuel elements and embrittles them. In this research we investigate the use of nanoscale coatings by atomic layer deposition (ALD) to address this issue. Ultrathin WN films were deposited by ALD on zirconia nanoparticles and yttria stabilized zirconia (YSZ) micropowders. Differential thermal analysis (DTA) was used to investigate the efficacy of the W/WN films in preventing hydrogen attack. The temperature at which hydrogen reacted with the sample increased with film thickness, thereby indicating that the film inhibited this reaction. However, the existence of a hydrogen reaction peak in the thickest film indicated a material with a smaller hydrogen diffusivity is necessary for a substantial decrease in hydrogen interaction with the substrate. Therefore, density functional theory was used to compare W to other high temperature refractory ceramics as alternative coating materials.

(ICACC-S18-P112-2020) Preparation and Structure of SiOC Fibers Derived from Cyclic Silazane/PAA Hybrid Precursor

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Ceramic matrix composite (CMC) materials have been considered a desired solution for lightweight and high temperature applications. Simultaneously, among all different CMC reinforcements, PolymerDerived Ceramic (PDC) fibers have gained attention for the intrinsic thermal stability and mechanical strength with simple and cost-effective synthesis techniques. Here, carbon-rich SiOCN fibers were synthesized via hand-drawing and polymer pyrolysis of a hybrid precursor of a cyclic silazane and polyacrylic acid (PAA). The type of silazane reported in this work is considered

as a major precursor for SiCN fibers, although it is unspinnable if used pure, due to its unfavorable physical properties (low viscosity) and chemical structure (cyclic rather than linear structure). The introduction of PAA to the silazane to create a hybrid precursor remarkably improved spinnability of the silazane and should be widely applicable to other unspinnable PDC preceramic polymers. Investigations on structural and compositional development of the fibers were mainly conducted via Raman spectroscopy, Fourier-Transform InfraRed spectroscopy (FTIR), Scanning Electron Microscopy (SEM), X-ray Photoelectron Spectroscopy (XPS), Nuclear Magnetic Resonance (NMR) and ThermoGravimetric Analysis (TGA) to determine spinnability, free carbon content, crosslinking and pyrolysis behavior of the fibers, respectively.

(ICACC-S18-P113-2020) Preparation and characterization of electrospun SiOC ceramic fiber mat from cyclic siloxane and organic spinning agent

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SiOC ceramic fiber mats are synthesized via pyrolyzing electrospun hybrid fiber mat from cyclic siloxane oligomer and organic spinning agent. Electrospun fiber mats are crosslinked at lower temperature in air then pyrolyzed at high temperature in Ar. The products are soft and flexible after pyrolysis and no obvious wrapping observed. Various charateriazations are performed, such as Raman, FTIR, NMR, XPS and tensile test.

(ICACC-S18-P114-2020) Flash spark plasma sintering of pure TiB_2

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A Spark Plasma Sintering (SPS) furnace was used to Flash-Sinter (FS) pure titanium diboride (TiB_2) powder. A pre-sintering green body (Ø= 20 mm, relative density 60%) was used for the Flash-SPS using a dieless configuration with current passing entirely across the sample. The results show that the samples were densified in very short time (< 60 seconds) up to 90-95% of theoretical density. TiB_2 obtained by Flash-SPS was characterized using XRD and SEM analysis in order to quantify texturization induced by the FSPS hot forging effect. Mechanical properties (Vickers hardness and elastic modulus) were measure in a direction parallel and transverse to the FSPS load. FSPS densification kinetics was compared with data available in the literature confirming enhanced plasticity promoted by the high temperature.

(ICACC-S18-P115-2020) High Temperature Testing of the Potassium Vapor Resistance of Ceramic Components in Magnetohydrodynamic Generator Systems

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Material selection for magnetohydrodynamic (MHD) generators is particularly difficult due to the high-temperature, high-velocity, potassium-seeded gas flow environment present in the channel. One of the most challenging material requirements is chemical resistance to potassium vapor exposure at high temperatures in the MHD channel. An ASTM test method for metal vapor corrosion of glass kiln refractories was adapted in order to evaluate potassium vapor reactions with four candidate MHD channel materials (MgO, Al_2O_3, YSZ, and CeO_2). Ceramic coupons were evaluated for their high temperature stability and potassium vapor resistance. Analysis of the results allowed the identification of the important failure mechanisms due to potassium vapor exposure which provides new insights for screening potential high-temperature ceramic insulators and conductors for MHD applications.

(ICACC-S18-P116-2020) Intrinsic Mechanical Properties of Zirconium Carbide Ceramics

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Zirconium carbide (ZrC_x) was synthesized by carbothermally reducing zirconia and carbon black in an effort to prepare phase pure ZrC_x and determine its intrinsic properties. The prepared ZrC_x had hafnium content in the range of 0.01 to 0.04 at% with known oxygen and nitrogen impurities. X-ray diffraction analysis indicated that the reacted power was nominally phase pure and Raman spectroscopy identified the ZrC_x as sub-stoichiometric. Powders were prepared with varying carbon stoichiometry and then hot pressed at a temperature of 2150°C at a 32 MPa applied pressure. ZrC_x compositions having a relative density of at least 95% were machined to produce specimens for mechanical property measurements. Mechanical properties were measured including Vickers hardness, elastic modulus, and fracture toughness at room temperature, along with flexural strength from room temperature to 2000°C.

(ICACC-S18-P118-2020) Effect of Preferentially Oriented GNPs on Spark Plasma Sintered Advanced Ceramics

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Graphene nano-platelets are considered, since some years now, as an attractive reinforcement for advanced ceramic materials. Indeed, GNPs present several advantages in terms of process ability while enabling improvement of the mechanical behavior of the material of selected functional transport properties. The present study reports on the processing and performances of GNPs reinforced alumina based and ZrB_2 based ceramic matrices. The effect of the GNPs content under 5%wt on both the densification behavior and microstructural development has been determined. The influence on the resulting properties of the extent of the preferential orientation of the GNPs within the matrix has been assessed. This degree of orientation was tailored by the specific conditioning approach of the GNPs/powder mixture and specific extrusion method used to pre-form the compact prior its densification. Concurrently, care was taken to achieve optimum dispersion of the nano-fillers within the matrices. The densification has been performed by SPS, the high heating rates, lower maximum temperature and reduced holding times being favorable to guarantee the integrity of the carbon-based nano-fillers. The evolution of mechanical properties like the hardness, elastic modulus and fracture toughness in function of the GNPs content is discussed. The evaluation of the electrical conductivity and its anisotropy is reported.

(ICACC-FS4-P119-2020) Luminescent film for asphalt roads using polystyrene waste combined with strontium aluminate

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The intensive use polymers has created a tremendous negative impact worldwide with the poor use of their derived wastes. On the other hand, ternary roads, particularly in developing countries demand new alternatives for artificial illumination, as the investment in regular electrical light sometimes is limited because some are isolated with low traffic and therefore less used. This study is about the development of a luminescent film using strontium aluminate in combination with a polymeric matrix made of recycled polystyrene, which is not only beneficial because of the solid waste utilization, but also because the luminescent ceramic particles reduce the electric

energy consumption and thus contribute to a clean environment as well. These materials have been characterized with electron scanning microscopy, rheology, adhesion and photoluminescence tests.

(ICACC-FS4-P120-2020) Incorporation of Civil Construction Waste (CCW) for the production of structural blocks of red ceramics

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The construction industry has been growing year after year all over the world, and in addition to this growth has been increasing its contribution in environmental pollution whether in the production of raw material or in the generation of large amounts of solid waste. The construction residue (CCW) presents great potential for reuse in cement and ceramics, but research related to its application in ceramics is generally restricted to blocks for sealing purposes. Therefore, the objective of this work is the evaluation of the potential of incorporation of construction waste for the production of ceramic blocks for structural purposes. The CCW was collected from a company which performs its processing. After that, eight specimens were prepared at 20 MPa for each firing temperature (850, 950 and 1050 ºC), with incorporations of 0, 10, 20, 30 and 40%. The physical and mechanical properties of ceramic specimens such as linear retraction, water absorption and bending stress at all temperatures were evaluated. The results showed that the addition of CCW caused an improvement in the mechanical resistance and reduction of the water absorption, as the percentage of incorporation was increased, which is positive, as far as the firing temperatures were concerned, a greater need of to meet the parameters stipulated by the Brazilian standard.

(ICACC-FS4-P121-2020) Colloidal Technique for Applying CFRPP to Multi-Material Structure

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As a method to increase the mechanical properties of carbon fiber reinforced thermoplastic (CFRTP), we focused on a laminated structure (millefeuille structure) composed of a soft and a hard materials. Producing CFRTP with resin films and CFs, PP film and PA6 film are alternately laminated, but the interfacial properties between CF and resin, PP and PA6, were not enough to prepare high mechanical properties of CFRTP. So, we aim to improve their surface adhesions by adsorbing the polymer particles on the surface of CF, which play a role like an adhesive. And also, to apply CFRTP to multi-material structure, the voltage was applied to the metal in the colloidal solution to adsorb the polymer particles on the metal surface. Thereby, the metal surfaces are modified to obtain fine defects and adhesive to the resin in one step for short time.

(ICACC-FS4-P122-2020) Physical, mechanical and microstructural characterization of clay bricks containing bauxite tailings

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Worldwide, the mining activity has great economic and social relevance, but also causes large environmental impacts. It is necessary to adapt this productive chain to the sustainable development proposed in the last decades. An alternative is the reuse of by-products, such as ore tailings, to produce new building materials. Thus, the present work aimed at the application of bauxite tailings (red mud) in the manufacture of construction clay bricks. A mixture design of experiments was developed, using the following components: the red mud and two clayey materials (gray clay and yellow clay). Ten mixtures were produced, and cylindrical specimens were formed by uniaxial pressing. After firing at 950°C, the ceramic pieces were characterized for firing linear shrinkage, bulk density, apparent porosity, water absorption and compressive strength. In addition, the microstructure was also verified. The results showed that the red mud contributed to the increase of apparent porosity and water absorption. According to the standard specifications for water absorption and mechanical strength, the optimal composition was defined: 30.8% of tailings, 40.6% gray clay and 28.6% yellow clay. This study showed that the mixture design of experiments is a good tool for the formulation of new products, and the use of red mud is environmentally and technically viable for the production of red ceramic.

(ICACC-FS4-P123-2020) Recycling of Sewage Sludge into Clay Bricks

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This work has as its objective to evaluate the use of sewage sludge from wastewater treatment plant into heavy clay ceramic body. Compositions were prepared with amounts of 0 and 5% wt.% of sludge incorporated into the clay body. Rectangular specimens were obtained by 20 MPa pressure molding and then fired at temperatures ranging from 800 to 1100ºC in a laboratory furnace. Characterization of the waste was done by X-ray diffraction, X-ray-fluorescence, sedimentation and sieving techniques, scanning electron microscopy, differential scanning calorimetry and thermogravimetric analysis. Ceramic properties related to the water absorption, linear shrinkage and flexural rupture strength were determined. The results indicated that the incorporation of the sludge into clay bricks brings an energy saving during the firing stage of the ceramic. With respect to the physical and mechanical properties, the waste slightly decreases the water absorption and increases the mechanical strength of the fired ceramic. Finely, the temperature exercises a strong influence on the evaluated properties.

(ICACC-FS4-P124-2020) Removal of phosphate and fluoride ions in waste water by using waste gypsum board and usage of collected fluorapatite (FAp) for adsorbent of ammonia gas

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Gypsum board is widely used for wall in buildings by applying its good fire resistance and usability. However, management of waste gypsum board from demolition process is one of the serious environmental problems because its disposal amounts will increase year by year. In this study, we attempted usage of waste gypsum board as calcium resources for waste water treatment. We appeared that gypsum powder was useful for removal of phosphate ions in waste water and obtain dicalcium phosphate dihydrate (DCPD) or hydroxyapatite (HAp). DCPD and HAp were also useful for removal of fluoride ions in waste water efficiently. From this process, nanoscaled fluorapatite (FAp) was obtained. The collected FAp shown extreame adsorption property on ammonia gas. From these results, usage of gypsum was useful for cascade recycling for phosphate and fluoride ions in waste water and adsorption of ammonia gas without any virgin resources.

(ICACC-FS4-P125-2020) Effect of fluorapatite (FAp) on reactivity of dicalcium phosphate dihydrate (DCPD) with fluoride ions in the environments

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Dicalcium phosphate dihydrate (DCPD) reacts with fluoride ions and forms stable fluorapatite (FAp). In our previous studies, we appeared that this reaction required induction of nano-scaled precursor particles on the DCPD particles and the nano-scaled precursor consisted of hydroxyapatite (HAp)-like calcium phosphate. This induction process connect to a few hours of lag time that is no change of fluoride ions concentration. The lag time is serious problems for waste water treatment because this is prolonged by various coexistence ions such as magnesium, cadmium and fluoride. Therefore, decrease of the lag time is one of the important approaches on improvement of reactivity of DCPD with fluoride ions. In this study, we attempted to develop functional material consisting apatite-DCPD hybrid for removal fluoride ions in waste water. We appeared that FAp nano-scaled particles was induced on the DCPD particles by using solution containing only calcium, phosphate and fluoride ions in weak acidic pH. Amount of FAp affected to decrease the lag time. We also estimated suitable amount of FAp for improving DCPD reaction with fluoride ions.

(ICACC-FS4-P126-2020) A study of local diatomaceous earth (Kapatagan, Northern Mindanao Philippines) as an additive for the Commercial Ceramic Wall

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In this study, locally available diatomaceous earth material from Kapatagan, Lanao del Norte Philippines was utilized as an additive to the standard ternary mixtures of commercial wall tiles. The physical properties of Kapatagan Diatomaceous earth (KDE) revealed 52.49% water absorption, 19.28% fired shrinkage, 0.92 bulk density, and 48.79% apparent porosity. Based on the surface morphology, it shows the tangled porous particles of KDE called diatoms and the coalescence behavior of these particles was observed after sintering. X-ray diffraction pattern revealed that muscovite and quartz are evident in the raw KDE and the appearance of cristobalite after sintering to 1150C. The incorporation of 5% KDE which was sintered at 1235C increases the water absorption up to 0.48% along with the apparent porosity up to 0.80% and decreases the fired weight down to 66.92 g. Surface morphology showed a modification of pores such as interparticle porosity which is responsible for the slight decrease in Modulus of Rupture (MOR) down to 6.03 MPa. Based on the sintered experimental commercial wall tiles with 5% KDE, the X-ray diffraction patterned showed three major phases such as quartz, mullite, and cristobalite. For comparison, the commercial ceramic wall tile was fabricated and investigated.

(ICACC-FS4-P127-2020) Evaluation of the incorporation of eucalyptus firewood ash on clayey ceramic

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Ashes are a type of solid waste that can cause atmospheric pollution, generating serious health problems in the population and contaminating the soil and the underground water bed. A technological alternative to reduce the environmental impact caused by undiscriminated throw of ashes is its incorporation into clayey ceramic. This work has for its objective to characterize physical and mechanical properties of clayey ceramic incorporated with eucalyptus firewood. Mixtures of kaolinitic clay from the municipal area of Campos of Goytacazes, State of Rio de Janeiro, Brazil, were prepared with additions of 0, 5, 10 and 20% in weight of ashes from eucalyptus firewood. Specimens were 20 MPa uniaxially press-molded and fired at 900°C. The firing properties evaluated were diametrical shrinkage, water absorption and mechanical strength by diametrical compression. The results showed that the incorporation of 10% wt. of firewood ash enhanced the properties of the red ceramic. The plasticity was optimised, the water absorption was decreased and the mechanical strength was increased.

(ICACC-FS4-P128-2020) Effect of molding condition on Thermoplastic Polyimide Impregnation behavior to Plain Woven Carbon Fabrics

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During molding of carbon fiber reinforced thermoplastics (CFRTP), sufficient resin impregnation to fiber yarns is important. On the other hand higher viscosity of molten thermoplastics inhibits resin impregnation to the interspace among fibers. The purpose of this study was to clarify the relation among molding conditions and resin impregnation to plain woven carbon fabrics experimentally and analytically. In this study, CFRTPs were produced by Micro-Braiding, Film Stacking and Powder method. In addition resin impregnation behavior was modeled based on Darcy's law. As a result, analytical resin impregnation prediction showed good agreements with the experimental results. In addition, the void content could be greatly reduced by pressurizing cooling.

(ICACC-FS4-P129-2020) Reuse of iron ore tailing, from the disaster involving the Fundão's dam rupture, in the production of clay bricks

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1. Federal University of Viçosa, Civil Engineering, Brazil
2. State University of Northest Fluminense, LAMAV, Brazil
3. Federal University of Viçosa, Soil Department, Brazil
4. Federal Fluminense University, Department of Agricultural Engineering and Environment, Brazil

The intent of this research was to study a new alternative for the recycling of iron ore tailing, aiming at its application in construction clay bricks. For such purpose, a simplex design of experiments was developed, using three components: the iron ore tailing and two clayey materials. The raw materials were subjected to physical, chemical, mineralogical and morphological characterization. Next, the mixtures defined by the experimental design were prepared, and cylindrical samples were produced by press moulding. After the firing at 850°C, 950°C and 1050°C, the specimens were characterized in terms of firing linear shrinkage, bulk density, apparent porosity, water absorption and compressive strength. The obtained results show that the incorporation of tailing in ceramic masses decreases the linear shrinkage and mechanical strength, and increases the apparent porosity and water absorption. Then, the optimal composition was determined, that contains 29,1% (by mass) of iron ore tailing

and meets the standard specifications (national and international). This research showed the viability of manufacturing construction clay bricks with good technical quality, presenting advantages from environmental, economic and technical points of view.

Thursday, January 30, 2020

4th Pacific Rim Engineering Ceramics Summit

Current trends: Energy Issues

Room: Coquina Salon E
Session Chairs: Junichi Tatami, Yokohama National University; Tohru Suzuki, National Institute for Materials Science

8:30 AM

(ICACC-PACRIM-027-2020) Nano-structural Engineering and Characterization of Electrode Materials for High-Areal-Capacity and Stable Lithium-Sulfur Batteries (Invited)

D. Kim*[1]

1. Korea Advanced Institute of Science and Engineering (KAIST), Dept. of Mater Sci & Eng, Republic of Korea

The development of high-energy-density batteries that can alternate the state-of-the-art Li-ion batteries is invariably worthwhile to meet the ever demand of energy requirement for electric transportation and grid energy storage system. Lithium-sulfur (Li-S) system renders a settlement to meet the ever demand of energy thanks to the high theoretical capacity (1,675 mAh g^{-1}) and gravimetric energy density (2,500 Wh kg^{-1}). Despite the energy benefit of Li-S, the commercialization of Li-S batteries is hindered by several obstacles; low areal capacity (<3 mAh cm^{-2}) of cathode stemmed from a low electrical conductivity of elemental sulfur, dissolution of lithium polysulfide intermediates in electrolyte, and dendritic growth of lithium metal anode bringing short-circuit current. These issues can be handled by nano-engineering of electrode materials which promotes the electrochemical reaction of Li-S batteries. Here we introduce the strategies to develop the performance of Li-S by porous carbon matrices for both cathode and anode with high areal capacity and suppressed lithium dendrite growth, respectively. A functional separator to surmount polysulfide dissolution is also discussed. In this presentation, we try our best to describing the Li-S to bear on, with recommendation of a suitable ways for high performance Li-S batteries.

9:00 AM

(ICACC-PACRIM-028-2020) A new approach to observe the motion of oxygen ions in solid oxide electrolytes (Invited)

W. Pan*[1]

1. Tsinghua University, State key lab of new ceramics and fine processing, China

Measurement of diffusion coefficient in solids would greatly increase the capacity in proper selection of candidate for solid electrolytes widely applied in energy conversion systems like solid oxide fuel cells and lithium ion batteries. Here, we present a facile approach to monitor the ion diffusivity in solids. An inhomogeneous distribution of ions in a closed system was induced at first via an external field force, and the spontaneous migration of ions can be created by the inner chemical driving force when removing the external field. Based on the recording of spatial and time dependence of ion distribution using a laser confocal micro Raman spectroscopy, and fitting with a proper model, the diffusion coefficient can thus be acquired. By altering the external force and detecting means according to different materials, this approach largely overcomes the limitations in operability and specificity of existing analysis methods.

9:30 AM

(ICACC-PACRIM-029-2020) Enhancement of thermoelectric performance by in-situ generation of defect structures (Invited)

K. Lee*[1]

1. Yonsei University, Republic of Korea

Thermoelectric is a technology for power generation and solid-state cooling by convergen between thermal and electric energy. To expand the applications of thermoelectric technology into power generation and domestic cooling, development of high-performance thermoelectric materials is required. Recently, the performance of commercial thermoelectric materials including Bi-Te-, skutterudite-, and half-Heusler-based alloys has been significantly enhanced through non-equilibrium processing technologies for in-situ generation of defect structures to simultaneously improve the electronic and thermal transport properties. We provide the approaches for the generation of defect structures which is effective to control both the electronic and thermal transport properties of conventional thermoelectric materials, and our recent progresses in the synthesis with defect structures is summarized.

10:20 AM

(ICACC-PACRIM-030-2020) Role of ceramic research in emerging energy technologies (Invited)

C. Balazsi*[1]; K. Balazsi[2]

1. HAS Centre for Energy Research, Hungary
2. Centre for Energy Research HAS, Thin Film Physics, Hungary

The world's population is expected to reach 10 billion by 2050 based on recent growth trends, an increase of 25 percent above current values. This increase is already challenging production and consumption patterns worldwide as industrialized and developing countries pursue economic growth under increasingly global stresses. The global energy consumption is expected to continue to grow primarily in developing countries. Their share of global energy consumption will grow from approximately 35% in 1990 to 60% in 2050. At European level, total energy demand is expected to increase 35% by 2030 compared to today. Energy supply security has for geopolitical reasons become an increasing concern especially in the US and the EU. Several renewable technologies are being developed and deployed in many countries, such as wind or solar energy in Hungary. However, at a global level renewable energy technologies contribute little to today's energy supply, and it will take a couple of decades before major contributions. At that time, we expect wind, biomass and solar to be strong candidates that could, even in a competitive market, change the energy supply towards a more environmentally benign system. In some of the scenarios presented in the lecture, the ceramic research, development and innovation for the next generation of nuclear power plants plays an important role in the years ahead, and later on even related to fusion power.

10:50 AM

(ICACC-PACRIM-031-2020) Transparent/translucent $MgAl_2O_4$ and MgAlON–based phosphors for solid state lighting and photocatalytic applications (Invited)

Z. Lences*[1]; M. Radwan[1]; P. Petriskova[1]; A. Czimerova[1]; P. Sajgalik[1]

1. Institute of Inorganic Chemistry, Slovak Academy of Sciences, Ceramic Department, Slovakia

Preparation and characterisation of transparent $MgAl_2O_4$ and translucent MgAlON spinel ceramics doped with lanthanides will be presented. A systematic study was performed to develop a processing methodology for elimination of microstructural defects in sintered samples and improving optical transparency. Soft spinel granules with improved compressibility were prepared from the starting powders by freeze granulation. Green pellets were CIP-ed by 400 MPa pressure. Spinel specimens with relative densities >99.9% were prepared by pressureless-sintering and subsequent HIP-ing at 1550 °C for 5 h in 200 MPa Ar gas. The MgAlON ceramics were sintered

at 1800 °C. The optical real in-line transmittance (RIT) of polished $MgAl_2O_4$ specimens was 95% of the theoretical. A series of translucent spinel MgAlON phosphors doped with different cations were prepared. The colors of emissions depending on the dopant were as follows: Eu – blue (460 nm), Ce – yellowish (570 nm), Yb – red (665 nm) and Er – red emission (637 nm, 684 nm, 745 nm). Some of the phosphors excited by green light emitted dark-red light (λ_{em} = 715-720 nm). The transparent spinel ceramics combined with titania will be tested for photocatalytic applications. This work was supported by APVV-14-0385 and VEGA2/0164/18 projects.

11:20 AM

(ICACC-PACRIM-032-2020) Design of Novel Sustainable Materials by Incorporating Principles of Circular Economy (Invited)

S. Gupta*[1]

1. University of North Dakota, Mechanical Engineering, USA

Circular Economy model is based on 3R principles of reducing, reusing, and recycling. Unlike traditional linear model, this model is healing and sustainable as it strives towards the use of renewables, eliminating the link between consumption and economy, and phasing out waste. Based on these principles, it is critical to design novel material systems from renewable sources like biomass. As a background, biomass is an important source for renewable materials where cellulose, hemicellulose, and lignin are the predominant constituents of the biomass. Traditionally, cellulose and hemicellulose are used as precursors for producing commercial materials like paper, sugar, and biofuels. Comparatively, the valorization rate of lignin is less than 2%. In this presentation, I will present some of the recent developments on fabricating green multifunctional materials by using benign and underutilized precursors like lignin and various agricultural waste like wheat straw, sugar beet etc for manufacturing bioplastics, solid lubricants, and porous scaffolds.

Current trends: Powder Processing

Room: Coquina Salon E
Session Chairs: Surojit Gupta, University of North Dakota; Michael Halbig, NASA Glenn Research Center

1:30 PM

(ICACC-PACRIM-033-2020) Room temperature densification of nitride particle-dispesed MgO ceramics (Invited)

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

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

For high-power solid-state lightening, phosphor components with high thermal conductivity is needed instead of phosphor particle dispersed resin. Although phosphor-in-glass has been developed, its thermal conductivity is limited. Furthermore, there is a possibility of degradation of phosphor by firing process. In this study, nitride particle dispersed MgO ceramics with high thermal conductivity was fabricated by room temperature densification process. Commercially available MgO powder and Ca-α SiAlON:Eu^{2+}, β-SiAlON:Eu2+, or $CaAlSiN_3$:Eu^{2+} were mixed, followed by uniaxial pressing and cold isostatic pressing. A small quantity of water was added to the green body. After packing the green body in a plastic bag under vacuum, isostatic pressure was applied at room temperature. The pressure was varied from 200 to 1000MPa. The relative density of the product was over 90%, though that was about 60% without any water. The fracture surface was faceted, which is like the microstructure of the product prepared by high temperature firing. The phase presents were MgO and a small amount of $Mg(OH)_2$. The luminescence property of the sintered body was the same as that of the raw material. Thermal conductivity depended on the raw powders and processing, and the maximum thermal conductivity was 8W/mK, which was about 100 times as high as conventional resin composites.

2:00 PM

(ICACC-PACRIM-034-2020) Exploring New Additive Compositions for Sintering of Silicon Carbide Ceramics at Low Temperatures (Invited)

Y. Kim*[1]; S. Kultayeva[1]

1. University of Seoul, Dept. of Materials Science & Engineering, Republic of Korea

The role of additives in the sintering of SiC ceramics can be regarded as not only densification aids, but also key elements for achieving high-performance properties because the electrical, thermal, and mechanical properties of SiC ceramics are influenced by the composition of additives as well as the microstructure of the resulting ceramics. Considering the decisive influence of the additive composition on the development of microstructure, grain boundary structure, and the resulting electrical, thermal, and mechanical properties of LPS-SiC ceramics, the composition of sintering additives should be carefully selected to achieve some desired properties for specific applications. In this presentation, recent efforts for finding new additive compositions for achieving high sintered density at low temperatures (≤1850 °C) with excellent mechanical strength and high thermal conductivity will be presented. A few successful examples of new ternary and quaternary additive systems including the Y_2O_3-Sc_2O_3-MgO, AlN-Y_2O_3-Sc_2O_3-MgO, and A_2O_3-Y_2O_3-MgO-CaO systems will be introduced. The Y_2O_3–Sc_2O_3–MgO additive system was very effective in achieving high-strength SiC ceramics (>1 GPa) and the AlN-Y_2O_3-Sc_2O_3-MgO, and A_2O_3-Y_2O_3-MgO-CaO quaternary additives are suitable for processing of fully ceramic microencapsulated fuels without an applied pressure.

2:30 PM

(ICACC-PACRIM-035-2020) Orientation control in ceramics by external fields (Invited)

T. S. Suzuki*[1]

1. National Institute for Materials Science, Ceramics Processing Group, Japan

Tailoring the crystallographic orientation in ceramics is very useful for improving their properties. A magnetic field is shown to be very effective in controlling the crystallographic orientation in bulk ceramics. We reported that the colloidal processing in a strong magnetic field was able to control the crystallographic orientation even in diamagnetic ceramics. When a strong magnetic field is applied to the particles in a stable suspension, the particles were rotated to an angle minimizing the system energy by a magnetic torque generated from the interaction between the magnetic anisotropy and the applied magnetic field. The orientation of the crystal depends on the axis having easy magnetization and one-dimensional orientation can be controlled. Furthermore, multi-axial crystalline orientation in ceramics can be controlled by geometric effect in addition of the magnetic field. The axes of anisometric particles were aligned by geometric effect and the other axis was oriented by a magnetic field, consequently the multi axis orientation was achieved. If Nylon was included as a pore former in slurries, textured ceramics with aligned tubal pore was fabricated after sintering. Highly structure ordered ceramics can be fabricated by magnetic field assisted colloidal processing.

3:20 PM

(ICACC-PACRIM-036-2020) Spark Plasma Sintering of Fine-grained Transparent Ceramics (Invited)

B. Kim*[1]

1. National Institute for Materials Science, Fine-Grained Refractory Materials Group, Japan

Highly dense and fine microstructures are essential to improve mechanical and optical properties, particularly for non-cubic ceramics. In this study, our efforts are introduced for achieving transparent ceramics with fine microstructures by using the

spark plasma sintering (SPS) technique. For fine grain sizes, low-temperature sintering of fine powder is indispensable. The sintering temperature was lowered using SiC mold. Since the electrical conductivity of the SiC mold is lower than that of conventional graphite, the current effect was more activated to enhance the densification. At low temperatures, however, unexpected grain growth can occur. The unusual growth behavior was examined in detail during low-temperature sintering of alumina, and was explained qualitatively using a concept of dynamic grain growth. By controlling sintering conditions, we suppressed the unusual grain growth, and fabricated such fine-grained transparent ceramics as Al_2O_3, Y_2O_3, AlN, HAP:$Ca_{10}(PO_4)_6(OH)_2$ and FAP:$Ca_{10}(PO_4)_6F_2$. Particularly, the HAP and FAP ceramics show excellent optical properties. In the presentation, the current effect on the suppression of grain growth during SPS of Y_2O_3 is also discussed.

3:50 PM

(ICACC-PACRIM-037-2020) Fabrication of transparent γ-AlON and its plasma etching properties in comparison with other transparent ceramics (Invited)

D. Yoon*[1]

1. Yeungnam University, School of Materials Science and Engineering, Republic of Korea

Transparent polycrystalline aluminum oxynitride (γ-AlON) was fabricated by the pressureless two-step sintering of α-Al_2O_3 and AlN after adding a small amount of MgO and Y_2O_3. The process was based on two assumptions. The first was the utilization of AlN-deficient non-stoichiometric composition to increase the cationic vacancies and the second was the selection of the 1st step sintering temperature that suppresses the formation of γ-AlON phase to achieve a high density after the 2nd sintering step. The 1st and 2nd sintering steps were performed at 1610 – 1650 and 1940 – 1990°C, respectively, for 10 hours in a 2.5 atmospheric nitrogen pressure, and the optimal sintering conditions were determined. The fabricated γ-AlON showed a mean grain size of 164 – 248 μm without the presence of significant scattering centers, where the sample prepared using an optimal condition revealed a very high transmittance of 84.7% along with comparable mechanical properties. Moreover, fluorocarbon plasma etching properties of the fabricated γ-AlON were examined for up to 3 hours under an incident plasma power of 500 W compared with other transparent ceramics, such as sapphire, Mg-spinel, and Y_2O_3.

4:20 PM

(ICACC-PACRIM-038-2020) Effect of DC Current on High Temperature Flow Behavior of Polycrystalline Zirconia Ceramics (Invited)

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

1. National Institute for Materials Science (NIMS), Japan
2. The University of Tokyo, Department of Materials Science, Japan

Flash sintering phenomena, which occurs by applying DC current directly to ceramic powder compacts, can succeed to lower the sintering temperature of several ceramic powders. However, the effect of electric field is still unclear. In order to clarify the effect of electric current on high temperature phenomena, therefore, the present study was carried out to examine the tensile behavior of polycrystalline zirconia ceramics under the several temperature and electric field/current conditions. By applying the electric power higher than a critical value E_c, the flash event similar to that of powder sintering occurs even in dense zirconia ceramics. For lower than E_c, the applied electric current increases sample temperature depending on the applied value, but does not enhance the rate of deformation. For higher than E_c, on the other hand, the electric current enhances the rate of the deformation to about several times as compared with that of without current conditions. The enhanced deformation cannot be interpreted only by the increment of sample temperatures. After the deformation under the electric current conditions, the tested sample shows slight gray color even in air, suggesting that the enhanced deformation would be related to oxygen vacancy formation.

4:50 PM

(ICACC-PACRIM-039-2020) Improved thermal conductivity of silicon nitride by intentional oxygen removal (Invited)

H. Kim*[1]; Y. Li[1]; Y. Park[1]; J. Ko[2]; M. Kim[1]; H. Kim[1]

1. Korea Institute of Materials Science, Republic of Korea
2. KIMS (Korea Institute of Materials Science), Republic of Korea

Oxygen is recognized as the most detrimental impurity to the thermal conductivity of Si_3N_4, a promising substrate material for high-power electronic devices. In this study, the ceramic processing was adjusted to allow intentional oxygen removal. Firstly, graphite powder was added to the BN powder bed. Owing to the strong reducibility, the powder bed could significantly facilitate the removal of SiO_2 from Si_3N_4 during nitridation and sintering process, resulting in altered chemistry of secondary phase, larger β-Si_3N_4 grains, and lower lattice oxygen. Secondly, novel sintering additive, $Y_2Si_4N_6C$, is applied to sinter Si_3N_4. In comparison to Y_2O_3 additive, $Y_2Si_4N_6C$ induced a higher nitrogen/oxygen ratio in the secondary phase, resulting in enlarged grains, reduced lattice oxygen content, increased Si_3N_4-Si_3N_4 contiguity and more crystallized intergranular phase. Lastly, a small amount of carbon is used to enhance the properties. The carbothermal reduction process significantly reduced the oxygen content and increased the N/O ratio of intergranular secondary phase. Those promoted the coarser elongated grains, lower lattice oxygen content, tighter Si_3N_4-Si_3N_4 interfaces and more devitrified intergranular phase due to the further carbothermal reduction of oxynitride secondary phase. Consequently, the intentional removal of oxygen enabled Si_3N_4 ceramic to gain a significant increase over 25% upto 40%.

FS1: Bio-inspired Processing of Advanced Materials

Bio-inspired Processing of Ceramics I

Room: Coquina Salon H
Session Chair: Florian Bouville, ETH Zürich

8:30 AM

(ICACC-FS1-001-2020) Bioinspired advanced ceramics from freeze casting and energized fields (Invited)

S. E. Naleway*[1]; I. Nelson[1]; T. Yin[1]; D. Porter[1]; P. Wadsworth[1]; M. Mroz[1]

1. University of Utah, Department of Mechanical Engineering, USA

Freeze casting is a bioinspired technique for the fabrication of tailored, porous ceramic materials with structuring down to the nanoscale. Mimetic of the growth of mammalian bone and other biomaterials where biopolymers template the deposit of biominerals to create complex composites, freeze casting employs a template of growing ice crystals to create a complex porous microstructure in any ceramic. We propose that this bioinspired technique can be controlled through either intrinsic (those that modify from within by altering the constituents) or extrinsic (those that apply external forces or templates) means. Through these classifications, examples of extrinsic (through energized external fields) freeze cast, bioinspired structures will be discussed with a focus on providing advanced control of the final material structure and properties. Applications in energy and filtration technologies will be discussed.

9:00 AM

(ICACC-FS1-002-2020) Pore size and morphology tunability through coarsening during freeze casting (Invited)

N. Arai*[1]; P. Voorhees[3]; K. Faber[2]

1. California Institute of Technology, Materials science, USA
2. California Institute of Technology, USA
3. Northwestern University, Materials Science and Engineering, USA

Freeze casting is a porous ceramic processing method which can be used to control pore size, pore morphology, and directionality. Conventionally, the pore size and morphology is tuned by parameters such as solids loading, solvent choice, and freezing front velocity. Moreover, several freeze-casting studies have employed solidification techniques such as grain selection processes and Bridgman directional freezing methods, which enable additional control of porosity. While most of the studies focus on the freezing step in freeze casting, no studies have focused on morphological evolution of the frozen crystals over time, namely coarsening. Here, isothermal coarsening of dendritic structures is employed as a means to tailor pore size and morphology in freeze casting of preceramic polymers. The effects of coarsening temperature and coarsening time on dendritic pores are investigated and two notable results are presented. First, the morphological transition from dendritic structure to honeycomb structure will be discussed. Second, the change in the pore size and pore size distribution as a function of coarsening time through use of mercury intrusion porosimetry will be reviewed. It is demonstrated that the cube of secondary pore size increases linearly with time, which is same phenomena found in solidification of alloys.

9:30 AM

(ICACC-FS1-003-2020) Biotemplated carbon electrodes: From local atomic structure to electrochemical properties (Invited)

J. Ramirez-Rico*[1]; A. Gomez-Martin[1]; J. M. Fernandez[1]

1. Universidad de Sevilla, Spain

Carbon-based electrodes have received considerable attention due to their low cost, tailorable microstructure, and the possibility of processing them by direct carbonization of naturally-available biomass resources. In energy storage applications, several material parameters need to be optimized, such as surface area, pore size or crystallinity, requiring understanding of structural evolution during carbonization and heat treatment, as well as the effect graphitization catalysts will have on the final structure. For instance, hard carbon represents the material of choice for sodium-ion batteries, whereas highly crystalline carbon is required in the case of lithium-ion batteries. The atomic pair distribution function is a very powerful tool for the analysis of materials with short-range atomic ordering. In this work we use high energy synchrotron x-rays and the PDF formalism to elucidate the correlation between carbonization parameters, structural evolution and electrochemical properties of biomass-derived carbon materials when used as electrodes in supercapacitors, sodium-ion and lithium-ion batteries. We find that local atomic structure can be tuned by adequately controlling carbonization and graphitization. Through the use of graphitization catalysts, highly crystalline carbon materials can be obtained from biomass sources, converting waste into a high added-value product.

Bio-inspired Processing of Ceramics II

Room: Coquina Salon H

Session Chair: Joaquin Ramirez-Rico, Universidad de Sevilla

10:20 AM

(ICACC-FS1-004-2020) Designing Materials from Their Solute State: The Pre-nucleation Cluster Pathway and the Use of Non-Classical Nucleation Theory in Materials Chemistry (Invited)

D. Gebauer*[1]

1. Leibniz University of Hannover, Institute of Inorganic Chemistry, Germany

The term "non-classical nucleation" refers to formation pathways of materials where larger species than monomeric chemical constituents play crucial roles. Indeed, so-called prenucleation clusters (PNCs) can lie on alternative pathways to phase separation, where the very event of demixing is primarily based on not the sizes of the species forming, as in the classical view, but their dynamics. Here, we provide an overview of recent developments in the mechanistic understanding of non-classical nucleation, showing the potential to use these notions for the target-oriented design of inorganic functional (hybrid) materials. The properties of solute PNCs are linked to those of the nascent solid, and can be influenced and controlled throughout the early stages of precipitation. The use of additives for controlling, e.g., polymorphism can therby be quantitatively explained, while the stabilization of liquid intermediates for the structuration of materials from the nano- to the mesoscale is especially appealing from a materials chemistry perspective. Non-classical nucleation theory provides a holistic framework for an improved understanding of sometimes surprising and odd, non-classical observations, which can be exploited for the development of new synthetic routes to advanced materials with new applications.

10:50 AM

(ICACC-FS1-005-2020) Multi-time and length-scale analysis of calcium carbonate room temperature sintering (Invited)

M. Haug[1]; A. Studart[1]; F. Bouville*[2]

1. ETH Zürich, Complex Materials, United Kingdom
2. Imperial College London, Department of Materials, United Kingdom

Sintering is an ubiquitous step in all ceramic processing to go from a porous body to a dense ceramic. We use extreme heat to drive this densification, but also high pressure or current. But dense ceramic materials in the Earth's upper crust can be formed at mild pressures and nearly ambient temperature. This represents a tremendous reduction in energy for densification compared to technical ceramic sintering. Inspired by this geological phenomenon, we demonstrated that small amounts of water and pressures up to 500 MPa can densify calcium carbonate compacts up to 85% relative density within only a few minutes. While the densification process in geological carbonates is presumably based on dissolution-diffusion-precipitation, the mechanisms underlying the densification of synthetic carbonate nanoparticles remain unknown. Even more so because other materials sintered using hydrothermal cold sintering shows different densification behaviour. We used a two-pronged approach to understand the mechanism behind this atypical densification: changing macroscale processing parameters, but also combining in situhigh resolution synchrotron tomography with multi-scale indentation technique. Our findings provide a new perspective into the cold compaction of nanopowders with water and open promising routes for the manufacturing of CO_2-based structural materials at mild processing conditions.

11:20 AM

(ICACC-FS1-006-2020) Bioinspired functional composites: Transparent, strong, and tough

T. Magrini*[1]; F. Bouville[2]; A. Lauria[1]; H. Le Ferrand[1]; T. Niebel[1]; A. Studart[1]

1. ETH Zürich, Department of Materials, Switzerland
2. Imperial College, Department of Materials, United Kingdom

Materials combining optical transparency and mechanical strength are highly demanded for electronic displays, structural windows and in the arts, but the oxide-based glasses currently used in most of these applications suffer from brittle fracture and low crack tolerance. We have developed a simple and upscalable processing route to fabricate bulk transparent composites with a brick-and-mortar architecture inspired by seashells that can effectively slow down the propagation of cracks during fracture of the silica-based material. To achieve optical transparency in this brick-and-mortar microstructure, we infiltrate the percolating network of glass platelets (the bricks) with a refractive-index-matching polymer matrix (the mortar). Mechanical characterization shows that our bioinspired glass composites are up to 3-fold tougher than common glasses, while keeping flexural strengths that are comparable to transparent polymers, silica- and other conventional glasses. The high fracture resistance of the composites arises from the interplay of several extrinsic toughening mechanisms, namely crack deflection, polymer bridging and platelet pullout. In summary, this study demonstrates that implementing biological design principles into glass-based materials at the microscale opens a promising new avenue for the manufacturing of structural bioinspired materials combining antagonistic functional properties.

11:40 AM

(ICACC-FS1-007-2020) Multifunctional metal–ceramic composites with nacre-like architecture

E. Poloni*[1]; F. Bouville[2]; T. Niebel[1]; A. Studart[1]; C. Dreimol[1]

1. ETH Zurich, Complex Materials, Switzerland
2. Imperial College, United Kingdom

Some natural organisms have evolved structures that combine strength and toughness to increase their chance to survive predators and environmental stresses. One of the best examples of a strong and tough natural material is the nacreous layer present in multiple mollusk shells. Over the last decades, material scientists have taken inspiration from the brick-and-mortar architecture of nacre by combining ceramics bricks and polymers mortars using various processes. However, the use of a polymer matrix in nacre-like composites has limited the strength and elastic modulus of the "mortar phase" and thus the achievable toughness. The candidates of choice to make a stronger and stiffer mortar would be metals, but they have been quite difficult to process at the nanometre scale required to fit within the spacing between micron-scale ceramic bricks. To solve this issue, we developed a general processing route to fabricate metal–ceramic multifunctional composites with a nacre-like architecture. Micron-sized ceramic platelets, used as building blocks in such architectures, are coated with the desired metallic phase precursors via a non-aqueous sol–gel synthesis. The control over the composition and the microstructure of the final metallic phase during casting and Spark Plasma Sintering makes it possible for the composites to be tough and strong but also magnetic and/or electrically conductive.

Mechanical Properties of Bio-inspired Ceramics I

Room: Coquina Salon H

Session Chair: Simone Sprio, National Research Council of Italy

1:30 PM

(ICACC-FS1-008-2020) Ultra-tough and impact resistant glasses with bioinspired architectures (Invited)

F. Barthelat*[1]; Z. Yin[2]; F. Hannard[2]

1. University of Colorado, Mechanical Engineering, USA
2. McGill University, Mechanical Engineering, Canada

Glass is a widely used engineering material because of its optical properties, hardness, durability, thermal and chemical stability. However, at ambient temperature glass is brittle with poor impact resistance. Despite improvements in strength (tempering) and impact behavior (lamination), glass components remain by far the weakest structural elements in vehicles, buildings and electronic devices. In this work we have developed a series of transparent, glass-based materials that overcome the inherent brittleness of glass, with designs and architectures inspired from mineralized tissues such as bone, teeth or mollusc shells: (i) Hard and stiff building blocks, (ii) thin, soft and highly deformable interface materials, (iii) precise, near-periodic 3D architectures to induce collective micro-mechanisms over large volumes. We have applied these guidelines to a variety of glass-based materials with various architectures combined with transparent thermoplastic elastomers: Ultra-tough glass with jigsaw-like sutures, cross-ply laminated glasses that can stretch by up to 80%, nacre-like glass panels with superior impact resistance and graceful, quasi-ductile failures. The fabrication methods we used can be applied to the large-scale production of high-performance glasses for a wide range of applications including protective structures, windows, photovoltaic systems, building materials and electronic devices.

2:00 PM

(ICACC-FS1-009-2020) Bioinspired Architected Materials: When geometry enables novel mechanisms and better performance (Invited)

M. Shishehbor[1]; M. Hosseini[1]; J. Rivera[2]; N. Yaraghi[2]; N. Suksangpanya[1]; D. Kisailus[2]; P. Zavattieri*[1]

1. Purdue University, Lyles School of Civil Engineering, USA
2. University of California, Riverside, Chemical and Environmental Engineering, USA

The focus of this work is to understand the role of the inner architecture in naturally-occurring materials through a hypothesis-driven approach that involves experiments, theory, computational modeling and 3D printing. In particular this talk will focus on a biomineralized and non-biomineralized architectures (mantis shrimp, chitons and beetles) and discuss some interesting mechanisms that we encountered only when the material is subjected to extreme loads. These experimental observations helped us formulate some of the hypotheses that were subsequently validated through new computational models and printed concept.

2:30 PM

(ICACC-FS1-010-2020) Effect of microstructure and architecture on the mechanical behavior of bio-inspired layered ceramics (Invited)

R. Bermejo*[1]

1. Montanuniversitaet Leoben, Structural and Functional Ceramics, Austria

The design of "bio-inspired" layered ceramic composites with residual stresses can yield significant flaw tolerance as compared to monolithic ceramics. The location of layers with compressive stresses, either at the surface or embedded in the structure, affects the mechanical response of the composite. In this work, the combined effect of microstructure and architecture on the mechanical behavior of layered ceramics is explored. Equiaxed and textured

microstructures are alternated in adjacent layers to induce in-plane residual stresses, with the textured layers being located at different positions in the architecture. Two design approaches are investigated: (i) architectures with compressive layers at the surface, and (ii) architectures with compressive layers embedded in the structure. In the first case, the mechanical behavior in terms of strength distribution and subcritical crack growth resistance of the layered composites is analyzed on a low temperature co-fired multilayer ceramic. In the second case, the flaw tolerant capability of the architecture is evaluated on an alumina-zirconia layered ceramic as a function of the location of the compressive layers within the structure and their microstructure. A combination of experiments and modelling shows the potential of layered architectures in the design of future ceramic components with enhanced structural integrity.

Mechanical Properties of Bio-inspired Ceramics II

Room: Coquina Salon H
Session Chair: Raul Bermejo, Montanuniversitaet Leoben

3:20 PM

(ICACC-FS1-011-2020) The Structural Form (Invited)

L. Frattari*[1]

1. Altair, USA

The expression Structural Form is used to define the co-existence of two aspects: Form and Structure. In the constructive practice, form and structure are not always integrated and can appear separately; the skin of a building hides a different inner structure, or the style of an object is often independent from its structural system. In many cases the Structural Form is the result of man's adaptation to special conditions: climatic, historical, economic, cultural. In nature that definition is adopted by the life forms following the evolutionary principles that rule the development of biological forms. In mostly of life-forms, structure and form tend to be coincident. Branches of trees and skeleton bones have a specific Structural Form which define their function and design. In this sense Structural Form has a natural meaning. The natural meaning of Structural Form can be adopted in special forms of structural elements, created to build specific architectures. This concept was applied in the past to build architectures based on empirical knowledge and it has been abandoned in the last century to follow abstract forms. The experiences shown in the present work have demonstrated how structural optimization procedures, like topology optimization, can mimic natural processes adding aesthetic value to architectural structures and industrial design products.

3:50 PM

(ICACC-FS1-012-2020) A nature-inspired unconventional process generates 3-D biomorphic ceramics with unusual mechanical performance and enhanced biological behaviour (Invited)

S. Sprio*[1]; A. Ruffini[1]; S. Panseri[1]; M. Montesi[1]; R. Cavuoto[3]; F. Salamanna[2]; M. Fini[2]; D. Bigoni[3]; A. Tampieri[4]

1. National Research Council of Italy, Institute of Science and Technology for Ceramics, Italy
2. Laboratory of Preclinical and Surgical Studies, Rizzoli-RIT Department, IRCCS-Rizzoli Orthopedic Institute, Italy
3. University of Trento, Department of Civil, Environmental and Mechanical Engineering, Italy
4. National Research Council of Italy, Institute of science and technology for ceamics, Italy

A novel, unconventional fabrication approach based on biomorphic transformation of a natural wood generated a 3D porous apatite scaffold showing hierarchical architecture at the base of superior mechanical properties, unusual for ceramics, and biological behaviour, in respect to a macroporous sintered ceramic. The process, based on heterogeneous chemical reactions between biomorphic solids and reactive gases, did not require the use of high temperature sintering for the final consolidation, thus the final scaffold could retain highly bioactive composition, multi-scale porosity recapitulating in detail the lymphatic system of the original wood, and damage-tolerant mechanical performance. The unique association of these features in a bone scaffold gave unusual mechanical properties, superior to those of sintered porous ceramics, and greatly enhanced the activity of mesenchymal and endothelial cells in vitro and in vivo, thus being promising as a new generation biomaterial able to solve clinical needs in regenerative medicine, still prevented by the ineffective mechanical and biological properties of sintered bioceramics.

4:20 PM

(ICACC-FS1-013-2020) Mechanical characterization and constitutive behavior of 3-D supercrystalline ceramic-organic nanocomposites

B. Bor*[1]; D. Giuntini[1]; B. Domènech[1]; M. V. Swain[2]; G. A. Schneider[1]

1. Hamburg University of Technology, Advanced Ceramics, Germany
2. University of Sydney, Aerospace, Australia

In recent years, there have been various developments in the field of bioinspired materials. In this context, nacre is an interesting model material thanks to its special "brick-and-mortar" structure and ceramic-organic composition. To mimic nacre's unique combination of strength and toughness, a bottom-up approach has been implemented, starting from the nanoscale of the functionalized nanoparticles and going up in the hierarchy. The supercrystalline domains obtained in the first hierarchy which consist of tightly packed nanoparticles were used as the building blocks of the second hierarchy. In order to gain information about the constitutive behavior of the first hierarchy, mechanical characterization was conducted at this level. Superlattices formed by self-assembly of organically-grafted nanoparticles were pressed for shaping and heat-treated to induce the crosslinking of the organic ligands which give the final strength of the material. Supercrystalline bulk 3D pellets were characterized mechanically with techniques range from nanoindentation to micro-bending, -compression, and fracture toughness. A thorough overview of the nanocomposites' rheology, strength, hardness, stiffness and fracture toughness has emerged, leading to guidelines towards the production of bulk supercrystalline materials with tailored mechanical properties.

4:40 PM

(ICACC-FS1-014-2020) Processing of Directionally Porous Sintered Lithium Titanate (LTO) Employing Ice-templating and Characterization of Compressive Mechanical Properties

R. Parai*[1]; D. Ghosh[1]; T. Walters[1]; J. Marin[1]; B. Meechan[1]; S. A. Danquah[2]; G. Koenig[3]

1. Old Dominion University, Mechanical and Aerospace Engineering, USA
2. Norfolk State University, Center for Materials Research, USA
3. University of Virginia, Department of Chemical Engineering, USA

Ice-templating is an emerging technique for processing of bio-inspired materials, in which phase separation of solvent crystals and ceramic particles followed by freeze drying and sintering result in ceramic foams with directional porosity. Bio-inspired, ice-templated sintered lithium titanate (LTO) porous ceramics are potential for energy storage. However, to utilize them, one important aspect is to understand compressive mechanical behavior of ice-templated LTO materials in relation to processing variables, composition, sintering temperature, and microstructure. In this presentation, we will discuss the synthesis of ice-templated sintered LTO materials, and the role of different variables in tailoring length-scale features of hierarchical microstructure and the corresponding effects on compressive mechanical properties and failure behavior. We have utilized different methods to prepare aqueous LTO suspensions and understand the impact of the methods on ice-templated microstructure and compressive mechanical properties for optimizing the

process variables to achieve desired porosity, hierarchical microstructure, and mechanical properties. We expect that the results will provide insights into the synthesis and structure-property relationships of ice-templated porous LTO materials.

5:00 PM

(ICACC-FS1-015-2020) Processing bio-inspired graphene/alumina composites for lightweight bearings (Invited)

V. Garcia Rocha*[1]; G. Menendez[1]; S. Evans[1]; G. Min[1]

1. Cardiff University, School of Engineering, United Kingdom

The development of new structural ceramic matrix composites with a combination of superior mechanical and electrical properties is an exciting field with a large number of engineering applications such as bearings for helicopter rotors; bearing assemblies in X-ray tubes; and/or machine spindles for high-speed rotation. In this talk we show the challenges of maximizing electrical and mechanical properties of graphene/ceramic composites by a research methodology based on wet chemistry synthesis, processing and the selection of the appropriate precursor of graphene (graphene oxide). The key novelty of our research is to combine water-based chemistry and processing with Spark Plasma Sintering that could be easily adopted by ceramics manufacturers. This methodology will ensure the minimum amount of graphene is used that guarantees the desired microstructure and electrical conductivity. Processing materials in a way that the material microstructure resembles the hierarchical structures present in nacre or molluscs shells is an inspirational and novel route to achieve the mechanical properties goal, which in turn could also help to achieve the electrical conductivity goal.

FS3: Molecular-level Processing and Chemical Engineering of Functional Materials

Polymer Derived Ceramics: Properties and Applications

Room: Coquina Salon C

Session Chairs: Aleksander Gurlo, Technische Universitaet Berlin; Gabriela Mera, TU Darmstadt

8:30 AM

(ICACC-FS3-008-2020) Polymer Derived Functional High Temperature Materials (Invited)

K. Lu*[1]; N. Yang[1]; D. Erb[1]

1. Virginia Tech, USA

There have been increasing needs for high thermal stability functional materials. In this study, we present our work on electrically conductive materials and magnetic materials obtained by in situ synthesis of silicon oxycarbide (SiOC)–based systems. When using TiO_2 nanoparticles and Ti organic compounds as additives to form TiC_xO_y composites, higher pyrolysis temperature leads to more TiC formation but lower thermal stability. With TiO_2 particle addition, the highest electrical conductivity is 5.03 S cm^{-1} at 400°C, the highest for air atmosphere condition. With Ti organic compound addition, TiC leads to high electrical conductivity at elevated temperatures; the maximum conductivity is >1000 S/m conductivity for Si-O-C-Ti ceramics. The key issue for tuning the SiOC–TiC_xO_y system for both thermal stability and electrical conductivity is to avoid the destabilization of the SiOC system. For magnetic Ni-containing silicon oxycarbide (SiOC-Ni), the formation of nickel silicides (Ni_xSi_y) is completely suppressed by the effect of water vapor during the pyrolysis. The fundamental phase evolution process and mechanism are explained. This series of SiOC-Ni materials also demonstrates exciting ferromagnetic behaviors. Their new semiconducting behavior with soft ferromagnetism presents promising application potentials for magnetic sensors, transformers, actuators, etc.

9:00 AM

(ICACC-FS3-009-2020) Functional ceramics via precursor chemistry coupled with forming methods (Invited)

M. Balestrat[1]; A. Lale[1]; O. Hanzel[2]; Z. Lences[2]; P. Sajgalik[2]; S. Bernard*[1]

1. CNRS, IRCER, France
2. Institute of Inorganic Chemistry, Slovak Academy of Sciences, Ceramic Department, Slovakia

Silicon-based non-oxide ceramics (SiC, Si_3N_4) have attracted much attention, primarily due to their good mechanical and chemical properties, and also their reliability at room and elevated temperatures. They have great potential for many industrial uses as engineering components. The addition of a second ceramic (nano) phase to SiC or Si_3N_4 leads to materials which promise applications in many fields and offer solutions for most of the market demands. However, the preparation of these materials is still a challenging task according to the fact that the conventional processes unavoidably lead to size and structure inhomogeneities of the different phases and presence of impurities which affect the properties. The Polymer-Derived Ceramics (PDCs) route offers new preparation opportunities in ceramic sciences. The molecular origin of preceramic polymers and the possibility to design advanced ceramics in particular forms play a major role in the preparation of functional ceramics. In this talk, we will present our last results on the modification of polycarbosilanes and polysilazanes with metal-organic species to offer functionalities to the materials obtained after the thermo-chemical conversion of precursors. The chemistry behind the preparation of materials will be described before investigating the properties and applications of the final ceramics.

9:30 AM

(ICACC-FS3-010-2020) Precursor-derived $TiNb_2O_7$ based nano-composites for lithium-ion batteries (Invited)

R. Kumar*[1]

1. Indian Institute of Technology Madras, Metallurgical and Materials Engineering, India

We report a simple method to producing $TiNb_2O_7$ (TNO) nano-composites with carbon micro-tubes reinforcements (TNO/CMT) via precursor route. As-pyrolyzed precursors (without CMT) yielded pristine TNO with a specific surface area (SSA) of 28 m^2/g, whereas pyrolyzed precursors with carbon source (TNO/CMT) yielded stoichiometric $TiNb_2O_7$ and non-stoichiometric $Ti_{0.712}Nb_{0.288}O_2$ with increased SSA of 89 m^2/g. Transmission electron microscopy (TEM) and N_2 adsorption/desorption isotherm revealed existence of good contact between the CMT and TNO particles which resulted in improved electrical conduction. This unique combination of stoichiometric and non-stoichiometric phases coupled with high SSA in TNO/CMT nano-composites resulted in better rate capability and cyclic stability even at higher C-rates in contrast to pristine TNO, exemplifying the potential of this class of materials as anode materials in Li-ion batteries.

Polymer Derived Ceramics and Glasses

Room: Coquina Salon C

Session Chairs: Kathy Lu, Virginia Tech; Ravi Kumar, IIT BHU

10:20 AM

(ICACC-FS3-011-2020) Thiol-ene click chemistry assisted additive manufacturing of ceramics from preceramic polymers (Invited)

A. Gurlo*[1]; X. Wang[1]

1. Technische Universitaet Berlin, Chair of Advanced Ceramic Materials, Germany

Here we introduce a versatile stereolithographic route to fabricate Si-containing thermosets that yield high performance ceramics upon thermal treatment. Our approach is based on a fast and inexpensive thiol-ene free radical addition that can be applied for different

classes of preceramic polymers with carbon-carbon double bonds. Due to the rapidity and efficiency of the thiol-ene click reactions, this additive manufacturing process can be effectively carried out using conventional light sources on benchtop printers. Through light initiated cross-linking, the liquid preceramic polymers transform into stable infusible thermosets that preserve their shape during the polymer-to-ceramic transformation. Through pyrolysis the thermosets transform into glassy ceramics with uniform shrinkage and high density. The obtained ceramic structures are nearly fully dense, have smooth surfaces, and are free from macroscopic voids and defects.

10:50 AM

(ICACC-FS3-012-2020) Molecular Approaches Towards Novel Functional Composites Containing Low-Dimensional Nanocarbon (Invited)

G. Mera*[1]

1. TU Darmstadt, Materials Science, Germany

The protection of the environment by increasing energy efficiency as well as by developing new clean energy sources is one of the most important global challenge of our days. Within this context, carbon-based materials and (nano)composites have been shown to be highly promising for energy conversion and storage purposes. Thus, nanocarbons possessing various morphologies (and dimensionality) and size-/surface-dependent (and hence modulated) physical-chemical properties have been synthesized and studied concerning their potential use in applications related to energy conversion, energy storage and thermal management solutions. In the present talk, synthetic strategies to access nanocomposites containing 0D-nanocarbon (nanodiamond, onion-like carbon), 1D-nanocarbon (carbon nanotubes), 2D-nanocarbon (graphene and nanographene), as well as 3D-nanocarbon (multilayer graphene) dispersed phases will be introduced. The prepared materials will be discussed within the context of their preparative accessibility from tailor-made single source precursors as well as their thermodynamic stability. Moreover, selected functional properties thereof will be highlighted and assessed in the light of prospective energy-related applications.

11:20 AM

(ICACC-FS3-013-2020) Aluminate glasses: Chemistry, phase composition and luminescence (Invited)

R. Klement[2]; K. Haladejova[2]; M. Majerova[3]; J. Kraxner[2]; E. Bernardo[4]; D. Galusek*[1]

1. IIC SAS, Joint Glass centre, Slovakia
2. Alexander Dubcek University of Trencin, FunGlass, Slovakia
3. Institute of Measurement SAS, Slovakia
4. University of Padova, Dipartimento di Ingegneria Meccanica, Italy

Aluminate glasses are transparent in IR, UV and VIS, so they represent ideal host matrix for optically active dopants. Due to their low phonon energy in comparison to silicate glasses, non-radiative transitions are suppressed and high efficiency of luminescence isbe expected. They can also accommodate higher concentrations of optically active dopants, such as rare earth or transition metal elements, in comparison to their single- or polycrystalline counterparts of identical composition, such as yttrium- or ytterbium aluminium garnets, or the respective rare earth aluminate perovskites. The main disadvantage of aluminate glasses is that they are usually relatively difficult to prepare: their preparation requires intense source of heat due to their high melting temperatures, high cooling rates due to their low glass forming ability, and highly homogeneous and pure raw materials. In the presentation we summarise our efforts in preparation of various types of aluminate and aluminosilicate glasses with luminescent properties. Modified sol-gel methods have been applied for synthesis of precursor powders with sufficient purity, homogeneity and phase composition. The glasses were prepared by flame synthesis from the precursor powders: the influence of processing conditions, chemical composition, doping levels, and controlled crystallization on luminescent properties of prepared glasses is summarised and discussed.

11:50 AM

(ICACC-FS3-014-2020) Upcycling of iron rich inorganic waste in functional glass-ceramics (Invited)

A. Rincon[1]; P. Rabelo Monich[1]; D. Desideri[1]; E. Bernardo*[1]

1. University of Padova, Department of Industrial Engineering, Italy

The intensive mechanical stirring of suspensions of recycled glass and inorganic waste powders in 'weakly activated' aqueous solutions (e.g. 1-2.5 NaOH) has been established as a first step for obtaining highly porous glass-ceramic foams, followed by viscous flow sintering, at 800-1000 °C. Since the foaming does not occur upon sintering, the firing aims just at the consolidation of glass powders with concurrent incorporation of pollutants from iron rich waste (slag from Cu metallurgy, fly ash from coal combustion, vitreous residues of plasma processing of municipal solid waste). Engineered mixtures allows for the obtainment of chemically stable foams, from treatments in air, according to several leaching tests. Treatments in nitrogen are even more significant, since they extend the conditions for stabilization and determine novel functionalities. In fact, treatments in nitrogen may promote the formation of relatively silica-poor silicate crystal phases, even operating with soda-lime glass (providing a lower stabilization in air, compared to glass from the recycling of pharmaceutical vials); the enhanced silica content of the residual glass phase justifies the higher durability. In addition, the change in the atmosphere favours the formation of magnetite (Fe3O4), in turn enabling a significant electromagnetic shilding effectiveness, in a wide frequency range, owing to electrical conductivity and ferrimagnetism.

Materials for Energy Applications

Room: Coquina Salon C

Session Chairs: Thomas Fischer, University of Cologne; Akihiko Ito, Yokohama National University

1:30 PM

(ICACC-FS3-015-2020) 2D materials-based nanostructured interfaces for membrane and energy applications (Invited)

P. Miele*[1]

1. Ecole Nationale Superieure de Chimie de Montpellier, France

The most fundamental phenomena for nanostructured membrane are the control of interfaces. Their performances can be controlled by the enhanced geometric area of the nanostructured interfaces. Thus, an accurate control of the geometry (size, porosity etc.) and interfaces is primordial to achieve the balance between large/control interface areas and efficient transport conditions. Here, we used different synthesis techniques such as atomic layer deposition (ALD), electrospinning, electrodeposition, nanospheres lithography, pyrolysis of preceramic precursors, etc. as the main tools for the creation of controlled nanostructured interfaces. 2D material are very attractive for many applications particularly as sensors, electronics, adsorbents and catalysis devices. In the case of boron nitride, novel properties can arise from BN nanosheets (BNNS) due to the high surface area and the edge structures. Here, we used the exfoliation as the main tool for the creation of controlled two-dimensional nanostructured interfaces in order to investigate their performances. 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 bio-nanocomposites materials for packaging. Recent works using the Pickering emulsions technique for the preparation of porous materials will be described.

2:00 PM

(ICACC-FS3-016-2020) Chemical Engineering of Functional Materials for Improved Electrodes: Application to technologies for an effective energy transition (Invited)

J. R. Morante*[1]

1. IREC, Catalonian Institute for Energy Research, Spain

Once of the major challenge of our society is to displace petrochemical processes as backbone of the energy transition. For it, renewably-powered electrosynthesis or the implementation of solar refineries together with the ability to store energy are key issues. In all of these cases, the properties of the electrochemical electrodes become to determine the overall performances of the proposed technologies and processes. In this contribution, the configuration and the use of appropriate functional materials as well as the technological procedure to be implemented will be reviewed. We provide a techno-economic offering targets that would need to be met for economically compelling industrial implementation to be achieved We describe the technical challenges and economic barriers to marketable electrosynthesized chemicals and chemical & electrochemical energy storage.

2:30 PM

(ICACC-FS3-017-2020) Ion conducting polymers that emulate LiPON as ceramic adhesives, coatings, binders: Towards all solid-state batteries (ASBs) (Invited)

R. M. Laine*[1]; E. Temeche[1]; X. Zhang[1]

1. University of Michigan, Materials Science and Eng., USA

Dendrite formation in liquid electrolyte batteries causes short circuits and cell failure prompting efforts to use ceramic electrolytes. Ceramic electrolytes offering Li^+ conductivities commensurate with liquid electrolytes, 0.1-1 mS/cm, include LATP and c-LLZO. Unfortunately, LATP undergoes irreversible reduction during cycling and c-LLZO undergoes Li dendrite penetration along grain boundaries. Application of thin films (<200 nm) of gas phase deposited, amorphous LiPON block dendrite penetration. Unfortunately, such films offer Li^+ conductivities of 10^{-3}-10^{-5} mS/cm. We have successfully synthesized LiPON-like polymers to explore their utility as a coatable equivalent of gas phase deposited LiPON in the assembly of ASBs. The polymer precursors synthesized will, on heating to > 300 °C, transform to LiPON like glasses. As such, they can be used as noted in the title to create protective interfaces with ceramic electrolytes, as adhesives to mate solid-state battery components, or as non-fugitive binders to promote densification of powder compacts. These results contrast with gas phase deposited LiPON in that the precursor derived LiPON conductivities are of the order of 0.1-1 mS/cm on various substrates including LATP, LLZO, and $LiAlO_x$. We plan to present data on ASB cycling with a variety of cathode materials and Li anodes.

Morphology Control in Materials Processing

Room: Coquina Salon C

Session Chairs: J. Morante, IREC, Catalonian Institute for Energy Research; Richard Laine, University of Michigan

3:20 PM

(ICACC-FS3-018-2020) Precursor Chemistry and Strategies for Nanowires of Metastable Composition (Invited)

S. Barth*[1]

1. Goethe University Frankfurt, Physics Institute, Germany

The lecture will cover important aspects of precursor chemistry for the synthesis of functional nanomaterials. Specific attention will be paid to the synthesis of inorganic nanowires. Gas phase and solvent-based approaches for the controlled formation of these anisotropic nanostructures with metastable composition will be described in more detail. Highly crystalline Ge-based nanowires and nanorods can be synthesized at low temperatures. The process conditions enable the formation of materials with metastable composition and significantly altered physical properties. For instance, Sn concentrations of >10 times the solubility limit in Ge according to the phase diagram leads to a direct bandgap semiconductor material. Therefore, the crystal growth has to be conducted under kinetic control for the formation of metastable material composition. The Sn at substitutional sites in the Ge lattice leads to altered physical properties such as efficient light emission in the mid-IR from these nanowires. In addition to growth studies, the thermal stability of the prepared material has been investigated in detail and decomposition paths at low temperature have been identified to be initiated by solid state diffusion of metallic clusters enabling dissolution of $Ge_{1-x}Sn_x$ and recrystallisation of $Ge_{1-y}Sn_y$ with x>y in its trace.

3:50 PM

(ICACC-FS3-019-2020) Fabrication of Boron Nitride Fibers by Forcespinning Method

D. Santiago*[1]; M. Lizcano[1]

1. NASA Glenn Research Center, Materials and Structure Division, USA

The unique multifunctional properties of boron nitride (BN) nanomaterials are identified as a parameter that would revolutionize electric propulsion in Aeronautics due to the lightweight ceramic with chemical inertness, high strength, high electrical resistivity and high thermal conductivity. Hexagonal BN (h-BN) nanofibers will enable new high-performance fibers that can be used in ceramic or polymer matrix composites, or thin films to provide revolutionary multifunctional ceramics for extreme environments and structures. Polymer derived h-BN materials have been previously demonstrated, providing an avenue to tailor properties of the ceramic end product. This effort also uses forcespinning (FS) technology that produces continuous non-woven nanofibers in a range of diameters depending on the processing parameters with a large production rate of 1 g/min allowing for manufacturing scale production. FTIR, SEM, TGA and XRD were used to characterize the materials in each processing steps.

4:10 PM

(ICACC-FS3-020-2020) High-speed Epitaxial Growth of Functional Oxide Films Using Metal-organic Chemical Vapor Deposition and Their Luminescence and Magnetic Responses (Invited)

A. Ito*[1]

1. Yokohama National University, Environment and Information Sciences, Japan

Chemical vapor deposition (CVD) has great diversity to synthesis functional and structural ceramic films using metal-organic precursor vapors. Irradiation of intense laser beam into CVD process results in the rapid synthesis and significant self-oriented growth of various functional ceramic films, including fast epitaxial growth of single-crystalline thick films on single-crystal substrates. A high-speed epitaxial growth via CVD technique is advantageous for single crystal growth as compared to the melt-solidification process because it can reduce process temperature by half and can produce incongruent melting compounds feasibly from a vapor phase. This talk will introduce you to recent achievements on high-speed epitaxial growth of functional oxide films, including rare-earth oxides, silicates, and ferrites. Characterizations, and luminescence and magnetic responses of these epitaxial thick films will be demonstrated.

4:40 PM

(ICACC-FS3-021-2020) Influence of precursor chemistry in magnetic field CVD (mfCVD) (Invited)

T. Fischer*[1]; D. Stadler[1]; S. Mathur[1]

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

In addition to temperature, pressure and electromagnetic radiation, electric and magnetic fields can be employed as external stimuli in materials processing to influence structure, morphology and phase formation. While the influence of electric fields has been investigated more thoroughly, reports of magnetic fields, especially in chemical vapor deposition (CVD) remain scarce and limited to few examples, where for example magnetic metal catalysts guide the directional growth of CNTs. Here we report the influence of external magnetic fields on the decomposition and phase and morphology evolution of transition metal oxides and nitrides based on molecular single source precursors. The precursor composition has direct influence on the thermochemical as well as magnetic properties and thus directly affect the growth kinetics under applied magnetic fields. By correlating depositions under varying deposition temperatures, field strengths and deposition times, magnetic field CVD (mfCVD) offers a versatile additional degree of freedom in optimizing gas phase deposition processes. Morphological and structural characterization of the resulting thin-films using SEM, TEM, XRD and XPS are corroborated by XAS and X-PEEM analysis to gain a deeper insight in the decomposition and growth mechanism.

FS4: Green Technologies and Ceramic/ Carbon Reinforced Polymers

Mechanical Behavior or Ceramic/Carbon Reinforced Polymers and Composites III

Room: Halifax A/B

Session Chairs: James Hemrick, Oak Ridge National Laboratory; Hua-Tay Lin, Guangdong University of Technology

8:30 AM

(ICACC-FS4-010-2020) Prediction of notched strength of thin-ply CFRP laminates with various ratios of 0-degree layer (Invited)

R. Higuchi*[1]; R. Aoki[1]; T. Yokozeki[1]

1. University of Tokyo, Department of Aeronautics and Astronautics, Japan

Recently, freedom in the design of the composite laminated structure in terms of ply thickness and stacking sequence per certain thickness have been improved because of the development of thin-ply technology. Owing to this improvement, there is some possibility that the laminates with a high ratio of 0-degree ply (more than 50%) will be applicable in structural components where the loading direction is limited in a certain direction. Therefore, this study focuses on the effect of the ratio of the 0-degree ply on the notched strength of thin-ply laminates. For this purpose, the progressive damage simulation scheme was developed. The developed scheme utilizes three kinds of damage models to predict fiber breakage, matrix crack, and delamination under both tensile and compressive loading. For the verification of the developed scheme, the predicted notched strengths were compared with experiments in several basic stacking sequences. Finally, the simulations of various laminates with different ratios of 0-degree ply were conducted to investigate their effect on notched strength.

9:00 AM

(ICACC-FS4-011-2020) Mesoscale modeling of intra-laminar fatigue damage in composite laminates considering ply thickness

R. Aoki*[1]; R. Higuchi[1]; T. Yokozeki[1]

1. University of Tokyo, Department of Aeronautics and Astronautics, Japan

This study aims to propose a mesoscale damage model in composite laminates under fatigue loading considering the effects of ply thickness. Focusing on intra-laminar damage in composite laminates, fatigue damage evolution law is defined. The intra-laminar damage is numerically described based on continuum damage mechanics using damage variables in the fiber, transverse and shear direction, respectively. Especially, in the transverse direction and the shear direction where damage in the matrix is dominant, the ply thickness is added as a parameter in the model because the ply thickness has a significant effect on the transverse and shear strength. A fatigue damage simulation with the proposed damage model is carried out on several laminates with various ply thicknesses to predict the stiffness degradation as a function of the number of loading cycles. This study discusses the damage delay effect and the change of stiffness reduction when the ply thickness changes. Furthermore, the proposed fatigue damage models and simulation method are validated by comparing with experimental results.

9:20 AM

(ICACC-FS4-012-2020) Qualification of Polymeric Composites for Piping Repair by Compressive Testing

K. Silva*[1]; M. O. Moreira[1]; R. G. Almeida[1]; F. P. Lopes[1]; E. A. Carvalho[1]; C. F. Vieira[2]

1. State University of Northern Rio de Janeiro, Advanced Materials Laboratory, Brazil
2. State University of the North Fluminense, Advanced Materials Laboratory, Brazil

One of the major failures of petroleum fluid pipelines today is external corrosion caused to the marine environment. Definitive piping repair request a production shutdown and it have a risk of explosion due to spark generated during the welding process, if is used the commonly repair system, cutting and welding in the metal. This work investigates a modified epoxy resin incorporating particulates to develop a definitive repair by polymeric composites, avoiding the stop production and another advantage is that no hot work is used during its execution. In addition, it is good to point out the lower cost compared to traditional repairs. Epoxy resin samples and their composites with particulates were subjected to a compression testing, and the composites presented better consistency to be applied and mechanical strength for repair application. The particulates used were recycled glass, calcite and mica. Nowadays, for practical purpose, two standard guide the design of composite repair system, ISO TS 24817 and ASME PCC2. For compressive testing was following the ASTM D695. The results indicates that all mixtures can be used for permanent piping repairs.

9:40 AM

(ICACC-FS4-013-2020) Enhancement of Bending Strength, Thermal Stability and Recyclability of Carbon-Fiber-Reinforced Thermoplastics by Using Silica Nano Colloids (Invited)

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

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

To develop a technique for recycling highly valuable carbon fibers from carbon-fiber-reinforced thermoplastics (CFRTPs) using nylon, heat-resistant silica nano colloids were synthesized through a sol–gel reaction. The surfaces of carbon fibers were modified with silica nano colloids via electrodeposition. The fabricated CFRTP showed homogeneous strength because the silica particles acted

as spacers between the carbon fibers, which adhered to each other owing to their hydrophobicity in the nylon resin. Additionally, the mechanical properties of CFRTP were maintained above room temperature. When carbon fibers were reclaimed from the CFRTP via heating up to 500 °C in air, the silica nano colloids deposited on the carbon fibers helped prevent the tensile strength of the fibers from degrading due to the oxygen in air during the heating process. The present study demonstrated that the silica nano colloid surface modification enhanced the mechanical properties, thermal stability, and recyclability of CFRTPs.

Innovative Processing of Ceramics and Composites for Environmental Sustainability and to Minimize Energy Utilization and Pollution

Room: Halifax A/B

Session Chairs: Gurpreet Singh, Kansas State University; Satoshi Kobayashi, Tokyo Metropolitan University

10:30 AM

(ICACC-FS4-014-2020) Cold Sintering: A Natural Path to a Sustainable Sintering Process and Possibly a Circular Economy (Invited)

C. Randall*[1]

1. Penn State University, Materials Science and Engineering, USA

Cold Sintering involves a transient phase that permits the densification of particulate materials at low temperatures 300 ºC and below. Using this approach, many ceramic and composite materials have been driven to high densities at temperatures far below the melting point under a uniaxial pressure. Densification stages and kinetic scaling laws follow two distinct stages namely particle compaction and dissolution-precipitation processes, respectively. The grain growth process is consistent with classical behavior, but with a much lower activation energy than in a purely thermally driven process. Despite these initial trends having been identified, there is much to be investigated in the liquid-solid interface and intergranular phase development. Now ceramics, metal and polymers can be processed under a common platform in one step processes. With controlling the forming process new nanocomposites can be fabricated. Polymers, gels and nanoparticulates can be dispersed, interconnected and sintered in the grain boundaries of a ceramic matrix phase. This then could lead to new devices and devices that can impact important products that are required for a more sustainable economy.

11:00 AM

(ICACC-FS4-015-2020) Understanding and Designing Interfaces and Defects in Perovskite Solar Cells (Invited)

J. Correa-Baena*[1]

1. Georgia Institute of Technology, School of Materials Science and Engineering, USA

Perovskite solar cells promise to yield efficiencies beyond 30% by further improving the quality of the materials and devices. Electronic defect passivation, and suppression of detrimental charge-carrier recombination at the different device interfaces has been used as a strategy to achieve high performance perovskite solar cells. In this presentation I will discuss the role of electronic defects and how these can be passivated to improve charge-carrier lifetimes and to achieve high open-circuit voltages. I will discuss the characterization of 2D and 3D defects, such as grain boundaries, crystal surface defects, and precipitate formation within the films, by synchrotron-based techniques. The importance of interfaces and their contribution to detrimental recombination will also be discussed. As a result of these contributions to better understanding 2D and 3D defects, the perovskite solar cell field has been able to improve device performance. Albeit the rapid improvements in performance, there is still a need to improve these defects to push these solar cells beyond the current state-of-the-art.

11:30 AM

(ICACC-FS4-016-2020) Carbonate Ceramics via Microbial Curing (Invited)

R. Riman*[1]; D. Kopp[1]; P. Kim[1]; C. Ma[1]

1. Rutgers University, Materials Science & Engineering, USA

Utilization of captured carbon dioxide (CO_2) by converting it into valuable products, such as fuels, chemicals, plastics, and building materials is thought to be necessary for sufficient carbon management. A breakthrough process patented by Rutgers University called gas-assisted reactive hydrothermal liquid phase densification (g-rHLPD) has been shown as effective in utilizing CO_2 by curing/densifying synthetic and mineral silicates at temperatures below 100ºC. Unfortunately, the g-rHLPD curing method cannot be used to produce large cast and pre-cast monoliths (>20 cm) due to a limitation caused by CO_2 diffusion into the uncured structure. Additionally, obtaining CO_2 in sufficient quantities to satisfy the needs of large volume businesses (e.g., carbonate cement) can be costly and logistically impossible in today's carbon economy. This presentation will describe an alternate carbonation approach that is independent of waste/dilute CO_2 streams and air-capture technologies and instead uses CO_2 generated by microbes to create a carbonate-bonded ceramic monolith. This presentation will discuss the microbial carbonation process for the production of a myriad of composite structures and provide a perspective on how its contribution to sustainability can impact a broad spectrum of business in performance enhancing products while diminishing CO_2 emissions.

12:00 PM

(ICACC-FS4-017-2020) Ceramic composites as a way to mitigate pollution worldwide

H. Colorado*[1]

1. Universidad de Antioquia, Colombia

Ceramic based composite materials are by weight the materials type most fabricated worldwide. With the increasing pollution and all the derived environmental problems, ceramic composites fabricated with residual ceramic materials are a high impact solution not only to the environment, but also to new business. This presentation explore the progress using diverse solid wastes for making ceramic composites worldwide and also present some case studies in Colombia.

Recycling of Ceramics and Composite Wastes

Room: Halifax A/B

Session Chair: Henry Colorado, Universidad de Antioquia

1:30 PM

(ICACC-FS4-018-2020) Recycling of Refractory Ceramic Waste Materials (Invited)

J. G. Hemrick*[1]; J. Waters[1]

1. Reno Refractories, Inc., R&D, USA

Although recycling of refractory ceramic waste materials is of interest for environmental and sustainability reasons, the mechanics and economics of such efforts are not currently available. Yet as raw materials become more scarce and environmental regulations tighten, such efforts will be required. This project looks at utilizing an internal waste stream, waste material from a refractory manufacturing shape shop, as a source of high-quality recycled material for use in virgin product. Material from damaged or out of spec shapes will be crushed, characterized and reintroduced as a raw material at various ratios and the effect on the virgin material will be analyzed.

2:00 PM

(ICACC-FS4-019-2020) Use of Glass Polishing Sludge Waste to Produce Clayey Rustic Floor Tiles

C. F. Vieira*[1]; G. G. Delaqua[1]; H. Colorado[2]

1. State University of the North Fluminense, Advanced Materials Laboratory, Brazil
2. Universidad de Antioquia, Colombia

The kaolinitic clays from Campos dos Goytacazes, state of Rio de Janeiro, Brazil, are known by their refractory behavior with elevated porosity after the firing stage. In the present work, the incorporation of up to 30 wt% of a sludge waste from the polishing of cutted glass plates into a typical kaolinitic clay body was evaluated. The raw materials were characterized by XRD, XRF, particle size distribution and DTA/TGA techniques. Specimens were extruded and fired at temperatures ranging from 800 to 1100°C. The technological properties were evaluated in terms of water absorption, linear shrinkage, and flexural rupture strength. The microstructure of the ceramics was evaluated by SEM. The results indicated that the waste incorporation improved both the ceramic water absorption and the mechanical strength. Finally, this work indicated that clayey rustic floor tiles production is a viable and technically advantageous alternative for recycling this type of waste, also bringing real benefits to the quality of ceramics.

2:20 PM

(ICACC-FS4-020-2020) Utilization of Local Quarry Waste Material from Northern Mindanao, Philippines for the Production of Ceramic Wall Tiles

M. Ventures[1]; E. d. Magdaluyo[3]; E. Salamangkit-Mirasol[4]; M. Zabala[5]; M. Fuji[2]; R. V. Virtudazo*[1]

1. Mindanao State University-Iligan Institute of Technology, Department of Materials & Resources Engineering and Technology (Ceramic Engineering Program), Philippines
2. Nagoya Institute of Technology, Japan
3. University of the Philippines, Philippines
4. Mariano Marcos State University, Department of Materials Science and Engineering (Ceramic Engineering Program), Philippines
5. Yamada Technology Corporation, Research and Development Department, Philippines

Ceramic wall tiles are materials with excellent mechanical properties and low water absorption. These are industrially processed material through high sintering temperature of 1200-1260C and fast firing cycles which requires high-quality fluxes. However, there is a high cost and limited reserves of traditionally used material which is the feldspar. In this study, a local source quarry waste material from Canete Aggregate, Bonbon, Iligan City, Philippines was partially utilized to determine its fluxing potential to the standard formulation. The physical properties revealed zero percent water absorption and 12.26% firing shrinkage which is also similar to the traditionally used feldspar. The partial replacement of 10% quarry waste sintered at 1235C produced a ceramic wall tile with low water absorption of 0.31% and a high modulus of rupture of 7.57 MPa. The surface morphology showed both open and closed porosity, elongated needle-shaped crystals, and other crystalline phases in the glassy matrix. X-ray diffraction revealed that quartz, mullite, and cristobalite are the major phases formed in the sintered body.

2:40 PM

(ICACC-FS4-021-2020) Recycling Strategies for Glass-reinforced Themoset Composite Materials (Invited)

S. Bull*[1]; A. Yadav[1]

1. Newcastle University, Engineering, United Kingdom

The growth of polymer composite use is affected by environmental legislation, disposal cost and the lack of suitable recycling strategies. In recent years, industry has controlled the amount of production waste by improvements in design and manufacturing techniques but end-of-life waste now dominates material for disposal. About one third of composite waste is sent for energy recovery from the polymer matrix by incineration. This is very successful for recovering clean glass fibre at the expense of the loss of fibre mechanical properties. Mechanical recycling involving shredding and milling waste composite has shown some success but the glass fibre is powdered and the recyclate is only useful as a low value filler material. Chemical or feedstock recycling methods involving pyrolysis, gasification or solvolysis of the polymer matrix have been widely investigated with the aim to depolymerise the material or create useful monomers/chemicals but also involve high temperatures, pressures or chemical environments that lead to glass fibre damage. Methods to address the surface degradation of glass fibre during thermal and thermochemical processing are thus essential. This presentation discusses the techniques to reprocess the surface of glass fibre to recover its mechanical properties after thermal or thermochemical treatment. The potential for producing remanufactured composites of high value will be discussed.

3:30 PM

(ICACC-FS4-022-2020) Damage evaluation of recycle carbon fibers in focusing on reuse for CFRPs (Invited)

T. Irisawa*[1]

1. Nagoya University, Japan

Accordingly, the development of recycle method for carbon fiber reinforced plastics (CFRPs) have been required and several methods have already been proposed. Especially, the methods of recovering carbon fibers (Re-CFs) from CFRPs have been investigated. As typical methods, chemical process by using solvents and pyrolytic process under steam or air at high temperature are known. For Re-CFs, it is important to maintain properties for reuse as reinforcement fiber of CFRTPs. In past studies, it was already reported that Re-CFs suffers various damages and the damages differs depending on the recovering process. However, the detail of damage mechanism is still unclear, hence this study focuses on the damage evaluation of Re-CFs. The damage of Re-CFs recovered by chemical process was little though the tensile strength of Re-CFs recovered by pyrolysis process decreased compared with unused carbon fibers. In the case of pyrolysis process, it is difficult to prevent the thermal decomposition of CFs. Furthermore, it is revealed that the interaction between CFs and matrix was remarkably influenced on the damages. On the other hand, Re-CFs recovered by pyrolysis process had a high interfacial adhesion against the polymer, and this result become a big merit for reuse of Re-CFs.

Environmental, Infrastructure, Energy, Biological, Space, Transportation, Building, and Sport Applications

Room: Halifax A/B
Session Chair: Gustavo Costa, NASA Glenn Research Center

4:00 PM

(ICACC-FS4-023-2020) Temperature response of CFRP exposed to simulated lightning current

S. Kamiyama*[1]; Y. Hirano[2]; T. Okada[2]; T. Sonehara[3]; T. Ogasawara[1]

1. Tokyo University of Agriculture and Technology, Mechanical System Engineering, Japan
2. Japan Aerospace Exploration Agency, Japan
3. Shoden Corporation, Japan

CFRP have been widely applied to primary aircraft structures. One of the serious hazards for aircraft is lightning strike. Lightning strike event on CFRP is caused by thermal, electrical and mechanical phenomena. Because of these complexities, the effects of each phenomenon on lightning strike damage are not clarified in detail. Especially, temperature response of CFRP exposed to lightning current, which is extremely high electric current, has not been

reported in earlier works. Conduction tests, in which electric current is directly applied to an electrode fastened to CFRP, were carried out to clarify the effect of simulated lightning current on Joule heat generation. To investigate the effect of Joule heat generation caused by lightning current, impulse current in accordance with SAE ARP 5412B was applied. The maximum current was 40 kA. To estimate the electrical properties of CFRP, a static current of 3 A was applied before the impulse current test. Numerical analysis based on finite element analysis were carried out to simulate Joule heat generation. Numerical analysis results of static current tests showed good agreement with experimental results. However, temperature distribution of the impulse current test obtained by the numerical analysis was much higher than that obtained by experimental results. These results suggest that material properties of CFRP may be changed under high current such as 40 kA.

4:20 PM

(ICACC-FS4-024-2020) Polymer Derived Silicon Oxynitride (SiON) Coatings for Corrosion Protection of Steels (Invited)

K. Lu*[1]

1. Virginia Tech, USA

Due to its vast industrial applications, steel is often exposed to a wide variety of harsh environments leading to corrosion/oxidation damage. In this work, polymer derived silicon oxynitride ceramic coatings for steels based on perhydropolysilazanes (PHPS) have been explored for low temperature and high temperature use in corrosive environments. They were fabricated by pyrolysis of perhydropolysilazane (PHPS) up to 800°C in air. The chemical structure evolution, pyrolysis behavior, and corrosion properties were investigated. The coatings change from being hydrophobic to being hydrophilic above 200°C pyrolysis temperature. Potentiodynamic polarization tests show improved corrosion resistance for SiON coated carbon steel in a 0.6 M NaCl water solution. Higher pyrolysis temperature leads to higher hardness and Young's modulus coatings. The high-temperature oxidation behaviors of SiON coated AISI 441 substrates were studied in $Ar+O_2$, $Ar+H_2O$, and $Ar+CO_2$ atmospheres at 800°C for 100 hours. The SiON coated AISI substrates showed better performance in all three atmospheres. The thin oxide scale was observed on top of the SiON coating for all the atmospheres, which was attributed to the reaction between oxidizer species in the atmosphere and metallic species (Cr, Mn, and Fe) diffused through the SiON coating from AISI 441 substrate.

4:50 PM

(ICACC-FS4-025-2020) Examination of the applicability of topology optimization technique for designing truss-lattice structures made of unidirectional CFRP

K. Shinomiya*[1]; T. Ogasawara[1]

1. Tokyo University of Agriculture and Technology, Mechanical System Engineering, Japan

At present, topology optimization method is widely used for optimizing the geometries of solid parts made of isotropic materials. The objective of this study is to examine the applicability of topology optimization technique for the basic design of truss-lattice structures made of unidirectional carbon fiber reinforced plastic composites (UD-CFRP). The target object was a box-section cantilever beam subjected to both bending and torsion loads. For the first step, topology optimization was adopted for a cantilever beam made by isotropic materials subjected only to bending load. After optimizing the geometry of the cantilever beam, fiber direction of UD-CFRP was assigned as the maximum or minimum principal stress vector direction. As a result, it was demonstrated that the considerable weight reduction (approximately 50%) could be achieved with maintaining the strength and the bending stiffness. For the second step, topology optimization was adopted for a cantilever beam under both bending and torsion loads. Two methods, namely one-stap and multi-step optimization methods, were examined. As a result, it was implied that the multi-step optimization method was more effective for optimizing the box-section cantilever beam subjected to both bending and torsion loads.

S1: Mechanical Behavior and Performance of Ceramics & Composites

Small Scale Testing

Room: Coquina Salon D

Session Chair: Matthew Appleby, NASA Glenn Research Center

8:30 AM

(ICACC-S1-047-2020) In situ studies on mechanical behavior of flash-sintered TiO_2

J. Li[1]; J. Cho[1]; H. Charalambous[2]; H. Wang[1]; X. Phuah[1]; T. Tsakalakos[3]; A. Mukherjee[4]; N. Bernstein[5]; S. Hellberg[5]; H. Wang[1]; X. Zhang*[1]

1. Purdue University, Materials Engineering, USA
2. Argonne National Lab, USA
3. Rutgers University, USA
4. University of California, Davis, USA
5. Naval Research Laboratory, USA

Ceramic materials have been widely used for structural applications. However, most ceramics have rather limited plasticity at low temperatures. A majority of ceramics fracture well before the onset of plastic yielding. The brittle nature of ceramics arises from the lack of dislocation activity and the need for high stress to nucleate dislocations. Here we have investigated the deformability of TiO_2 prepared by a flash-sintering technique. Our in situ studies show that the flash sintered TiO_2 can be compressed considerably under room temperature without noticeable crack formation. Distinct deformation behaviors have been observed in flash-sintered TiO_2 deformed at different testing temperatures, ranging from room temperature to 600°C. Potential mechanisms of temperature dependent plasticity are discussed.

8:50 AM

(ICACC-S1-048-2020) Measurement of mechanical properties of $BaTiO_3$ layer in multi-layered ceramic capacitor using a microcantilever beam specimen

J. Tatami*[1]; H. Yamaguchi[1]; M. Iijima[1]

1. Yokohama National University, Japan

The measurement of the mechanical properties of electroceramics is important for improving the reliability of ceramic components and devices. Although information of the mechanical properties at the microscale level, especially under tensile or bending stress, is desired, this has not yet been reported. In this study, a bending test using a microcantilever beam specimen was applied to measure the mechanical properties of the $BaTiO_3$ layer in a multi-layered ceramic capacitor. As a result, a nonlinearity relationship between stress and strain was observed during the bending test. The bending strength and failure strain, measured using the microcantilever beam specimen, were much higher than those measured using bulk specimens and almost the same or higher than those of conventional Si_3N_4 ceramics, as the fracture origin was nano-size.

9:10 AM

(ICACC-S1-049-2020) In-situ study on diamond/SiC interfacial strength of diamond/SiC composite

Y. Zhang*[1]; C. Hsu[2]; P. Karandikar[3]; C. Ni[1]

1. University of Delaware, Material Science and Engineering, USA
2. University of Delaware, USA
3. M Cubed Technology, Inc., R&D, USA

Diamond/SiC composites have been considered ideal material candidates for armor, semiconductor processing platform and high energy laser mirror substrates because of their outstanding mechanical

properties, desired electronic and thermal transport properties. Among the various techniques to fabricate diamond/SiC composites, the liquid Si infiltration method is cost-effective and capable of preparing near-net shape components with high quality under a relatively mild processing condition. The resulting product is formed through a high temperature process involving chemical reactions and phase transformations at an initial solid-liquid interface and the interfacial strength of diamond/SiC is an essential parameter for the performance of diamond/SiC composite. In this work, sub-micron dog-bone shaped specimens of diamond/SiC interface were prepared by focused ion beam (FIB) with Nano-Patterning and Visualization Engine (NPVE). The interfacial strength was measured utilizing a home-made in-situtensile stage inside SEM. All of the specimens were found to exhibit characteristic brittle fracture at the interfacial region of diamond/SiC and an average interfacial strength was measured to be 0.75 GPa, consistent with the reported strength of SiC, suggesting a strong interfacial bonding of diamond/SiC. The nanoscale fractography and the fracture mechanism are further evaluated.

9:30 AM

(ICACC-S1-050-2020) Characterizing the Influence of Crystal Orientation on Twin Nucleation in Ferroelastic Ceramics

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

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

Ferroelastic deformation is known to lead to increased toughness in many electroceramic and structural ceramic materials through the nucleation and motion of twins. However, the effect that local microstructure and crystal orientation have on ferroelastic twin nucleation is not yet fully understood. Here, the influence that crystal orientation has on twin nucleation has been examined using small volume single crystal deformation. The orientation of ferroelastic tetragonal zirconia grains have been measured using electron backscatter diffraction (EBSD). Micropillars as well as nanopillars of single crystals have then been prepared using a focused ion beam (FIB). In situ deformation in both scanning and transmission electron microscopes was used to measure the stress associated with twin nucleation for crystals of varying size and crystallographic orientation. Dislocation slip as well as twinning are possible during deformation of small volume specimens, therefore, the relative orientations of both twin and slip planes have been tracked and compared. Critical resolved shear stresses for both types of deformation have been analyzed. Determining the effect of crystal orientation on twin nucleation behavior will enable more informed microstructural design of toughened ceramics in the future.

Mechanics, Characterization Techniques, and Equipment

Room: Coquina Salon D
Session Chair: Kevin Strong, Sandia National Laboratories

10:10 AM

(ICACC-S1-051-2020) Photoluminescence Spectroscopy to Map Residual Stresses in Glass-to-Metal Seals

E. Huntley*[1]; K. T. Strong[1]; S. P. Meserole[2]; T. Diebold[1]

1. Sandia National Laboratories, Materials Mechanics and Tribology, USA
2. Sandia National Laboratories, Applied Optical/Plasma Science, USA

In components with two materials, such as glass-to-metal (GtM) seals, residual stress can reduce long-term reliability. Therefore, it is important to be able to accurately measure residual stress within these components. The residual stress can be from a large strain due to the mismatch of thermo-physical response of the two materials or a small strain due to stress and/or structural relaxation. Both modeling and experimental measurements were conducted on multiple GtM seals constructed with CGI 930 glass with purposely added alumina particles. The alumina particles have an established Cr fluorescence pattern and the shift in position of these peaks can accurately measure the strain of the alumina crystals. Photoluminescence spectroscopy (PLS) technique was utilized due to its non-destructive nature and high spatial resolution. PLS maps of these components were analyzed and compared to the models developed previously. The alumina was varied from 1–15 vol% to study the effect of alumina content on the response of the Cr fluorescence peak. Sandia National Laboratories is a multiprogram laboratory managed and operated by National Technology & Engineering Solutions of Sandia, LLC (NTESS), for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525. SAND NO. 2019-9715 A.

10:30 AM

(ICACC-S1-052-2020) Modeling the effects of transverse matrix crack damage accumulation and microstructure on electrical resistivity of melt-infiltrated ceramic matrix composites

M. P. Appleby*[1]; E. Maillet[2]; G. N. Morscher[3]

1. NASA Glenn Research Center, USA
2. GE Research, USA
3. University of Akron, Mechanical Engineering Dept., USA

Recent studies have demonstrated the potential of employing electrical resistivity (ER) measurements as a health monitoring and inspection technique for ceramic matrix composite (CMC) components. Critical to the implementation of ER for CMC material characterization is an understanding of the complex coupled electrical-mechanical response to damage accumulation as well as the effect of composite microstructure and constituent contribution to composite ER. The present work develops a physics-based microscale model that predicts the mechanical and electrical behavior of melt-infiltrated (MI) CMC specimens subjected to progressive transverse matrix crack accumulation. The overall goal is to better understand and validate the relationship between damage mechanisms and electrical resistance, allowing for predictive modeling of varying laminate architectures and load histories.

10:50 AM

(ICACC-S1-053-2020) Acoustic Emission Monitoring of Microcracking in Woven SIC/SIC Ceramic Matrix Composites

P. Ealy*[1]; G. N. Morscher[2]; A. Ritchey[3]

1. University of Akron, Mechanical, USA
2. University of Akron, Mechanical Engineering Dept., USA
3. Rolls-Royce, USA

Gas turbine engine developments have favored the use of lightweight materials with a focus on higher firing temperatures to increase turbine efficiency. Presently, Nickel based superalloys have reached their use limits due to ever increasing firing temperatures and the limit of advanced cooling technologies. Ceramic matrix composites have focused as the optimal materials to solve this problem due to high specific strength and high temperature capabilities. These characteristics make ceramic matrix composites as prime candidates for use in aerospace turbine engines. Furthermore, to use these materials in jet engines damage progression and failure properties must be characterized using a variety of techniques. In this study, woven SIC/SIC ceramic matrix composites are subjected to monotonic tension and 4-point flexural tests for the purpose of monitoring micro-fracture mechanisms via acoustic emission (AE). Multisensor approaches were used in order to isolate and detect early stage damage. Advanced time-of-arrival and location techniques were used to locate sources. Waveform analysis was used to characterize and identify sources.

11:10 AM

(ICACC-S1-054-2020) Flexural Strength of CMC Tubes Used as Components in Nuclear Applications: ASTM Draft Standard Using Transverse Loading for Flexural Behaviour

M. G. Jenkins*[1]; J. E. Gallego[1]

1. Bothell Engineering and Science Technologies, USA

Plans are afoot by US DOE to use advanced materials for core and reactor-unit components in various advanced reactor concepts. Ceramic matrix composites (CMC), specifically SiC fibre-SiC matrix composites, could greatly expand the design window for various components in terms of operating temperatures, application stresses, and service lives, as compared to heat-resistant metallic alloys, while significantly improving accident tolerance and safety margins. Examples of CMC tubular components include fuel rods, control rod sleeves, and control rod joints. Possible failure modes for these components include axial and hoop tension, axial flexure, axial and diametral compression, and axial shear. A draft ASTM standard test method has been developed and submitted for full-consensus ballot to determine the flexural behaviour of ceramic matrix composite tubes subjected to bending moments produced by transverse loading. Empirical tests of composite tubes provided validation of the parameters specified in the test method. The draft standard test method addresses the following experimental issues -- test specimen geometries/preparation, test fixtures, test equipment, interferences, testing modes/procedures, data collection, calculations, reporting requirements, precision/bias.

11:30 AM

(ICACC-S1-055-2020) Life Prediction of Carbon-Reinforced CMCs in Oxidizing Environment Using Electrical Resistance

R. Mansour*[1]

1. Teledyne Scientific Company, Composite Materials, USA

The use of ceramic matrix composites components in aeronautics applications requires a thorough understanding of their behavior under oxidizing environments. Due to their low oxidation resistance, carbon fiber oxidation rate is an important parameter in the development of any lifing model to estimate the service life of these materials. In this respect, the oxidation rate of carbon-based CMCs was determined during creep testing at 800°C in air using electrical resistance. To understand the effect of the composite matrix on the oxidation of carbon fibers, two matrices were investigated in this work: bare CVI-SiC matrix and CVI-SiC matric followed by PIP SiC infiltration. In both cases, dominant increase in electrical resistance was observed prior to ultimate failure.

11:30 AM

(ICACC-S1-056-2020) Real Time Imaging of the Failure Process in WHIPOX Alumina CMCs Using High Temperature X-ray Tomography

D. L. Liu*[1]; J. Ell[2]; H. Barnard[2]; S. Reh[3]; R. O. Ritchie[2]

1. University of Bristol, School of Physics, United Kingdom
2. Lawrence Berkeley National Laboratory, USA
3. DLR - German Aerospace Center, Germany

German Aerospace Center (DLR) developed three types of oxide ceramic-matrix composites (CMCs) including WHIPOX, UMOX and OXIPOL. The latter two types were made by polymer infiltration and pyrolysis, while WHIPOX (Wound Highly Porous Oxide) was a Alumina based CMC using continuous fibre bundle infiltration and winding process. This work studies the failure process of WHIPOX CMC under bending over a range of temperatures (RT, 900°C, 1050°C and 1200°C) in Air using synchrotron X-ray computed micro-tomography (XCT). First of all, the measured flexural strength was consistent with previous uniaxial tension tests indicating the size of the samples used was adequate. Secondly, the RT failure comprised of diffused vertical cracks due to fibre breakage accompanied by fibre/matrix delamination linking up existing matrix pores. At elevated temperatures, e.g., 1050 and 1200°C, fibre/matrix delamination became the primary failure mechanism. As multiple XCT scans were captured during bending to failure, digital volume correlation (both local and global approaches) was used to derive the 3D displacement field hence stress-strain behaviour at different temperatures. Finally, a systematic mapping of the residual stresses in the fibre and matrix were acquired and the relaxation of these stresses was considered to be one of the contributing factors to the high temperature delamination.

S3: 17th International Symposium on Solid Oxide Cells (SOC): Materials, Science and Technology

Electrodes Development

Room: Crystal

Session Chair: Shiwoo Lee, National Energy Technology Laboratory

8:30 AM

(ICACC-S3-045-2020) An Active and Resilient Isostructured Bilayer Oxygen Electrode for Intermediate-Temperature Reversible Solid Oxide Cells (Invited)

K. Huang*[1]

1. University of South Carolina, Mechanical Engineering, USA

Reducing the resistances of oxygen reduction reaction (ORR) and oxygen evolution reaction (OER) while retaining the stability of an oxygen electrode, even in the presence of air contaminants such as Cr, H_2O and CO_2, is of paramount importance to the current commercial development of intermediate-temperature reversible solid oxide cells (IT-RSOCs). In this presentation, a multifunctional, coarsening-resistant, air-contaminants-tolerant and yet ORR/OER-active oxygen electrode (OE) for IT-RSOCs is described. The testing results show that new bilayer OE exhibits a much better ORR/OER activity and stability than the single-layer benchmark LSCF-GDC OE. Remarkably, the new bilayer OE also demonstrates an exceptionally high resistance to Cr-attack, a critical issue to the development of commercial meal-interconnect loaded SOFC/SOEC stacks/systems. Theoretical analysis is also presented to understand the fundamental reasons for the improved ORR/OER activity and stability observed under real-world operating conditions.

9:00 AM

(ICACC-S3-046-2020) Chemically Assisted Electrodeposition: A Facile and Versatile Route to Prepare Perovskite Oxide Thin Films and Nanostructures for SOFC Applications (Invited)

J. Lee*[1]

1. Chosun University, Materials Science and Engineering, Republic of Korea

Perovskite-type metal oxides possess attractive features, including high conductivity (ionic, electronic, or mixed), electrocatalytic activity, and chemical/structural stability, which make them suitable for use in solid oxide fuel cells (SOFCs). Electrodeposition has attracted significant attention as a promising approach to synthesizing high-quality metal oxides. This method allows the preparation of various oxide materials, including perovskite oxides, with tailored physicochemical properties suitable for their specific applications by controlling deposition parameters. In this study, we demonstrate the fabrication of perovskite-type $LaMO_3$ (M = Mn, Co) thin films and nanostructures via a facile electrochemical route. The proposed method involves the "chemically assisted electrodeposition" of hydroxide (electrochemical nitrate reduction and in-situ hydroxide co-precipitation), followed by thermal conversion of hydroxide to oxide. Our experimental results demonstrate that the composition, nanostructure, and morphology of the perovskite metal oxides strongly depend on the electrodeposition parameters, such as the precursor concentration, applied current density, and solution

pH. The materials and electrochemical properties of the fabricated perovskite oxides are studied, and then, their feasibility in SOFCs is discussed.

9:30 AM

(ICACC-S3-047-2020) Electrocatalytically Active Cathode Interlayers for IT-SOFC Prepared by Spray Pyrolysis

B. Kamecki*[2]; J. Karczewski[1]; P. Z. Jasinski[2]; S. Molin[2]

1. Gdansk University of Technology, Poland
2. Gdansk University of Technology, Laboratory of Functional Materials, Faculty of Electronics, Telecommunications and Informatics, Poland

Solid Oxide Fuel Cells performance strongly depends on efficient cathode materials and their microstructure. Electrode-electrolyte interface plays a crucial role in expediting the oxygen reduction reaction (ORR) kinetics of intermediate-temperature solid oxide fuel cells (IT-SOFCs). One of the possible approaches to increase the electrochemical activity of IT-SOFCs is introducing high electrocatalytically active interlayers between the oxygen electrode and the solid electrolyte. In this research, spray pyrolysis was used to produce thin electroactive layers of various materials, such as $Sm_{0.5}Sr_{0.5}CoO_{3-\delta}$, $La_2NiO_{4+\delta}$, $Pr_2NiO_{4+\delta}$, $PrBaCo_2O_{5+\delta}$, $La_{0.6}Sr_{0.4}CoO_{3-\delta}$, $La_{0.6}Sr_{0.4}FeO_{3-\delta}$, $La_{0.8}Sr_{0.2}Co_{0.2}Fe_{0.8}O_{3-\delta}$, on the electrode-electrolyte interface. The influence of the microstructure on the ohmic and polarization resistances has been investigated by impedance spectroscopy of symmetrical electrodes in the temperature range of 500-700°C in wide range of oxygen partial pressures. Distribution of relaxation times (DRT) analysis was applied to impedance data and enabled the quantitative deconvolution of spectra and allows to determine individual reactions/processes occurring at the oxygen electrode. The presented research is part of the project, carried out within First TEAM programme of the Foundation for Polish Science (grant agreement nr. POIR.04.04.00-00-42E9/17-00).

9:50 AM

(ICACC-S3-048-2020) Activation energies of oxidation and diffusion of $(La_{1-x}Sr_x)Ni_{0.9}Mn_{0.1}O_{4+\delta}$

Y. Sadia*[1]; M. A. Niania[2]; S. Skinner[2]

1. Ben-Gurion University of the Negev, Material Engneering, Israel
2. Imperial College London, United Kingdom

One of the biggest obstacles to intermediate temperature SOFCs is a good cathode which is compatible with common electrolytes. La_2NiO_4 (LNO) is such a cathode showing both chemical and mechanical compatibility with common electrolytes such as CGO and LSGM. However, any attempt to increase the performance on LNO by doping has proved insufficient, increasing one property but reducing another. In this study we attempt to elucidate the effect of changing the Sr content on Sr and Mn doped LNO. Samples containing 10-50% Sr were created using a citric acid sol gel route and their electrical properties have been studied. The impedance behaviour of half-cells was measured with LSGM electrolyte. In addition, the oxygen diffusion was tested using $^{18}O_2$ isotope exchange. The influence of combined Mn and Sr content on the oxygen exchange kinetics and oxygen diffusion kinetics was determined using exchange results, DRT analysis of the impedance results and traditional impedance spectroscopy techniques.

10:30 AM

(ICACC-S3-049-2020) Study of Direct Electrochemical Oxidation of Methane at Ceria/gas Interface (Invited)

W. Jung*[1]

1. Korea Advanced Institute of Science and Engineering (KAIST), DMSE, Republic of Korea

One of the key advantages of solid oxide fuel cells is that they can use hydrocarbon fuels in addition to pure hydrogen, in principle, without additional reforming systems, and $CeO_{2-\delta}$ (ceria)-based oxides play an important role as a catalyst for hydrocarbon activation and as an inhibitor of carbon coking. However, even for the simplest hydrocarbon molecule, CH_4, the mechanism of electrochemical oxidation on the ceria surface remains largely unknown. This has partly been due to the difficulty to monitor the targeted electrochemical reactions from various chemical reactions occurring simultaneously in typical porous metal/ceria composite electrodes. Here, we present a polarizable, Sm-doped ceria thin-film model electrochemical cells capable of selectively investigating CH_4 direct-oxidation on the ceria surface. Combined in operando X-ray photoelectron spectroscopy and DFT calculation reveal that the ceria surface catalyzes the C-H cleavage and the overall electrode reaction rate is dominantly determined by the H_2O formation step. These observations end the longstanding academic debate over the direct use of CH_4 and provide an ideal electrode design for high performance.

11:00 AM

(ICACC-S3-050-2020) Nano-metal socketed electrode for high perfomance RSOCs (Invited)

J. Myung*[1]

1. Incheon national university, Dept. of Materials Science and Engineering, Republic of Korea

Reversible solid oxide cells (RSOCs) operated in severe conditions (low/high Po2, high temperature, high steam), the ideal fuel electrode materials has not been found yet to accomplish of its requirements such as electronic and ionic conductivity, and catalytic activity with strong stability in redox condition. Conventional Ni-YSZ cermet has been regarded as the embodiment of these functionalities, but the electrode still suffered from agglomeration and oxidation during redox condition. Recent nano-structured electrode designs have been developed to microstructures consisting of a porous mixed ionic electronic conductor backbone decorated with metallic nanoparticles by impregnation method. However, these are still hindered by the lack of cost and time-effective methods. Here, a simpler alternative, will be introduced, exsolution whereby the catalytically active metal is substituted in the crystal lattice of the backbone in oxidizing conditions and exsolved on the surface as designed metal particles under reduction condition. Here I demonstrated a simple and highly effective in situ method for producing nanostructured electrodes capable of delivering high performances in both fuel cell and electrolysis mode.

11:30 AM

(ICACC-S3-051-2020) Enhancing Low-Temperature Solid Oxide Fuel Cell Performance and Durability by Tuning Surface Chemistry

Y. Huang*[1]; I. Robinson[1]; E. Ostrovskiy[1]; S. Horlick[1]; E. D. Wachsman[1]

1. University of Maryland, USA

Solid oxide fuel cells (SOFCs) are solid-state devices that can convert electrochemical potential to electricity with high efficiency and fuel flexibility. However, the high polarization resistance and durability of cathodes are key factors currently limiting the commercial deployment of SOFCs. In particular, insufficient cathode activity and surface cation precipitation after the long-term operation are considered as main challenges for low-temperature SOFCs. Here we show different cation segregation processes on common low-temperature SOFC cathodes, perovskite $La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_3$ (LSCF), $Sm_{0.5}Sr_{0.5}CoO_3$ (SSC), and double perovskite $PrBa_{0.5}Sr_{0.5}Co_{1.5}Fe_{0.5}O_6$ (PBSCF) over a wide range of operating conditions, and the effects of temperature, time, oxygen partial pressure, gaseous contaminant (CO_2), and material stoichiometry are shown. The cation segregation mechanism is further discussed. Also, we demonstrated the performance and the long-term stability can be significantly enhanced by modifying the cathode surface. After surface modification, the cathode area specific resistance (ASR) is reduced over an order of magnitude with high stability for over 1000 hours.

11:50 AM

(ICACC-S3-052-2020) Structure control of Ni impregnated YSZ using spark plasma sintering (SPS)

Y. Sadia*[1]; N. Madar[1]; S. Kalabukhov[1]; Y. Gelbstein[1]; N. Frage[1]

1. Ben-Gurion University of the Negev, Material Engneering, Israel

Ni impregnated anodes are the most common type of anodes used in solid oxide fuel cells. While these anodes are commonly fabricated using tape casting, screen printing or similar processes, fine control of the microstructure remains a major goal. One way to control porosity is through very fast powder metallurgy techniques. One such technique, which allows for very fast densification and a high level of porosity and the microstructure control is SPS. In this study, several samples of Ni-impregnated YSZ anodes were fabricated using both a new SPS approach and by a conventional tape casting method. The resulting samples were tested using BET, EIS and SEM techniques. Comparison of these approaches for SOFC anodes fabrication will be discussed.

Proton Conducting Fuel Cells

Room: Crystal

Session Chair: Mihails Kusnezoff, Fraunhofer IKTS

1:30 PM

(ICACC-S3-053-2020) Promising nanocomposites materials for oxygen- and proton-conducting membranes: Structural and transport properties, performance of catalytic membrane reactors (Invited)

Y. N. Bespalko*[1]; N. F. Eremeev[1]; V. A. Sadykov[1]

1. Boreskov Institute of Catalysis, Heterogeneous catalysis, Russian Federation

Design of compact reactors based on selective oxygen permeable membranes separating O_2 from air for fuels oxidation as well as hydrogen permeable membranes producing pure H_2 from a mixture of fuel reforming products now attracts a lot of attention. This work reviews results of research aimed at design of inexpensive and robust nanocomposite materials for such membranes, optimizing their composition, real/nanostructure and texture to ensure a high oxygen/hydrogen mobility, namely, O/H self-diffusion coefficients estimated by detailed mathematical analysis of temperature –programmed isotope heteroexchange of their lattice oxygen with $C^{18}O_2$, and hydrogen –with D_2 in static/flow reactors. The highest O_2 mobility was provided by $PrNi_{0.5}Co_{0.5}O_3$–$Ce_{0.9}Y_{0.1}O_2$ nanocomposites, while that of H_2 – by nanocomposites comprised of (Ni, Cu) alloys + doped rare-earth niobates or tungstates-molybdates. Asymmetric supported membranes were constructed by supporting nanocomposite layers on Ni-Al alloy open foam substrates with catalytic layers on the fuel side. Catalytic membrane reactors demonstrated a high oxygen/hydrogen permeability along with a high activity and stability in real conditions of fuels reforming, thus being promising for the practical application. Supported by the Russian Science Foundation Project 16-13- 00112

2:00 PM

(ICACC-S3-054-2020) Ceramic Proton Conductors: From Energy Conversion to Green Chemistry

M. E. Ivanova*[1]; W. Deibert[1]; C. Lenser[1]; N. H. Menzler[1]; O. Guillon[2]

1. Forschungszentrum Juelich GmbH, IEK-1, Germany
2. Forschungszentrum Juelich, IEK-1, Germany

The global effort for reaching highly efficient CO_2-neutral energy supply requires innovative technological solutions based on high performance materials. Ceramic proton conductors (PC) with tailored properties gain increasing scientific and industrial interest due to their multifaceted low temperature applications. Reversible PC electrolysis/fuel cell devices can convert and store electrical surplus from renewables into H_2 or NH_3, which is then used as fuel when power generation is required. Renewably electrified PC co-electrolysers utilize CO_2 emissions electrochemically to i.e. CH_4, CH_3OH, or Syngas, while natural gas fuelled PC cells can co-generate electricity and valuable chemicals, e.g. olefins or aromatics (i.e. C_2H_4, C_2H_2, or C_6H_6). Such concepts of ceramic PC integration offer significant process intensification resulting in higher overall energy efficiency, products selectivity and yields. The present talk will give an overview on different functional and utilization aspects of selected proton conductors (e.g. $BaZr_{1-x}(Ce,Y)_xO_{3-\delta}$, $La_{5.5}WO_{12-\delta}$) and ceramic composites ($BaZr_{1-x}(Ce,M)_xO_{3-\delta}$:$Ce_{1-x}M_xO_{2-\delta}$) in the light of achieving green and sustainable economy.

2:20 PM

(ICACC-S3-055-2020) Bilayer Electrolyte for Proton Conducting Solid Oxide Electrolysis Cells

H. Tian*[1]; W. Li[1]; G. Bo[1]; L. Ma[1]; X. Liu[2]

1. West Virginia University, USA
2. West Virginia University, Mechanical & Aerospace Engineering, USA

Proton conductors $BaZr_{0.1}Ce_{0.7}Y_{0.1}Yb_{0.1}O_{3-\delta}$ (BZCYYb) and $La_2Ce_2O_7$ (LCO) are combined to create an interface active and steam-tolerant electrolyte for high-performance proton-conducting solid oxide electrolysis cells. The readily fabricated LCO/BZCYYb bilayer electrolyte can be densified at a temperature of as low as 1300 °C versus ~1600 °C for the benchmark steam-stable $BaZr_{0.8}Y_{0.2}O_{3-\delta}$ electrolyte. With $Pr_2NiO_{4+\delta}$ as the anode and Ni as the cathode catalyst, the cell yields a current density of 975 and 300 mA/cm^2 under a 1.3 V applied potential at 700 and 600 °C, respectively. This performance is among the best of all H-SOECs equipped with a chemically stable electrolyte so far. The LCO layer effectively protects the BZCYYb layer from the high concentration of steam in a practical SOEC operation condition. The cell without the LCO layer shows degradation in terms of an increased electrolyzing potential from 1.07 to 1.29 V during a constant 400 mA/cm^2 operation at 700 °C. In contrast, the bilayer electrolyte cell maintains the same electrolyzing potential of 1.13 V under the same conduction for a 102 h operation. These findings demonstrate that this synergic bilayer electrolyte design is a vital strategy to overcome the dilemma between performance and stability faced by the current benchmark Zr- or Ce-rich Ba(CeZr)$O_{3-\delta}$ electrolysis cells to achieve excellent performance and stability at the same time.

2:40 PM

(ICACC-S3-056-2020) Microstructure evolution in nickel and yttrium doped barium zirconate thin-films

D. Jennings*[1]; S. Ricote[2]; J. Santisó[4]; I. Reimanis[3]

1. Colorado School of Mines, Materials and Metallurgical Engineering, USA
2. Colorado School of Mines, Mechanical Engineering, USA
3. Colorado School of Mines, USA
4. Catalan Institute of Nanoscience and Nanotechnology, Spain

Yttrium doped barium zirconate (BZY) thin films with nickel ($BaZr_{0.81}Y_{0.15}Ni_{0.04}O_{3-\delta}$) and without nickel ($BaZr_{0.85}Y_{0.15}O_{3-\delta}$) were deposited on single crystal strontium titanate with the purpose to study metallic nickel exsolution at elevated temperatures under reducing conditions. Time of flight secondary ion mass spectrometry is used to obtain concentration profiles through the film thickness to quantify exsolution kinetics. High resolution transmission electron microscopy provides insight into the nucleation and growth of metallic nickel particles on the BZY surface. A unique crystallographic orientation relationship between nickel and BZY is observed. It was also observed that the thin films are unstable in relatively oxidizing environments, decomposing during a heat treatment at 800 °C in argon. The morphology of the decomposed film and the mechanism of decomposition in thin-film BZY samples are discussed.

S6: Advanced Materials and Technologies for Rechargeable Energy Storage

Na-ion Battery

Room: Tomoka A
Session Chair: Sevi Murugavel, University of Delhi

8:30 AM

(ICACC-S6-038-2020) Size Induced Structural Changes and Charge Transport Mechanism in maricite-$NaFePO_4$: An In-Depth Study by Experimental and Simulations (Invited)

S. Murugavel*[1]

1. University of Delhi, Physics and Astrophysics, India

The development of new and novel electrode materials for energy storage devices has become an intensive research by materials science community because of its importance in portable electronic devices. As per the global demands for the requirement of energy storage/supply increasing exponentially, the prices of the lithium ion batteries are also rising due to limited resources of lithium in the earth crust. In this regard, sodium-based cathode material could be the alternative choice for the next generation of rechargeable batteries. In this context, we have carried out systematic crystallite size dependent structural and charge transport investigations on maricite-$NaFePO_4$ (m-NFP) through experimental and theoretical simulation. Rietveld refinement analysis reveals that decrease in the unit cell parameters which lead to the volume contraction upon reduction in crystallite size. We observe polaronic conductivity enhancement of approximately an order of magnitude at the nanoscale level as compared with its bulk counterpart. In parallel, we performed DFT calculations on m-NFP with different sodium ion vacancy concentrations. The obtained structural parameters are in excellent agreement with the experimental results. The nanocrystalline m-NFP with better kinetics will open the new avenue for its usage as cathode material in sodium ion batteries.

9:00 AM

(ICACC-S6-039-2020) Understanding the reversible anionic redox in new layered Na-ion cathodes (Invited)

J. Liu*[1]

1. Oak Ridge National Laboratory, USA

The large voltage hysteresis is one of the biggest barriers to realizing reversible lattice oxygen redox for Li/Na-ion cathodes with high energy density. Great efforts have been devoted to exploring novel cathode systems where intrinsic reversible oxygen redox chemistry can be realized with small voltage hysteresis. More recently, in order to stabilize the cathode structure upon electrochemical cycling by tuning the covalency of metal oxygen bonds, 4d and 5d transition metal have been introduced to both Li-ion batteries and Na-ion batteries. It is thus important and timely to extend these concepts to the low-cost 3d transtion metal-based cathodes. Very recently, a few new P3-type layered oxides, such as $Na_2Mn_3O_7$ have been reported to be able to dramatically suppress the voltage hysteresis when using lattice anionic redox. However, the structure and electronic structure origin of this small voltage hysteresis has not been well understood. In this presentation, through systematic studies using EPR, in situ XRD and neutron pair distribution function analysis, a unified theory will be proposed to explain the observed small voltage hysteresis in the two compounds mentioned above. I will also briefly discuss the interesting structure evolution for these compounds during the initial charge and discharge.

9:30 AM

(ICACC-S6-040-2020) Solid State NMR Characterization of Vanadium Fluorophosphates for Na-ion Batteries (Invited)

H. Nguyen[1]; P. Sanz Camacho[1]; J. Olchowka[1]; C. Masquelier[3]; L. Croguennec[2]; D. Carlier*[1]

1. ICMCB, France
2. ICMCB-CNRS, France
3. LRCS, France

In the scope of finding new positive electrode materials for Li-ion or Na-ion batteries, the deep understanding of the link between their structure, electronic structure and electrochemical behavior is crucial. As the presence of defects or disorder may play a critical role, a local characterization of the materials is highly required. To that extent, Magic Angle Spinning Nuclear Magnetic Resonance (MAS-NMR) appeared to be a key tool. For paramagnetic materials, it allows to probe both, the local structure and the local electronic structure thanks to the Fermi contact interaction. In order to assign the signals and understand the spin transfer mechanism through the chemical bonds, we have been developed the use of ab initio calculations for some years. Some recent studies of the characterization of defects or disorder in layered oxides or V-phosphate materials will be presented. Using MAS-NMR we showed that $Na_3V_2(PO_4)_2F_3$, which are promising materials for positive electrode application in Na-ion batteries, exhibit some O-defects leading to the formation of V^{4+} ions locally. These V^{4+} ions are forming a vanadyl-type bond with the defect O, and affect the electrochemical cycling performances. Then we study the whole solid solution series $Na_3V_2(PO_4)_2F_{3-y}O_y$ and other $Na_3(V,M)_2(PO_4)_2F_{3-y}O_y$ materials with M = Fe or Al and discuss the local ionic arrangement.

10:20 AM

(ICACC-S6-041-2020) P3-$Na_{0.8}Fe_{0.5}Mn_{0.5}O_2$ layered oxide as cathode material for Na-ion batteries

A. Tripathi[1]; S. G. Reddy[1]; P. Balaya*[1]

1. National University of Singapore, Department of Mechanical Engineering, Singapore

Layered oxides have been rigorously evaluated as potential cathode materials for Na-ion batteries. Amongst them, O3 and P2 type oxides have been largely the focus of such studies[1-3]. This work aims at studying P3-$Na_{0.8}Fe_{0.5}Mn_{0.5}O_2$ (P3-NFM) as a possible cathode material for Na-ion battery. P3-NFM delivers a discharge capacity of 150 mAh/g at 0.02 A/g in a wide voltage window. The storage performance is considerably improved using a modified cycling protocol, which is a combination of wide and narrow voltage windows during formation and subsequent cycles. When operated in such a modified voltage window, the material delivers a capacity of 120 mAh/g with a capacity retention of 90% up to 50 cycles, without using any additive in electrolyte. Unlike other high-performing P-type layered oxides which require pre-sodiation as they are Na-deficient in pristine state, P3-NFM does not require usage of sacrificial salts for full cells as it Na-sufficient in its pristine state itself. Operando XRD of P3-NFM battery reveals reversible layered to rock-salt type phase (layered + rock-salt) during battery operation. This makes P3-NFM to be a model material to evaluate the reasons of reversibility in layered to rock-salt type phase transformations.

10:40 AM

(ICACC-S6-042-2020) Mastering of particle size and morphology of the puckered layer γ′-V_2O_5 polymorph for enhanced Na electrochemical properties

R. Baddour-Hadjean*[1]; N. Emery[1]; B. Laik[1]; D. Batyrbekuly[1]; z. Bakenov[2]; J. Pereira-Ramos[1]

1. CNRS, ICMPE, France
2. National Laboratory Astana, Center for Energy and Advanced Materials Science, Kazakhstan

Due to the cost and low availability of Li sources, NIBs are attracting considerable interest as tomorrow's world batteries. Here we report the interesting capability of γ′-V_2O_5 toward Na insertion. Nearly 1 Na^+/ mole (145 mAh g^{-1}) can be inserted in γ′-V_2O_5 at a high working potential of 3.3 V. However, only a 50% efficiency is observed during the first charge process. Further cycles exhibit an excellent capacity retention (70 mAh g^{-1} after 70 cycles at C/10). To solve the charge efficiency issue, a downsizing approach was performed using planetary ball milling. A strong effect of the crystallite size (90 nm for the as prepared sample vs. 35 nm for the ball-milled powder) is observed on the shape of the voltage-composition curve. Also, the charge efficiency is strongly improved (90% efficiency, 127 mAh g^{-1}). A structural study reveals a nano-size effect that promotes a wide single phase domain at the expense of diphasic region for the ball milled sample. A mastering of the particle morphology has also been conducted: coral-like porous γ′-V_2O_5 powder leads to a quantitative charge process at a high voltage of 3.4 V, enhanced rate capability and excellent cycle life (130 mAh g^{-1} after 60 cycles at C/10).

11:00 AM

(ICACC-S6-043-2020) Active materials for Na ion batteries

M. Balordi[1]; M. Bini[2]; M. Broglia*[1]; G. Carini[1]; D. Capsoni[2]; F. Cernuschi[1]; A. Gentile[3]; S. Marchionna[1]; M. Nuti[2]; I. Quinzeni[2]; R. Ruffo[3]

1. RSE - Ricerca Sistema Energetico, Italy
2. Università degli Studi di Pavia, Chemistry Department, Italy
3. Università degli Studi Milano Bicocca, Materials Science Department, Italy

In view of a high demand increase of batteries for EV and stationary electrochemical storage, earth abundant and environmental friendly active materials for batteries have to be developed. Na ion batteries (NIB) is an interesting alternative to Li ion batteries, especially for stationary applications. Recently, MXenes have been proposed as anodic material for NIB devices. In this work, the preliminary results of an experimental activity aimed to study the effects of the process parameters (i.e. MAX phase synthesis parameters by SPS and the subsequent chemical exfoliation recipe) on the electrochemical performances of MXenes (Ti3C2Tx) based NIB half-cell will be presented. For the cathodic compart, the eco sustainable material Na0.44MnO2 was chosen because of its unique three-dimensional tunnel-structure able to tolerate stresses during the insertion/ de insertion of sodium, that creates structural changes. In this work, multi-angular rod-shaped crystals Na0.44MnO2 have been synthesized and electrochemically tested in half –cell. Furthermore to improve stability, capacity and electronic conductivity of Na0.44Mn O2 part of the Mn was substituted with eco compatible elements like Fe, Ti, Si and Cu. All the samples were widely characterized from a structural, morphological and spectroscopic point of view. Preliminary electrochemical analysis, on half-cells, shows the different electrochemical properties due to the peculiarities of the dopant ion.

Li-ion Battery: Anode Materials and Cathode Materials

Room: Tomoka A

Session Chairs: Palani Balaya, National University of Singapore; Do Kyung Kim, Korea Advanced Institute of Science and Engineering (KAIST)

1:30 PM

(ICACC-S6-044-2020) Nano-engineering and Characterization of Electrode Materials for High-Areal-Capacity and Stable Lithium-Sulfur Batteries (Invited)

J. Yun[1]; R. Ponraj[1]; D. Kim*[1]

1. Korea Advanced Institute of Science and Engineering (KAIST), Dept. of Mater Sci & Eng, Republic of Korea

Developing high-energy-density batteries beyond the state-of-the-art Li-ion batteries is inevitable to meet the energy demand for transportation and grid energy storage. Lithium-sulfur (Li-S) electrochemistry has the high theoretical capacity (1,675 mAh g^{-1}) and gravimetric energy density (2,500 Wh kg^{-1}) promising next generation high energy system. Despite the advantages of Li-S, the practical application of Li-S batteries is hampered by following drawbacks; i) low areal sulfur loading (<2 mg cm^{-2}) and resulting areal capacity (<3 mAh cm^{-2}) of cathode emanated from low electrical conductivity of elemental sulfur, ii) polysulfide dissolution in electrolyte during electrochemical reaction nibbling the capacity, and iii) lithium dendrite growth onto anode causing short-circuit current. These issues can be treated by nano-structural engineering of electrode materials which advance the electrochemical reaction of Li-S. Herein, we introduce the strategies to develop the performance of Li-S by architecture of porous carbon materials and use of metal oxide, expecting catalyst-assisted electrochemical reaction which facilitates the sulfur utilization and polysulfide chemisorption. In this talk, we try our best to explaining suitable ways for high performance Li-S batteries.

2:00 PM

(ICACC-S6-045-2020) Solvent-Free Preparation of High Energy, Binder-Free Electrodes Enabled by Dry Compressible Holey Graphene (Invited)

Y. Lin*[1]; J. W. Connell[2]

1. National Institute of Aerospace, USA
2. NASA Langley Research Center, Advanced Materials and Processing Branch, USA

Graphene is an atomically thick sheet consisting of a graphitic carbon network with excellent mechanical strength, electrical and thermal conductivity, and chemical stability. Holey graphene, a structural derivative of graphene, has an array of through-the-thickness holes across the lateral surface of the nanosheet. The presence of these holes has minimal detrimental effect on the graphene properties and leads to enhanced performance in applications such as electronics, sensors, and energy storage. For example, these holes allow more facile cross-plane ion and gas transport than intact graphene, making holey graphene an ideal electrode material for electrochemical energy storage. This presentation will focus on the ability of holey graphene to be compression molded into robust articles or architectures under solvent-free conditions without the need for potentially parasitic binders. The unique dry compressibility of holey graphene has enabled facile fabrication of high mass loading electrodes with both high density and high porosity, which have found use in supercapacitors and various high-energy battery systems such as lithium-oxygen, lithium-sulfur, and lithium-selenium batteries.

2:30 PM

(ICACC-S6-046-2020) Is n- and p- substitutionally doped C60 a promising material for Li ion batteries? A mechanistic study

Y. Chen[2]; C. Cho[3]; S. Manzhos*[1]

1. INRS, EMT, Canada
2. National University of Singapore, Singapore
3. Pusan National University, Republic of Korea

Fullerene-based materials including C60 and doped C60 have previously been proposed as anodes for lithium ion batteries. It was also shown earlier that n- and p-doping of small molecules can substantially increase voltages and specific capacities. Recent literature suggested that the voltage-capacity curve for lithitaition of C could also be modulated by doping. Here, we study ab initio the attachment of multiple lithium atoms to C60, substitutionally nitrogen-doped C60 (n-type), and boron doped C60 (p-type). We relate the observed attachment energies (which determine the voltage) to changes in the electronic structure induced by Li attachment and by doping. We compare results with a GGA functional and a hybrid functional and show that while they agree semi-quantitatively with respect to the expected voltages, there are qualitative differences in the electronic structure. We show that, contrary to small molecules, single atom n- and p-doping will not lead to practically useful modulation of the voltage-capacity curve beyond the initial stages of lithiation. We also show that using a molecular model, while neglecting aggregate state effects, it is possible to estimate and compare the shape of the voltage-capacity curve as well as to call the final state of charge.

2:50 PM

(ICACC-S6-047-2020) Why ALD Nanofilms on Cathode Materials Improve Li-ion Battery Performance

A. Hoskins[1]; W. W. McNeary[2]; S. Millican[3]; X. Liang[4]; A. W. Weimer*[2]

1. University of Colorado Boulder, Chemical Engineering, USA
2. University of Colorado, Boulder, Chemical and Biological Engineering, USA
3. University of Colorado, Department of Chemical and Biological Engineering, USA
4. Missouri University of Science & Technology, USA

The deposition of alumina ALD films on Li ion battery cathodes is known to enhance the cycling stability of lithium ion batteries and is commonly assumed to form a uniform film that optimally is thin enough to facilitate lithium diffusion while blocking side reactions of the electrolyte with the cathode material. Here, we elucidate the nature of low-cycle number ALD films on lithium nickel manganese cobalt oxide (NMC) cathode materials. Cathode particles were coated with ALD films deposited using various cycle numbers, and then studied using low energy ion scattering (LEIS) and secondary ion mass spectroscopy (SIMS). Surface analysis showed that low-cycle ALD films are not uniform nor uniformly thick over the surface of the NMC particles and that alumina ALD preferentially deposits on transition metal bound sites on the cathode surface and coats Li on the surface to a lesser extent. Contrary to current supposition, low-cycle ALD can improve the cycling stability of battery cathode materials through this preferential growth that stabilizes the transition metal oxides in the presence of electrolyte without blocking lithium intercalation pathways. This is the first study to determine that Li remains exposed on the as-synthesized surface of ALD coated cathode particles and that the ALD film is non-uniform and non-uniformly thick.

3:30 PM

(ICACC-S6-048-2020) Performance of High Energy and High Power Lithium-Ion Cells (Invited)

R. Bugga*[1]; F. C. Krause[1]; E. J. Brandon[1]

1. Jet Propulsion Laboratory, Electrochemical Technologies, USA

Li-ion batteries are being routinely used in planetary exploration missions, owing to their impressive performance characteristics, including high specific energy, long life and good low temperature performance. Historically, batteries with custom, large-format cells of 10-50 Ah have been used in various Mars rovers, landers and orbiters. However, due to improved specific energies (~250 Wh/kg), and safety from built-in safety devices, and simplicity in their charge electronics, commercial 18650 Li-ion cells are now being considered for use in future space missions, e.g., Europa Clipper. Several cell types, i.e., with different chemistries and from different manufacturers are currently being evaluated at JPL. The cell chemistries differ mostly in cathodes, i.e., either lithium nickel cobalt aluminum oxide, Li-rich nickel manganese cobalt oxide or blends with lithium manganese spinel oxide. Various performance tests, including cycle life and high power characterization at different temperatures and charge characterization at low temperatures for ascertaining Li plating are being carried out in conjunction with Electrochemical Impedance Spectroscopy measurements. In addition, destructive physical analysis of the cells have been performed to analyze the electrode materials, and correlate them with the perform characteristics.

4:00 PM

(ICACC-S6-049-2020) Early Transition Metal Doped Layered Oxide Lithium-ion Cathodes with No Cobalt (Invited)

J. Nanda*[1]; E. Self[1]; D. Darbar[1]; I. Belharouak[1]

1. Oak Ridge National Lab, USA

Lithium based transition metal (TM) based oxides are commercially used as cathodes for lithium-ion batteries for electric vehicles. Most common compositions have Ni,Mn and Co with typical ratio of 3-3-3 to 8-1-1 where the later compostion is 80% Ni-rich and 10% Mn and Co respectively. Among these three TM's cobalt is the one that is least abundant and mined only in certian parts of world where maintianing a global supply chain could be a challenge. Given such scenario, there has been a focussed effort to develop cathode compostion that has either very little cobalt or no cobalt. LiNi0.5Mn0.5O2 (LNMO) is a layered cathode material that has been extensively studied till date. One of the major issue with LNMO is Li-Ni exchange between the layers commongly referred to as "antisite effect" that potentially block lithium transport resulting in lower capacity retention and rate. The talk will report some of the progerss made to this effect by substituting small quantity (between 0.5-2 wt%) of early tarnsistion metal such as Mo, Ti and Zr in their respective d^0 oxidation state. For example, doping only 0,5 wt% Mo^{6+} in LMNO has significant effect in capacity retention and rate performance. Detailed investigation on the role on d^o cations in LNMO cathodes based on electron microscopy, X-ray Photoelectron Spectroscopy (XPS), XAS and Oxygen mass spectrometry will be presented.

4:30 PM

(ICACC-S6-050-2020) Surface Energy Measurement of Nanocrystalline $LiNiO_2$ Cathode Material

S. Dahl*[1]; R. Castro[2]

1. University of California, Davis, Chemical Engineering, USA
2. University of California, Davis, Material Science & Engineering, USA

Lithium nickel oxide has beneficial electrochemical properties for cathode materials in lithium ion batteries with theoretically high capacity and better cost efficiency. Nanoparticles have the potential to improve lithium battery technology by enhancing surface area and the kinetics of lithium diffusion. However, synthesis of stoichiometric $LiNiO_2$ nanoparticles is a challenging process due to structural and thermal instabilities. A modified Pechini method with polymeric precursors produced nanocrystalline $LiNiO_2$ with the decomposition and calcination performed under oxygen flux. The synthesis process produced nanoparticles without the highly stable Li_2CO_3 phase through the use of higher calcination temperatures, which is known to deteriorate intercalation properties. However, the decrease in crystallite size to nanometer scale causes an increase in the surface energy of particles which can impact the total energy of the system and stability of the particles. A water adsorption

calorimetric technique was utilized to experimentally quantify the surface energy of nanocrystalline $LiNiO_2$ particles to quantify the expected instability.

S9: Porous Ceramics: Novel Developments and Applications

Engineering Applications of Porous Ceramics II

Room: Coquina Salon F
Session Chair: David Smith, University of Limoges

8:30 AM

(ICACC-S9-010-2020) Interface modification for the adsorption of viruses on porous ceramics structures and nanofibers (Invited)

T. Graule*[1]; K. Domagala[1]; S. Yuezbasi[1]

1. Empa, Laboratory for High Performance Ceramics, Switzerland

Water purification is necessary to prevent the spread of different diseases, which are caused by water-borne germs like bacteria, protozoa, and viruses. Our studies are focusing on the physical separation of germs from water applying nanostructured, ceramic based filters as well as modified fibers originating from CNT's and granular oxide materials. Carmics based virus filtration (MS 2 bacteriophages; size 25 nm, PZC 3) was recently performed with an efficiency of more than 99,9 % by such a modification, the highly active, porous ceramic surface was received in combination with various oxides, but with limited lifetime. As an alternative solution, CNT based nanofibers and granular type, mixed oxides were applied recently for virus filtration, after modification of the nanostructured surface by copper oxide.

9:00 AM

(ICACC-S9-011-2020) Development of Copper (oxide)/alumina Granules for Effective Virus Removal from Water

S. Yuezbasi*[1]; J. Mazurkow[1]; K. Domagala[1]; S. Pfeiffer[1]; T. Graule[1]

1. Empa, Laboratory for High Performance Ceramics, Switzerland

Poor water quality causes risk for public safety and health by distribution of pathogens and pollutants. Conventional filters improve drinking water quality through removal of human pathogenic protozoa and bacteria; however, most of them are insufficient in removal of viruses (with a typical size of ~25 nm) due to large pores of the filters. In this study, we developed new ceramic filter materials that can be mass-produced with a low-cost technique i.e. spray dried Cu_xO_y-Al_2O_3 granules for effective virus filtration. Electron micrographs of the granules demonstrated highly porous surface and sub-surface of morphology (with homogenously dispersed copper (oxide) nanoparticles) due utilization of commercial tabular alumina powder. Upon heat treatment under different atmospheres, different copper oxidation states e.g. Cu(0), Cu(I) and Cu(II) were achieved and their virus removal capacity were tested using flow experiments. In the case of CuO, there was no significant removal of viruses. MS2 was completely removed, from contaminated waterfor materials containing metallic Cu or Cu_2O. High antiviral properties of these materials were associated with positive surface charge of Cu_2O and Cu (IEP > 9.5) according to zeta potential measurements. It is suggested that electrostatic attraction of oppositely charged surfaces is thus the major contributor of high virus removal efficiency.

9:20 AM

(ICACC-S9-012-2020) Sustained release of antiviral drugs in surface functionalized organic-inorganic hybrid particles

A. Tamayo*[1]; A. Martin-Illana[2]; R. Cazorla-Luna[2]; F. Notario-Perez[2]; J. Rubio[1]; M. Veiga-Ochoa[2]

1. Institute of Ceramics and Glass, CSIC, Spain
2. Universidad Complutense de Madrid, Facultad de Farmacia, Spain

Controlled drug delivery systems are extensively studied from several decades. These systems are formed by a support material, organic, inorganic or hybrid where the drug is loaded. In this work we have explored the ability of functionalized of organic-inorganic hybrid particles with a silane coupling agent as a smart drug delivery system. We have synthesized the hybrid particles though the sol-gel method and by using a silicon alcoxyde and silicon polymer. After pyrolysis at temperatures where the polymer-to-ceramic conversion takes place, high surface area particles with a bimodal pore distribution were obtained. By changing the disposition of the silane molecule on the surface of the particles, the interaction mechanism with the antiretroviral drug molecule loaded is different as well giving as a result that the drug uptake and release do not strictly depend upon the specific surface area but on the functionalization of the surface instead. Free amino groups are required to achieve a smart pH-responsive material, a condition that is only achieved in those materials containing a silane monolayer.

9:40 AM

(ICACC-S9-013-2020) Structural and electrochemical characteristics of (oxy)cabide derived carbons obtained through wet and dry etching of polymer derived ceramics

A. Tamayo*[1]; M. A. Rodriguez[2]; A. Mazo[1]; J. Rubio[1]; F. Rubio[1]

1. Institute of Ceramics and Glass, CSIC, Spain
2. University of Extremadura, Spain

Advances in supercapacitors are delivering better-than-ever energy-storage options and, in some cases, they can compete against more-popular batteries in a range of markets. A variety of electrode and electrolyte materials are possible, being the carbon (nano) materials among the top choices as electrodes for energy storage applications owing to their versatile structures ranging from 0D to 3D and tunable surface chemistry. The addition of different elements and different carbon sources to the polymer derived ceramics allows the obtaining of several carbon structures which are developed at the different stages during the pyrolysis process. Etching away the electrochemically inactive active phases such as SiO_2 and carbides was performed in order to isolate the pure carbon phases. It was observed that the wet etching produces mesoporous materials easily accessible to the electrolyte during the electrochemical characterization whereas the dry etching lead to the obtaining of extremely high surface areas in the form of micropores. The surface characteristics as well as their textural properties have been obtained and correlated with their electrochemical performance for being used in double layer capacitors.

Modeling and Properties of Porous Ceramics

Room: Coquina Salon F
Session Chair: Thomas Graule, Empa

10:20 AM

(ICACC-S9-014-2020) Role of neck formation between particles in the thermal conductivity of green and partially sintered oxide ceramics (Invited)

D. S. Smith*[1]; J. Martinez[1]; S. Oummadi[1]; A. Alzina[1]; B. Nait-Ali[1]

1. University of Limoges, IRCER, France

A ceramic green body, formed by uniaxial pressing, is constituted of a compact powder associated with typically 40-60% pore volume fraction. The typical thermal conductivity of alumina ceramic

green bodies is in the range 0.5 to 0.8 $Wm^{-1}K^{-1}$. This low value can be explained by the pore volume fraction and predominant role of the thermal resistance of particle - particle contacts. During thermal treatment for sintering, neck formation between particles, densification and grain growth take place. This work focusses on the role of neck formation in the thermal conductivity of the green body. Our results on oxides such as alumina, tin oxide and zirconia show that increases by a factor between 2.5 and 7 can be uniquely imputed to this first stage of sintering. Information on the particle – particle contact area was deduced from BET specific surface area measurements. It was observed that the thermal conductivity of the green body evolves linearly with the contact area between particles. A simple model linking thermal conductivity to increased particle-particle contact area during the first stage of sintering was developed and tested.

10:50 AM

(ICACC-S9-015-2020) Mechanical Performance of Block Copolymer-Templated Ceramics and Nanocomposites

T. Patel*[1]; D. Street[1]; J. Bowen[1]; R. Wheeler[1]; L. Rueschhoff[1]; M. Cinibulk[1]; M. B. Dickerson[2]

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

Structural ceramics provide exceptional mechanical properties at elevated temperatures. The inherent drawback of ceramics is its brittle nature, which paves way for catastrophic failure and consequently undesired for practical applications. However, at the nanoscale, materials properties have the capability to be enhanced through careful control of structure and morphology. This talk will focus on nanocomposites based on polymer-derived ceramics and composites with a range of block copolymer-templated nanostructures. Combining these architectures with polymer or ceramic matrices increases the strength of the materials relative to their individual constituents. Ultimately, the resulting structure/processing/property relationships will provide key insight in the design and development of structural, aerospace components that may experience extreme conditions during service.

11:10 AM

(ICACC-S9-016-2020) Micromechanical modeling of ice-templated porous ceramics using smeared cracking approach

S. Sattar*[1]; S. Kravchenko[2]; O. Kravchenko[1]

1. Old Dominion University, USA
2. Purdue University, USA

Damage propagation of ice-templated porous alumina with lamellar wall is simulated by a micromechanical finite-element framework using periodic representative volume element (RVE) with the smeared cracking model. The RVE model with hexagonal lamellar structure is developed based on the microstructure of ice-templated alumina. The periodic boundary conditions are applied to the RVE to simulate the bulk behavior of the material. The smeared cracking model is used to describe the damage initiation and failure propagation in the brittle porous material. Damage initiates in the direction normal to the maximum principal tensile stress when the stress reaches the strength of bulk alumina. The softening behavior after the failure initiation was specified by fracture toughness, defined in the smeared cracking model based on the critical displacement and the maximum tensile stress of the bulk alumina. The simulation results were validated using experimental results of the compression test of the ice-templated alumina. The modeling prediction of strength and modulus for loading in 0° and 90° with respect to ice crystal growth orientation showed good correlation with compression testing of ice-templated ceramics. Therefore, the proposed micromechanical RVE analysis is effective to determine the strength and the stiffness behavior of ice-templated ceramic and can be used for homogenization procedure.

11:30 AM

(ICACC-S9-017-2020) 3-D Visualization of Unfired (Green) Al_2O_3 Dry-Pressed Bodies

I. P. Maher[1]; R. A. Haber*[2]

1. Coorstek, USA
2. Rutgers University, Materials Science and Engineering, USA

Microstructural variability in ceramic green bodies are difficult to account for. In this study, alumina was the system analyzed and was processed by spray-drying alumina slurries with two varying binders; an acrylic emulsion binder and a polyvinyl alcohol (PVA) binder. The plasticity differences between the generated spray-dried granules was evaluated on a compaction scale to determine the effects of the varying binders. The density and microstructural characteristics of the formed alumina compacts were evaluated. This work developed methods to visualize the particle and pore arrangements in dry-pressed alumina green compacts in both two and three dimensions. Image analysis was conducted by use of a field emission scanning electron microscope and micro X-ray computed tomography to visualize the internal structure of the alumina granules and the formed alumina compacts. Visualizing the internal structure of the green compacts in both two and three dimensions was needed to further improve the microstructural capabilities of green ceramic materials. The developed visualization methods correlated a relationship of what processing parameters produce fewer green bulk microstructural variations. The improved green microstructural characterization techniques accomplished in this work will hopefully improve outcomes and reliability during ceramic processing.

Engineering Applications of Porous Ceramics III

Room: Coquina Salon F

Session Chair: Manabu Fukushima, National Institute of Advanced Industrial Science and Technology (AIST)

1:30 PM

(ICACC-S9-018-2020) Recent Advances in Development of Glass Foams (Invited)

Y. Yue*[1]; J. König[2]; R. R. Petersen[3]; M. B. Ostergaard[4]

1. Aalborg University, Denmark
2. Jozef Stefan Institute, Advanced Materials, Slovenia
3. Skamol, Department of R&D, Denmark
4. Aalborg University, Chemistry and Bioscience, Denmark

Glass foams are lightweight cellular glasses with significantly lower thermal conductivity compared to bulk glasses, and hence, a good thermal insulator for energy saving. They are superior over other insulation materials such as organic foams and mineral wool, since they possess water and steam penetration resistance (due to their closed porosity), freeze-thaw cycle tolerance, excellent chemical and thermal stability, and load-bearing ability. Another advantage of glass foams is that they are usually made from recycled glasses, and this benefits our environment. However, it is challenging to achieve the glass foams with the thermal conductivity comparable to that of mineral wool. The thermal conductivity is a key parameter for glass foam performances since energy saving is increasingly important for our environment and climate. To overcome this challenge, over the past seven years we have made considerable effort in optimizing the foaming agent chemistry and foaming conditions to get superior glass foams. To do so, we also carried out systematic studies to reveal the foaming mechanisms of waste glasses. Finally, we succeeded in achieving the thermal conductivity lower than the reported values. These advances in our glass foam research will be presented in my talk. We will also describe some perspectives of the future development and applications of glass foams.

2:00 PM

(ICACC-S9-019-2020) Gas Flow Method for Determining Wall Permeability of Particuluate Filters for Pressure Drop Model Development

M. L. Anderson*[1]; R. Stafford[1]

1. Cummins Inc., USA

Diesel particulate filters (DPFs) are critical components in the exhaust aftertreatment of diesel engine systems to meet regulatory requirements across the globe for particulate matter. The addition of particulate filters increases the backpressure on the engine, so a key design parameter for a DPF is the pressure drop across the component and one of the key factors in controlling the pressure drop is the permeability of the porous wall. If this value is known, accurate pressure drop models can be developed and assist in the design and selection of the optimal DPF for an application. Traditionally, this value is estimated based on flow testing behind an engine, which induces numerous noise factors. A laboratory method was developed to directly measure the permeability based on Darcy's Law using a small sample of material from a DPF. The use of this method has enabled the evaluation of different particulate filter materials and impact catalytic coatings have on the permeability. Future study will investigate the impact soot and ash have on the wall permeability.

2:20 PM

(ICACC-S9-020-2020) Controlled Permeability and Strength of Red Firing Clay Ceramic Filter Tempered with Iron Rich Oxide Material and Treated Mine Waste

L. I. Cabalo*[1]; E. Ibarra[1]

1. Mindanao State University-Iligan Institute of Technology, Department of Materials Resource and Technology, Philippines

Controlled porous material have been fabricated, composed of sintered red clay and non-plastic components tempered with iron rich oxide materials through slip casting technique. The iron rich oxide generally controls the pore size of the red clay based ceramics for various applications such as fluid filtration with controlled distribution. Three groups of different compositions were formulated in order to compare the permeability of the red firing clay based ceramics. The first formulations were made from red clay, silica, and iron rich oxide only, second and third compositions utilized the use of treated mining waste from CARAGA region to further enhance the strength of the fired ceramics. Rheological characterization of red clay ceramic slurries showed shear thinning behaviour. Samples were formed using plaster mould and fired at 1100 °C. Physical properties of formed ceramic filter shows increasing apparent porosity as the amount of loaded iron rich oxide increases. Increasing apparent porosity decreases the vacuum pressure and increases the fluid flow rate of about 2.86 L/h, which demonstrates a good red firing clay based ceramics for fluid filter applications.

Innovations in Processing Methods and Synthesis of Porous Ceramics II

Room: Coquina Salon F
Session Chair: Yuanzheng Yue, Aalborg University

3:20 PM

(ICACC-S9-021-2020) Freeze-casting of porous polysilazane-derived ceramics (Invited)

T. Konegger*[2]; R. Obmann[2]; G. Mikl[2]; S. Schörpf[1]; R. Liska[1]

1. TU Wien, Institute of Applied Synthetic Chemistry, Austria
2. TU Wien, Institute of Chemical Technologies and Analytics, Austria

Freeze-casting of preceramic polymers poses a versatile method to create materials exhibiting highly directional pore channels in combination with excellent thermal and chemical properties, thus being of particular interest for prospective applications in energy- and environment-related fields; however, intricate cross-linking pretreatments are often required in order to allow for the processing of liquid precursor systems. In this presentation, a new approach towards freeze-casting of polymer-derived ceramics called photo-polymerization-assisted solidification templating (PASST) is presented, employing a photo-induced thiol-ene click reaction to achieve low-temperature-cross-linking of a polysilazane precursor compound after directional solidification using a structure-directing solvent. After removal of the solvent and pyrolytic conversion of the preceramic polymer, ceramic materials with a wide variety of pore structures can be obtained. The effect of various solvents and nucleating agents on the resulting pore morphology as well as the correlation to permeability and strength characteristics are highlighted. The choice of the structure-directing solvent affects the resulting pore structure to a high extent, leading to the evolution of dendritic, lamellar, or columnar pore structures, and, as a consequence, highly diverse strength and permeability characteristics of the resulting materials.

3:50 PM

(ICACC-S9-022-2020) Microstructure Evolution, Structural Stability, and Compressive Mechanical Properties in Ice-templated Sintered Ceramics with Directional Porosity

D. Ghosh*[1]; M. Banda[1]

1. Old Dominion University, Mechanical and Aerospace Engineering, USA

In ice-templating technique, phase separation of solvent crystals and ceramic particles followed by freeze drying and sintering result in ceramic foams with directional porosity. Ice-templated ceramics exhibit macroscopic structural anisotropy, whereas contain equiaxed grains within the lamella walls and lamellar bridges. Variations of particle size and particle morphology along with the unidirectional solidification kinetics can result in the unprecedented modifications of the length-scale features in ice-templated ceramics and marked changes in compressive mechanical properties. However, there is a need to thoroughly understand the interactions of fine-grained ceramic matrix and anisotropic particles and microstructure evolution and structural stability during high-temperature sintering. In this work, we aim to understand the progressive interactions of fine-grained alumina ceramic matrix and alumina platelets and microstructural modifications within the lamella walls of ice-templated porous ceramics during sintering. Moreover, our aim is to connect the local structural modifications to the macroscopic mechanical response and identify the parameters that are critical to tailor the macroscopic properties. The results will be significant to further advance the structure-property relationships in novel ice-templated ceramics.

4:10 PM

(ICACC-S9-023-2020) Biomimetic freeze-cast ceramics and their property -porosity relations

K. Klang*[1]; K. G. Nickel[2]; T. Fey[1]

1. Friedrich-Alexander University Erlangen-Nürnberg, Department Material Science and Engineering, Germany
2. University Tuebingen, Applied Mineralogy, Germany

The cellular highly porous lightweight construction (Φ of 0.6 to 0.9) from the spines of the lance sea urchin are characterized by a high energy dissipating capacity at high strain rates, which is accompanied with quasi-plastic deformation based on multiple fracturing, even though the constituting material is basically brittle magnesium calcite. The correlation between structural and mechanical characteristics of the sea urchin spine is analyzed in depth to reveal their underlying operating principles in order to transfer them into ceramic-based materials. The mechanical behavior of the natural materials is compared to synthetic bioinspired materials of comparable porosity made from alumina manufactured via freeze casting. Both biogenic and bioinspired materials are open-cell networks with channel-like porosities and hence highly anisotropic in terms of pore shape. We demonstrate the systematics of the relation between

the compressive strength and e-modulus, the e-modulus and the porosity as well as the compressive strength and porosity in the light of pore shape and size for both biogenic and freeze-cast materials. We also discuss the applicability of the mechanical prediction of the Gibson and Ashby models and of the honeycomb model provided by Zhang and Ashby in a broader view for freeze-cast alumina ceramics.

4:30 PM

(ICACC-S9-024-2020) Effect of platelet raw particles on strength and thermal conductivity of gelation freezing derived thermal insulators

M. Fukushima*[1]; Y. Yoshizawa[1]

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

We will report strength and thermal conductivities of cellular ceramics through gelation and freezing route, investigating the effect of addition of coarse platelet raw particles on various properties. The addition of platelet particles could enhance the compressive strength, as those were unidirectionally oriented during freezing, while the shrinkage during sintering could be reduced by the addition. Thus, the thermal conductivities of final products can be lowered. In addition, we conducted the image based modelling techniques for mechanical properties and thermal conductivities of the insulators, by using three dimensional microstructures measured by X-ray computed tomography.

4:50 PM

(ICACC-S9-025-2020) Freeze casting of feather–light, cellulose–nanofiber–reinforced γ–Al_2O_3 foams

H. Hudelja*[1]; A. Kocjan[2]

1. Jozef Stefan Institute, Nanostructured materials, Slovenia
2. Jozef Stefan Institute, Slovenia

In the present work freeze casting was used for preparation of highly porous γ–Al_2O_3 ceramic foams with high specific surface area, requiring no sintering steps. For this purpose, γ–Al_2O_3 2D nanosheet-like particles, hierarchically assembled into micron-sized entities, with retained intrinsic "nano-scale" features, were synthesized by exploiting AlN powder hydrolysis as a straightforward and pure method for preparation of abundant amounts of γ–Al_2O_3 powder. As-prepared powder was dispersed in water and the effects of dispersant, divalent cations and/or cellulose nanofibers addition on the suspension stabilization, rheological properties and freeze casting behavior were studied. The foams were analysed for pore size distribution, specific surface area, thermal conductivity and compressive strength. As-formed foams showed low relative densities (2.3–8.9 %) with hierarchically distributed porosity, i.e., macropores (1–100 μm) and mesopores (4–7 nm), high surface areas (~160 m^2/g) and lowered thermal conductivities (0.08–0.18 W/mK). Addition of cellulose nanofibres turned out to be beneficial not only in terms of improved porosities, but also gave rise to remarkable strengths and rigidness for such light and porous green bodies.

5:10 PM

(ICACC-S9-026-2020) Porous Geopolymers Heterogeneous Catalysts for Biodiesel Production

P. Colombo*[1]; R. Botti[1]; M. Iinnocentini[2]; C. Paschoalato[2]; D. Flumignan[3]

1. University of Padova, Industrial engineering, Italy
2. University of Ribeirão Preto, Course of Chemical Engineering, Brazil
3. São Paulo Federal Institute of Education, Science and Technology, Brazil

This work investigates porous geopolymers (GP) acting as heterogeneous catalysts to produce biodiesel by transesterification reaction of soybean oil with methanol. Three types of GP systems were produced mixing metakaolin with an activating alkaline solution: Na-based, K-based GP and a mixture between them; they were treated at 110, 300, 500 and 700°C. Both powders and 3D printed lattices were designed and produced by DIW, adding PEG and a filler in the formulations and drying at 110°C. Porous structures with Ø ~24 mm x 9,6 mm height and unsupported struts were produced. The transesterification reaction was carried out using all the samples as a heterogeneous catalyst to evaluate the yield of biodiesel. According to the results obtained, it was verified that using GP both in powder and structure as catalyst, it was possible to obtain biodiesel from the transesterification of soybean oil. Comparing the materials with the same molar ratios, Na.K_GP treated at 500°C (powder) achieved the highest conversion (~98%). For the 3D structure tested in the reaction (3D_Na_GP1, 110 °C) a conversion was observed, but lower (~41%) compared to Na.K_GP, even in its powdered version (~53%), probably due to the presence of residual PEG.

S10: Modeling, Genome, Informatics, and Machine Learning

Multi-scale Modeling of Processing and Performances II

Room: Coquina Salon G
Session Chair: Gerard Vignoles, University Bordeaux

8:30 AM

(ICACC-S10-010-2020) Effect of irregular pores and inclusions on elastic properties of composites (Invited)

R. Piat*[1]; P. A. Happ[2]; I. Tsukrov[3]; B. Drach[4]

1. Darmstadt University of Applied Science, Germany
2. University of Applied Sciences Darmstadt, Mathematics and Natural Sciences, Germany
3. University of New Hampshire, USA
4. University of New Mexico, USA

In present paper the effect of pores and particles with irregular shapes on the elastic material properties of two-phase composites is studied. The irregular shapes of the real inclusions were approximated using smooth three-dimensional structures. For this needs the images of the microscopic particles were numerically approximated through smooth structures using methods of the computer algebra and were used for the following FE studies. The reference elements with typical inclusions with irregular shapes were determined and used for calculation of the effective material properties.

8:55 AM

(ICACC-S10-011-2020) Quest for clean perovskite-based materials for optoelectronic applications: Insights from first-principles (Invited)

G. Giorgi*[1]; M. Palummo[2]

1. The University of Perugia, Department of Civil & Environmental Engineering, Italy
2. University of Rome "Tor Vergata", Department of Physics, Italy

The recent impulse obtained by 3D hybrid organic-inorganic halide perovskites (OIHPs of stoichiometry ABX_3, A=organic cation; $B=Pb^{2+}$; X=halide) in solar devices due to unprecedented efficiencies (PCE>23%) still conflicts with the drawbacks that prevent their usage in mass production. The most detrimental ones are the instability towards moisture/heat due to the presence of the organic cation (mainly methylammonium, $MA=CH_3NH_3^+$, highly hydrophilic) and the environmental one due to the presence of Pb in the B-site of 3D OIHPs. Accordingly, two different routes are suggested to fix them. For the latter, replacing Pb with ecofriendly metallic cations (along with MA replacement with Cs in the A-site) seems to be effective, giving rise to ternary/quaternary all-inorganic perovskites (IPs) with optoelectronic features comparable to pristine 3D OIHPs. The stability issue may be solved reducing the dimensionality of OIHPs. In 2D OIHPs layers of PbI_6 octahedra are embedded between long chain hydrophobic (stabilizing) organic cation layers. In my talk I show the optical features, calculated including excitonic and local-field effects (GW+Bethe Salpeter Equation), of both 2D

OIHPs -relating the thickness of the inorganic part with the applicability in optoelectronics (LED vs solar)- and of recently synthetized Pb-free IPs, revealing the impact of such classes in optoelectronics.

9:20 AM

(ICACC-S10-012-2020) Mesoscale Modeling of the Formation of Line Compounds in Reaction-Bonded Materials such as SiC

P. Goins*[1]; S. P. Coleman[1]; M. C. Guziewski[1]

1. US Army Research Laboratory, USA

Historically, a number of numerical techniques have been developed for studying microstructure evolution in non-conserved fields or conserved fields with smooth and continuous free energy functions. However, to date, there is great difficulty in modeling line compounds such as those found in ceramics, especially in a computationally tractable manner. Here, we present a Monte-Carlo based approach to study phase evolution in diffusion-limited reaction-bonded line compound materials, using as an example the reaction between carbon and silicon sources to form SiC. This approach allows for large-scale 3-dimensional volumes to be considered with simultaneous diffusion-limited phase and grain evolution, which can be used to optimize the processing conditions and precursor particle size and shape distribution to control final SiC structure.

9:40 AM

(ICACC-S10-013-2020) Evaluating effects of irregular inclusions and interactions on elastic properties of composites

P. A. Happ*[1]; R. Piat[1]

1. Darmstadt University of Applied Science, Germany

In this study a variety of different particle shapes are created applying analytical functions to approximate an irregular topology. The upcoming process evaluates numerically the impact of the different surfaces on the elastic properties. In this process the finite element method is being used for calculating the contribution to the composite reinforcement in relation to the surface structure firstly in a single inclusion setup, secondly in a configuration with multiple inclusions. The latter configuration enabling to take a deeper on interaction behavior between inclusions. During the multiple inclusion configuration, the particle volume fraction is being varied to find a correlation between pack density and interaction behavior.

Multi-scale Modeling of Processing and Performances III

Room: Coquina Salon G
Session Chairs: Romana Piat, Darmstadt University of Applied Science; Giacomo Giorgi, The University of Perugia

10:20 AM

(ICACC-S10-014-2020) Multi-Scale Modeling of Hierarchical Microstructures in Diamond-SiC

S. P. Coleman*[1]

1. US Army Research Laboratory, USA

Microstructural features across nano- to meso-scale are known to influence the quasi-static properties and dynamic response of multi-phase ceramic composites, such as diamond-SiC. To elucidate the individual and correlated contributions of various microstructures features; the CCDC Army Research Laboratory recently established a multi-scale modeling effort spanning first principles and classical atomistics to mesoscale Potts Monte Carlo and phase field simulations. At the lowest length scales, high-throughput atomistic models investigate the influence of defects and interfaces on local physical properties. These models capture the variance of properties, which are used to parameterize mesoscale models looking at the influence of granular distributions on the macroscale response. Multi-scale modeling efforts in this work are integrated with experiments for validation with ultimate goal for materials design.

10:40 AM

(ICACC-S10-015-2020) Understanding the Energetics and Structure of Interfaces in Silicon Carbide-Diamond Composites using High-Throughput Atomistic Simulations and Machine Learning

M. C. Guziewski*[1]; D. Trujillo[2]; S. P. Coleman[1]; P. Alpay[2]

1. US Army Research Laboratory, USA
2. University of Connecticut, Materials Science and Engineering, USA

High throughput atomistic models, in conjunction with machine learning, are used to probe the uncertainty in the energetics and structure within interfaces of the silicon-silicon carbide-diamond system. In this work, a Metropolis style Monte Carlo algorithm is applied to a variety of tilt, twist and mixed character grain boundaries within and between the various phases of the Si-C system under consideration. This approach seeks to capture the variance in structure and energy beyond simply the five macroscopic degrees of freedom that describe the boundary geometry, though the consideration of factors such as metastable states, non-stoichiometric interfaces, and chemical environment near the interface. The produced data is used to train machine learning models to both further optimize the high-throughput technique and aid in the development of constitutive relations to connect energetics to macroscopic variables. The developed approach allows for the analysis of not just pristine interfaces, but interfaces that more closely resemble those which are seen experimentally. Additionally, the machine learned relation can be used to atomistically inform higher length scale models, allowing for higher fidelity mesoscale models.

11:00 AM

(ICACC-S10-016-2020) Modeling Polymeric Binder Removal in Porous Green Bodies

E. G. McAleer*[1]; E. K. Akdoğan[1]; J. Matthewson[1]; R. A. Haber[1]

1. Rutgers University, Material Science and Engineering, USA

Polymeric binders are added to ceramic powders during processing to provide temporary green strength to the ceramic part and are used extensively in slurry based ceramic processing such as tape casting and additive manufacturing. These binders become impurities while sintering, therefore the part must be heated to elevated temperatures high enough to degrade the polymers but lower than the temperature at which sintering begins to occur. This process is often conducted slowly, and heating cycles are generally found using trial and error. Having a model that accurately accounts for the kinetics, thermodynamics, and transport of the degraded polymer would be desirable in order to optimize the parameters of the binder removal process. In this work thermogravimetric analysis is used to obtain activation energy, reaction order, and pre exponential factors which can be used as inputs in the model. Current work focuses on creating well characterized green bodies with known model parameters in order to validate model outputs and understand further how green body parameters such as porosity and tortuosity affect the removal process.

11:20 AM

(ICACC-S10-017-2020) Simulation and Permeability and tortuosity of ceramics foams based on µCT

T. Fey*[1]

1. Friedrich-Alexander University Erlangen-Nürnberg, Department Material Science and Engineering, Germany

Cellular materials offer a wide spectrum of applications such as catalyst support structures, lightweight materials, energy adsorption, energy storage materials and filters. For filtration purpose an open-cell structure is needed. The cell window size is of particular

interest for permeability of liquids and gases. Also cell interconnectivity and resulting pore percolation network is strongly affected by the cell window size. Due to the fact that experimental permeability measurements are limited in pressure and temperature range as well as type of gas or liquid (e.g. molten metal), simulation of permeability is in particular focus of interest. Based on micro computertomography (μCT) data the pore network is extracted for permeability simulations and tortuosity calculations. Darcian (linear) and Forchheimer (non-linear) permeability can be calculated from the simulated pressure drop in a defined volume of a cellular ceramic foam. A certain pore path within this volume for e.g. minimum pressure drop can be expressed by calculation of tortuosity.

Multi-scale Modeling of Processing and Performances IV

Room: Coquina Salon G
Session Chairs: Peter Kroll, University of Texas, Arlington; Notker Roesch, TU Munich

1:30 PM

(ICACC-S10-018-2020) 2D microstructural modeling of transverse cracking during pyrolysis process of carbon fiber reinforced plastics (Invited)

Y. Shi*[1]; J. Neraj[1]

1. DLR - German Aerospace Center, Institute of Structures and Design, Ceramic Composites and Structures, Germany

During the manufacturing process of carbon fiber based C/C-SiC composites, the phenolic resin of prepreg is converted to carbon through high temperature pyrolysis treatment. This step induces a high amount of porosity within the microstructure. The pore morphology varies from fine pores throughout the composite up to large crack channels with block-like, pretty dense C/C fiber matrix areas. The different crack formations during pyrolysis strongly depend on the fiber/matrix interface strength and temperature. In the present study, the formation of crack pattern is simulated in a FE-model with consideration of effects of fiber/matrix interface strength, fiber volume fraction, temperature change and other essential microstructural properties. The presented models are successfully able to simulate the material behavior and the consequent generation of cracks during pyrolysis process. Microstructure of the material has been then analyzed and compared with simulation results with the help of image segmentation techniques using Python. Based on the crack area distribution obtained from the SEM-analysis, microstructures can now be compared qualitatively as well as quantitatively.

1:55 PM

(ICACC-S10-019-2020) A mesoscopic model with non-linear elasticity and phase transformation framework for the twinning-buckling behavior of TATB under dynamic loading: A Molecular Dynamics inferred constitutive law (Invited)

P. Lafourcade*[1]

1. CEA, France

A mesoscopic modeling of TATB is proposed and validated on MD simulations. The two dominant deformation modes observed at nanometric scale and limited stress are a buckling instability and a non-symmorphic twinning transformation. A thermodynamically consistent continuum model is detailed, with a non-linear elasticity in pressure constructed to reproduce a cold equation of state. The twinning-buckling observed in MD is modeled by using Phase-Field by Reaction-Pathway (PFRP). To validate the present constitutive law, we first study the response of the single crystal under uniaxial compressions for various directions in the basal plane and present one to one comparisons between both techniques. These comparisons are made possible thanks to continuum mechanistic tools that have been implemented in a MD code: a formalism that allows for imposing any deformation is used to activate new deformation mechanisms. Additionally, these mechanisms are detected and finely analyzed thanks to a local measurement of the Green-Lagrange strain tensor. Finally, an upscale is performed and simulations using the constitutive law are presented for time and space scales non reachable in MD. A large TATB polycrystal is generated and simulations of both hydrostatic and shock compressions are presented.

2:20 PM

(ICACC-S10-020-2020) Development of a Finite Element Model for Quasi-statically indented Oxide/Oxide Ceramic Matrix Composite in Ambient Environement

A. Nasirmanesh*[1]; A. K. Singh[1]

1. Baylor University, Mechanical Engineering, USA

Foreign object damage is a reality in ceramic matrix composites especially as their use are also sought in military vehicle and armor applications. In this research a micromechanics based finite element model was developed for quasi-statically indented oxide/oxide ceramic matrix composite in ambient environment. The model incorporated damage events of fiber breakage and matrix cracking and the indentation loading and unloading profiles were computationally created resulting in an approximate prediction for the residual dent depth. The model was valid for shallow dent depths of ceramic matrix composites with no back surface damage. Modeling results were compared to published experimental data and the results were found to be in good agreement.

2:40 PM

(ICACC-S10-021-2020) Numerical Simulation of Densification in Porous Preforms by Chemical Vapor Infiltration

V. Ramanuj*[1]; R. Sankaran[1]; B. Jolly[1]; R. Lowden[1]

1. Oak Ridge National Laboratory, USA

Chemical vapor infiltration (CVI) is a technology used for manufacturing complex 3D ceramic matrix composites for high temperature applications. Gaseous precursors are made to ingress a porous preform and deposit the desired solid phase through specific chemical mechanisms. One of the critical challenges encountered in the technique is to obtain uniform densification within the processed part. Quality of densification typically depends on the relative time scales of transport and chemical phenomenon. Experimental examination of the process becomes challenging due to extremely long manufacturing times and high material costs. We present a numerical model for simulating surface growth of a porous preform and analyze the densification quantitatively. Direct numerical simulations are performed to resolve the length scale of solid particles in the preform and capture growth and topological changes of the surface leading to formation of inaccessible voids. For versatility of applications and complex initial shapes, an additively manufactured 3D printed preform with large initial porosity is chosen. The simulation results are compared with experiments under conditions ranging from transport controlled to kinetic controlled regimes. The physical model and numerical implementation along with the effects of processing conditions on densification will be presented.

Modeling of Surfaces, Interfaces, and Grain Boundaries

Room: Coquina Salon G
Session Chairs: Yuan Shi, DLR - German Aerospace Center, Institute of Structures and Design; Paul Lafourcade, CEA

3:20 PM

(ICACC-S10-022-2020) Reduced Centers of the MoV Oxide Catalysts for Selective Hydrocarbon Oxidation (Invited)

A. Genest[2]; W. Li[2]; T. Fjermestad[2]; J. Arce Ramos[2]; G. P. Rugg[2]; N. Roesch*[1]

1. TU Munich, Germany
2. Institute of High Performance Computing, Singapore

The oxide materials MoV and MoVTeNb in their M1 phase are promising catalysts for selective oxidation reactions, e.g., the direct conversion of propane to acrylic acid or the dehydrogenation of ethane and propane to the corresponding olefins. Reactions over these catalysts enable an attractive shift in feedstock from oil to natural gas and are energy efficient. Yet, our understanding of these processes is rather limited due to the complex nature of the materials. Also, the work horse of computational catalysis, GGA-DFT, fails to provide a reliable description of these types of materials. We present efforts toward adequate models for the electronic structure of the materials, focusing on the distribution of polarons at V centers. In its ideal occupancy, the $MoVO_x$ catalyst material shows 60 ways for distributing 6 polarons on V centers. Many of them are energetically accessible, spanning a range of ~140 kJ/mol, thus crucially influencing the surface reactivity. Reduced centers nearby affect the oxidative dehydrogenation of hydrocarbons by up to 70 kJ/mol. In contrast, the hydrolysis of the catalyst seems to vary notably less with the distribution of reduced centers. Using a set of indicators, we predict the distribution of polarons within a margin of 5 kJ/mol. Model calculations also indicate the interplay between TeO moieties in channels of the material and the reducibility of various metal centers.

3:50 PM

(ICACC-S10-023-2020) Genesis of "Free" Carbon in Silicon Oxycarbide Ceramics (Invited)

P. Kroll*[1]

1. University of Texas, Arlington, USA

Modeling the thermal processing of polysiloxanes by various computational methods at different length scales we explore the structure formation of carbon within polymer-derived SiCO ceramics. Excess carbon develops through distinct stages. Initially, isolated carbon units of a molecular precursor are well dispersed throughout the polymer precursor. Upon annealing those units combine and form a "free" carbon phase embedded in and surrounded by the polymer and glass network. The "free" carbon forms distinct segregations of single-layered carbon flakes. These flakes percolate through the structure and form extended, graphene-like sheets. At this stage, the carbon segregations divide the surrounding matrix, essentially confining it in domains. Continuity and sizes of these domains are related to the amount of free carbon and to the composition of the material. At higher temperatures, the carbon sheets curl up to form tubular structures. Ultimately, tubular structures convert into large graphitic segregations. Presence of multi-walled carbon nanotubes in some polymer-derived SiCO caramics has recently been established. We will show that mechanical properties of SiCO ceramics with identical composition depend on the carbon morphology present in those ceramics.

4:20 PM

(ICACC-S10-024-2020) Role of organic cation-centered states in formamidinium lead iodide: Bulk vs nanoparticle models (Invited)

S. Manzhos*[1]; G. Giorgi[2]

1. INRS, EMT, Canada
2. The University of Perugia, Department of Civil & Environmental Engineering, Italy

Organic-inorganic halide perovskitess (OIHP) other than the classic methylammonium lead iodide (MAPI) are actively explored for use in perovskite solar cells (PSC) and perovskite light emitting diodes (PLED). The use of organic cations such for formamidimium (FA) has been reported to be advantageous, in particular in PLED. While the minimum of the conduction band (CBM) and the maximum of the valence band (VBM) of OIHPs are typically dominated by states due to the inorganic sublattice (such as PbI), it was suggested (PCCP 2019, 21, 8161) based on cluster calculatons that FA-centered states may be close to the CBM. We compute using density functional theory and compare the properties of bulk and small nanoparticles of FAPI and compare them to those of FA-doped MAPI. We consider the effects of van der Waals interactions and of the choice of the functional (GGA or hybrid) on the bandstructure as well as spin-orbit coupling effects. We observe FA-centered states in relative proximity to the CBM and a strong nanosizing effect whereby FA-centered states may fall through the CBM of the PbI sub-lattice in small nanoparticles. The results suggest that FA-centered states could have significant effect on electronic properties, as such states could be occupied (forming neutral FA species) by photoexcitation (in PSC) or electron injection (in PLED).

4:40 PM

(ICACC-S10-025-2020) First-Principles Study of Enhanced Tolerance to Impurity on Metal-Oxide/Pt in Polymer Electrolyte Fuel Cell Anode

N. Ozawa*[1]; M. Kubo[1]

1. Tohoku University, Institute for Materials Research, Japan

Polymer electrolyte fuel cell (PEFC) needs anode materials with high tolerance to poisoning by impurities such as CO, NH_3, H_2S in the fuel, which degrades performance of the PEFC. Olu et al. revealed that combination with metal-oxide such as RuO_2 improves CO tolerance of a polycrystalline Pt catalyst. For theoretical design of anode materials with high tolerance to impurity poisoning, it is necessary to understand the reason why the metal-oxide improves the CO tolerance of the Pt catalyst. Then, to reveal this mechanism by RuO_2 combination, we investigated CO adsorption on RuO_2/Pt(111) and Pt(111) by first-principles calculation. The adsorption energies of CO on RuO_2/Pt(111) and Pt(111) are 30.41 and 38.05 kcal/mol, respectively, indicating that the combination with RuO_2 decreases the adsorption energy of CO on Pt(111). Here, d-orbitals of Pt contribute to the chemical bond between Pt and CO. When the energy level of the d-orbital decreases, the adsorption energy of CO also decreases. The calculated d-band center on RuO_2/Pt(111) is –8.11 eV and lower than that on Pt(111) of –7.87 eV. Experimentally, the combination of RuO_2 suppress CO adsorption on Pt catalyst, which is in good agreement with our calculation. Therefore, we suggest the role of the combination of RuO_2, which changes the electronic states of the Pt atom and suppresses the CO adsorption on Pt(111).

5:00 PM

(ICACC-S10-026-2020) Parallelization of multi-step catalytic reactions: DFT-thermodynamics and Experiments

H. Choi*[1]

1. University of Cologne, Germany

In general, 'Task Parallelization' is the simplest and ultimate solution for bottle-neck problems of serial multi-step tasks. Catalytic reactions for energy conversion and storage are in many cases, serial

multi-step reactions having bottle-neck steps. In this work, we first suggest the concept of 'Task Parallelization' in multi-step catalytic reactions. As the prototype study, we applied this concept to oxygen evolution reaction (OER) on electrocatalytic materials. Since the catalytic activity of a material is the result of moderate adsorption energies of reactants and intermediates, mixture of multiple phases having different bottle-neck reaction steps can lead to excellent catalytic material composite, even though each phase is not much good. In this study, we used first-principles calculations and thermodynamic modeling to predict reaction pathways and facile synthesis of multiphase nanostructure. Our well-designed experiments prove this concept very well for OER electrocatalysis.

S16: Geopolymers, Inorganic Polymers and Sustainable Materials

Conversion to Ceramics; Novel Applications; Phosphates

Room: Tomoka C
Session Chair: Dong-Kyun Seo, Arizona State University

8:30 AM

(ICACC-S16-009-2020) Tailorable thermal expansion in ceramics synthesized by geopolymer crystallization (Invited)

A. J. Steveson*[1]; W. M. Kriven[1]

1. University of Illinois at Urbana-Champaign, USA

The use of geopolymers as high-performance thermal barrier coatings bypasses several of the technical challenges with implementing geopolymer technology in high-temperature environments and is therefore an ideal starting point for commercialization. In this work, we discussed how geopolymers are excellent candidates for thermal barrier coatings and showed how the dominant mechanisms of the temperature-driven structural evolution of samples belonging to the $K[AlSi_2O_6]$-$Cs[AlSi_2O_6]$ pseudo-binary synthesized by heat treatment of pure geopolymer allow for tailored phase transition domains and thermal expansion behavior. X-ray diffraction data was analyzed by Rietveld refinement and a second order polynomial equation was derived to describe the thermal expansion in three dimensions. The principal strain axes were examined in 3D using the Coefficient of Thermal Expansion Analysis Suite (CTEAS) software developed at UIUC. A wide range of tailorable thermal expansion was demonstrated to ensure a wide possibility for applications.

9:00 AM

(ICACC-S16-010-2020) Crystallographic studies of the leucite-pollucite system synthesized by geopolymer crystallization

A. J. Steveson*[1]; W. M. Kriven[1]

1. University of Illinois at Urbana-Champaign, USA

While geopolymers are well established for low temperature applications, their ceramic analogues have untapped potential at high temperatures. Because these materials can take advantage of the relative ease of geopolymer processing compared to traditional ceramic processing, they could foster a new era of ceramic technologies. Applications of geopolymers in high temperature environments require characterization of the crystalline phases that form. In this work, we examined the temperature-driven structural evolution of samples belonging to the $K[AlSi_2O_6]$-$Cs[AlSi_2O_6]$ pseudo-binary synthesized by heat treatment of pure geopolymer using in situ high-temperature synchrotron X-ray powder diffraction. The data was analyzed by Rietveld refinement and the mechanisms underlying the thermal expansion and phase transitions were elucidated by statistical methods. We demonstrated that high purity, single-phase ceramics could be synthesized with low-waste and low-energy requirements and show how crystalline ceramics synthesized by the geopolymer crystallization method can be expected to differ from other processing routes.

9:20 AM

(ICACC-S16-011-2020) Geopolymer materials for high-frequency antenna applications

I. N. Vlasceanu*[1]; A. Gharzouni[1]; O. Tantot[2]; E. Martinod[2]; V. Bertrand[2]; N. Feix[2]; M. Lalande[2]; C. Elissalde[3]; S. Rossignol[1]

1. IRCER, France
2. Xlim, France
3. ICMCB-CNRS, France

Nowadays, it is important to reduce the size of the connected devices used in communication and electronic for applications as detection and localization. The main request for these devices is to decrease the dimension, to avoid volatile organic compounds and to save energy to prevent the global warming. Among the opportunities to avoid volatile organic compounds a new eco-friendly material with dielectric properties appear as candidates. The geopolymer materials increasing interest over the last decades because of their high working performance, wide range and low environmental impact. Thus, this study aims is to develop a new dielectric material based on geopolymers for high-frequency antennas. To achieve the objective, different types of metakaolin were mixed with alkaline solutions and additives. Characterizations performed are thermogravimetric analysis, surface tension and dielectric measurements. For dielectric properties, it is important to control the water content of the samples. Thermogravimetric analysis showed that water content of the sample is affected by the type of metakaolin used and the quantity of Fe_3O_4 added (lower than 10%wt). Moreover, it was proved that addition of Fe_3O_4 causes a decrease of the surface tension. Dielectric properties of geopolymers were evaluated for frequencies between 2-3.3GHz. Consequently, geopolymers with Fe_3O_4 addition could be a promising candidate for antenna applications

9:40 AM

(ICACC-S16-012-2020) Calcium phosphate cement with car tire waste

C. F. Revelo[1]; H. Colorado*[1]

1. Universidad de Antioquia, Colombia

There is an increasing problem worldwide with the amount of car tires disposed improperly in landfills or other areas, mostly because its recycling requires more applications when the generation rate of this waste is considered. Therefore, a new solution of composites materials from a calcium phosphate cement matrix with waste rubber powders obtained from the car tire recycling were investigated in this research. Six formulations were fabricated and diverse characterization were conducted over the samples, which includes scanning electron microscopy, x-ray diffraction, adhesion tests matrix to rubber, and environmental tests. Results show the developed material can be an alternative solution for the use of tires after use.

Waste Materials

Room: Tomoka C
Session Chair: Nishant Garg, University of Illinois Urbana-Champaign

10:20 AM

(ICACC-S16-013-2020) Production of Porous Geopolymer Brick from Philippine Gold Mine Tailings

L. de Leon[1]; P. Labastida[1]; E. Labastida[1]; E. d. Magdaluyo*[1]

1. University of the Philippines, Philippines

Geopolymers are sought out substitutes for the usual cement-based bricks, as they are proven to produce less carbon footprint. This study aims to utilize gold mine tailings (GMT) from Aroroy, Masbate, Philippines as the main source of aluminosilicate minerals for the production of porous geopolymer bricks. The experiment used sodium hydroxide (NaOH) solution as the alkali-activator

for all the mixtures and the amount of sodium silicate (Na_2SiO_3), rice husk ash (RHA) and copper powder was incorporated as test variables, following the taguchi design. The effect in the addition of Na_2SiO_3, RHA, and copper on the compressive strength and apparent porosity was investigated. It was found out that increasing Na_2SiO_3 and RHA significantly increased the compressive strength while increasing copper levels significantly decreased the strenght. Analysis of variance (ANOVA) showed that only RHA and copper had a significant effect to the apparent porosity of the porous geopolymer bricks. Elemental analysis of the mine tailings and x-ray diffraction analysis of the geopolymer were also employed in this study.

10:40 AM

(ICACC-S16-014-2020) Evaluation of geopolymer mortar based on a binary blend of class F fly ash and ground glass fiber using a sodium silicate-free activator

O. A. Amer*[1]; P. Rangaraju[1]; H. R. Dezfouli[1]

1. Clemson University, Civil Engineering, USA

Geopolymer-based binders have gained extensive attention in the last two decades as a sustainable alternative to cement-based binders. Various studies have shown that geopolymer binders have a lower carbon footprint, compared to cement systems, by 10% to 80%. This wide range in the carbon footprint is based mostly on the type of alkaline activator used in the geopolymer system. In particular, geopolymers requiring sodium silicate ($NaSiO_3$)-based activators provide a lower reduction in carbon footprint. When Class F fly ash (FFA) is used as source material in geopolymers, due to its semi-crystallinity, lack of adequate CaO and reactive Al_2O_3 content, the use of $NaSiO_3$ and heat curing become essential to achieve good compressive strength. In contrast, recent studies on geopolymers, using ground glass fibers (GGF) as the source material, showed that excellent strengths were achieved without the use of $NaSiO_3$ and only at moderate curing temperatures. Therefore, the objective of this study is to investigate the use of $NaSiO_3$-free activator with a binary blend of FFA and GGF at different curing temperatures. To this end, use of GGF up to 50% replacement of FFA was investigated. Results have shown excellent performance of the FFA+GGF system using only NaOH solution when compared to fly ashes activated by a combination of NaOH and $NaSiO_3$ solution.

11:00 AM

(ICACC-S16-015-2020) Extensive reuse of waste glass in geopolymer-like materials (Invited)

D. Ramteke[2]; D. Galusek[3]; P. Colombo[1]; E. Bernardo*[1]

1. University of Padova, Department of Industrial Engineering, Italy
2. University of Trencin, FunGlass (Centre for Functional and Surface Functionalized Glass), Slovakia
3. IIC SAS, Joint Glass centre, Slovakia

The recycling of common soda-lime glass is far from being complete, since recovered material may contain small polymeric, metallic and ceramic contaminations, having a negative impact on the quality of new glass articles from remelting. Consolidation at low temperature is attractive, besides for the energy savings, for the perspective of full reuse of contaminated fractions, actually configuring an important kind of waste. The present study is dedicated to the development of geopolymer-like materials, by simple insertion of fine glass powders (<30 μm) in aqueous solution of sodium aluminate (45wt%), at a glass/solution proportion of 50/50 wt%. After dissolution at room temperature (3 h, under low speed mechanical stirring), the suspensions are cast in plastic moulds and cured at 80-100 °C, overnight. The reaction between constituents leads to microporous semi-crystalline monoliths (porosity of ~40%), featuring hydroxy-sodalite as main crystalline phase, and exhibiting no degradation upon testing in boiling water. Operating with soda-lime glass powders after a preliminary dealkalinization treatment (3 h in acidic solution, pH=5) leads to a change of the crystalline phase, with zeolite A formation. The mechanical properties (elastic modulus up to 11 GPa, bending strength up to 20 MPa) compare well with those of cementious materials. Preliminary experiences on the foaming of mixtures are presented.

11:30 AM

(ICACC-S16-016-2020) Alkali-activation of mineral waste: Effect of composition and curing on final properties (Invited)

B. Coppola[1]; T. Jean-Marc[1]; L. Montanaro[1]; P. Palmero*[1]

1. Politecnico di Torino, Applied Science and Technology, Italy

Impressive amounts of wastes are produced yearly by extraction and transformation of natural stones. The finest fraction, named mud, is totally landfilled or even directly dumped in the ecosystem, implying economic, environmental and health issues. Due to such concerns, the reuse of mineral muds is an urgent need. Furthermore, the incorporation of this waste in other industrial processes could generate new business opportunities, while preserving natural resources. In this light, this study deals with the alkaline activation of mineral muds to develop new materials for the construction sector. As a challenge, we investigated the use of such muds alone, i.e. without the addition of other raw materials or by-products prone to alkaline-activation. Local muds characterized by different compositions were here investigated, ranging from pure siliceous waste (granite mud, from caves located in Piedmont) to pure carbonate one (marble mud, from caves in Tuscany). Design of mixtures, processing, materials development towards the elaboration of both dense and foamed samples are here illustrated. In the case of marble muds, the role of curing conditions on the physical and mechanical properties is deeply analysed. Finally, mechanisms for the alkaline activation of such highly crystalline mineral waste are proposed, in view of achieving high mechanical resistance besides sufficient durability.

Alkali Activated Materials

Room: Tomoka C

Session Chair: Enrico Bernardo, University of Padova

1:30 PM

(ICACC-S16-017-2020) Dissolution of Calcined Clays at the Atomic Scale: Evidence of Reactive Al(V) Sites (Invited)

N. Garg*[1]

1. University of Illinois Urbana-Champaign, Civil and Environmental Engineering, USA

Calcined clays like metakaolin are attracting significant research attention because of their potential to used as geopolymers or alkali-activated materials. This potential depends largely on their reactivity, especially dissolution under alkaline conditions. Identification and characterization of reactive sites in these amorphous calcined clays has so far not been reported. Here, we report dissolution kinetics of kaolinite (1:1 clay) and montmorillonite (2:1 clay) calcined at different temperatures under alkaline conditions. Solution compositions are determined in batch dissolution experiments, whereas 27Al and 29Si MAS and CP/MAS NMR are used to investigate the structure of the undissolved residues to identify sites that are reactive and undergo preferential dissolution. The Si and Al dissolution rates for optimally calcined kaolinite are 4 and 12 times larger than the corresponding rates for optimally calcined montmorillonite, in accord with the much higher reactivity of calcined kaolinite compared to calcined montmorillonite clays. This superior performance of kaolinite is explained by novel 27Al NMR results, which show strong evidence for preferential dissolution of highly reactive pentahedral aluminum sites. This study, therefore, gives new atomic scale information on the dissolution of calcined clays, elucidating the crucial role of reactive Al(V) sites.

2:00 PM

(ICACC-S16-018-2020) Durability of alkali-activated materials based on carbon fly-ash (Invited)

M. C. Bignozzi*[1]

1. University of Bologna, DICAM, Italy

Carbon fly ash-based alkali activated materials may be used as passive fire protection system for steel structures and/or as high temperature resistant materials. Passive fire protection systems are used to slow down the temperature rise of the steel substrate in case of fire. High temperature resistant materials are used where high temperature process occurs (e.g. furnaces, reactors, etc.). For both applications the mix design of the materials plays an important role to obtain the desired characteristics. In particular the addition of dispersed phases with different nature, density and average size (e.g. silica sand, alumina powders, expanded perlite) can promote different thermal stability. In this paper some examples of different carbon fly ash-based alkali activated materials will be reported and described in view of their ability to resist to high temperature exposure. The results obtained by several techniques such as dilatometric analysis, heating microscope analysis and medium scale fire test together with physical-mechanical characterizations are fundamental to improve the knowledge of the investigated materials in terms of their durability.

2:30 PM

(ICACC-S16-019-2020) Alkali activation: An option for waste valorization and inertization (Invited)

I. Lancellotti*[1]; C. Leonelli[1]; L. Barbieri[1]

1. University of Modena and Reggio Emilia, Department of Engineering "Enzo Ferrari", Italy

Aluminosilicates of non-common use, such as incinerator bottom ash and steel slag, can represent sustainable raw materials for alkali activation. Differently from other industrial processes (e.g. clinkerization, sintering processes), alkali activation doesn't require high temperature treatments, nor the use of high carbon footprint raw materials. Reuse of these no hazardous residues in high percentages by alkali activation can be an option for their valorization. This route demonstrated that the reactive Si/Al ratio is an important parameter to take into account for a proper formulation and to obtain different degree of reactivity. Not only the chemical analysis is important but also the mineralogy, in particular for complex systems such as wastes. Furthermore, alkali activation is a proper option also for hazardous solid and liquid wastes containing heavy metals and soluble anions. For liquid wastes an innovative aspect is the exploitation of the water contained in the waste avoiding drying step in their final management reducing both the use of energy and handling. For both solid and liquid wastes the capability to ineritize cations and anions is studied by the leaching tests (EN 12457). The immobilization efficiency is high for heavy metals and oxyanions, which show values line with the disposal in landfill for no dangerous wastes and it is improved for long curing time.

Infrastructure and Construction; Sustainable Materials

Room: Tomoka C

Session Chairs: Ange-Therese Akono, Northwestern University; Sylvie Rossignol, Laboratoire SPCTS

3:20 PM

(ICACC-S16-020-2020) Compositional Optimization of Metakaolin-based Geopolymer Mortar

N. Lies*[1]; O. D. Huang[1]; M. Radovic[1]

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

Geopolymer as an aluminosilicate network polymer have gained interest in recent years as an eco-friendly alterative to ordinary Portland cement for structural infrastructure applications. They can be processed with waste and/or widely available natural materials in room temperature. In this study, a comprehensive set of metakaolin-based geopolymer and geopolymer mortars with construction sand are processed using different Si/Al ratios and water/solid ratio for geopolymer binder and cured at ambient conditions for up to 28 days. Pure geopolymer and geopolymer mortar samples were tested for compressive strength, shrinkage, density, open porosity, and water loss during curing. The compositions are compared to Portland cement samples processed under the same conditions. The results show that composition of geopolymers plays a key role especially on the compressive strength. When comparing geopolymer and Portland cement samples, geopolymer samples have significant advantages when it comes to shrinkage and density. Samples with various compositions are characterized with Scanning Electron Microscopy (SEM) and Energy Dispersion Spectroscopy (EDS), to examine the morphological and structural effect from the various parameters. Overall, the study showed that metakaolin-based geopolymer has properties that are sufficient to replace Portland cement as the binder in construction mortars according to ASTM C270.

3:40 PM

(ICACC-S16-021-2020) Thermal Conductivity and Flexure Strength of Geopolymer Composites for Geothermal Housing Foundations

D. Samuel*[1]; A. Stumpf[2]; W. M. Kriven[1]

1. University of Illinois at Urbana-Champaign, Materials Science and Engineering, USA
2. Prairie Research Institute, Illinois State Geological Survey, USA

In order to reduce CO2 emissions related to heating and cooling structures, geothermal loops may be used to help control temperature. These can cycle water from underground through tubing embedded in a structure's foundation, cooling the structure in warm weather and warming it in cool weather. The efficacy of these loops may be improved by adjusting the thermal conductivity of the foundation material. Geopolymers are an alternative to traditional Ordinary Portland Cement-based concrete but have poor thermal conductivity, typical of an oxide ceramic. We explored several thermally conductive materials, including graphite powder, alumina powder, and chopped carbon fiber, as additives to metakaolin-based geopolymers to investigate the change in thermal conductivity achievable. We also tested the four-point flexure strength of graphite- and alumina-reinforced samples to determine whether they would be mechanically suitable for foundation materials and examined the microstructures to better understand the interaction between the geopolymer and additives.

4:00 PM

(ICACC-S16-022-2020) Development of Metakaolin-based Geopolymer as Alternative Soil Stabilizer

O. D. Huang*[1]; R. Samuel[2]; A. Banerjee[3]; A. Puppala[2]; M. Radovic[1]

1. Texas A&M University, Materials Science & Engineering, USA
2. University of Texas at Arlington, Civil Engineering, USA
3. University of Texas at Arlington Research Institute, AIS Division, USA

Recently, geopolymers have been investigated as an eco-friendly alternative to ordinary Portland cements and polymers to stabilize soil and subgrade for various transportation infrastructure since they can be processed with waste and/or widely available natural materials at room temperature with good mechanical properties and durability. In this study, metakaolin-based geopolymer pastes with different compositions were used in the small amount (up to 30 wt.% of geopolymer) to stabilize two types of soil typical in Texas and south-west region of USA. The optimization of geopolymer paste composition was carried out for both low-clay and high-clay soil to achieve maximum compressive strength of the stabilized soil after during at room temperature. In addition, the durability properties of stabilized soils, such as leachability and strength after extended capillary soaking in water were tested. Results of this study

shows that geopolymer-treated soil can reach significant strength increase (up to 400%) with minimal volumetric changes after relatively short curing time of 28 days. In addition, significant reduction in the weight increase was demonstrated after 28 days of soaking in water, for the soil treated with geopolymer. Overall, geopolymer has demonstrated to be an effective soil stabilizer especially where time for construction is limited.

4:20 PM

(ICACC-S16-023-2020) Study of compressive strength of polymeric composites with epoxy resin and piassava particules

J. P. Carvalho[1]; A. C. Neves[1]; F. P. Lopes*[1]; S. N. Monteiro[2]; C. F. Vieira[1]

1. State University of the North Fluminense, Advanced Materials Laboratory, Brazil
2. Military Institute of Engineering, Brazil

The broom industry consumes a large amount of piassava fiber, however, much of the batch received is discarded as waste because it does not contain the minimum size necessary to obtain the product that is sold. From the detection of this disposal, there was the interest in reusing this waste to create a composite material that replaces the incorporation of synthetic fiber by natural, in order to minimize the environmental impacts caused by these products. Natural fibers are obtained from plants and vegetables and have been valued as an environmentally sound alternative to replace non-recyclable materials. These synthetic materials generate a high energy consumption in their manufacture and are widely used in composite materials. This work was focused on high performance floor - HPF. The HPF is widely used in industrial and hospital sectors, has facility of application, high strength and durability. Therefore, in this work the compressive strength of composites formulated with epoxy matrix and incorporation of 0 to 30% in volumetric fraction of piassava was analyzed. The piassava fiber was cut in Cutting Mill. Subsequently, scanning electron microscopy analysis was performed on the fractures of the samples. The results showed the potential use of this composite with natural fiber particle incorporation as an alternative to materials from synthetic sources.

4:40 PM

(ICACC-S16-024-2020) Analysis of polimeric composites reinforced by piassava to create a high performace floor

J. P. Carvalho[1]; A. C. Neves[1]; F. P. Lopes*[1]; S. N. Monteiro[2]; C. F. Vieira[1]

1. State University of the North Fluminense, Advanced Materials Laboratory, Brazil
2. Military Institute of Engineering, Materials Science Department, Brazil

Given the constant search of the academic community to create composites materials that minimize environmental impacts, in other words, eco-friendly, efforts have been concentrated to obtain equal or superior properties using natural raw materials. Thus, the interest to use plant fibers to reinforce polymer matrix composites has grown considerably. Among the advantages of using natural fibers is the low cost, biodegradability and non-toxicity of fibers. On the other hand, polymeric matrices are also from renewable sources, such as castor oil polyurethane. The objective of this work is to create natural composites and to evaluate their application viability for high performance floor - HPF. Due to its wide use in companies and hospital areas, the HPF is commonly used with the addition of synthetic fibers or mineral aggregate. Therefore, water absorption and compressive strength were evaluated in this study. The composites were formulated with castor oil natural matrix and the piassava fiber was cut in Cutting Mill. The fiber volume fraction ranged from 0 to 30%. In addition, scanning electron microscopy was used after specimen rupture to observe the microstructure and the link between the phases in the fracture region of the composites. The results were analyzed according to Brazilian Standards and showed a potential of competitiveness of the natural composite in relation to the others.

5:00 PM

(ICACC-S16-025-2020) Use of Bamboo Waste as a Strength in Composite Manufacturing for use as OSB Panels (Invited)

M. D. Lopes[1]; F. P. Lopes*[1]; C. F. Vieira[1]

1. State University of the North Fluminense, Advanced Materials Laboratory, Brazil

The objective of this work is the reuse of bamboo fiber waste in the manufacture of composites for use as OSB type panels and in search of greater employability of this waste that would go to landfills or incinerated for this type of material. In order to produce a material of similar properties to the Oriented Strand Board (OSB), composites were produced with a vegetable polyurethane matrix, in the proportion of 0.20 and 40% of aligned fibers of bamboo residues. In order to have a better understanding of the bamboo residue used in the present work, the specific fiber mass was determined and a microstructural evaluation made by scanning electron microscopy (SEM). Commercial and composite OSB specimens were made, they were evaluated for their physical properties with thickness swelling and water absorption tests and for their mechanical properties with flexural, impact, perpendicular tensile and screw pullout tests., and post-assay fractography was analyzed by SEM. The properties of bamboo fiber reinforced composites obtained results significantly superior to those of OSB being feasible the use of composite material for use as OSB.

S18: Ultra-High Temperature Ceramics

UHTC Applications and Oxidation

Room: Coquina Salon A
Session Chair: William Fahrenholtz, Missouri University of Science & Technology

8:30 AM

(ICACC-S18-001-2020) Current and Future UHTC Research (Invited)

E. Wuchina*[1]

1. Office of Naval Research, USA

Navy interest in ultra high temperature ceramics (UHTCs) has a 30+ year history of research and development. Current efforts funded by ONR include MPE (Multiple Principal Element) transition-metal carbides, nitrides and borides - a field initiated by the ONR MURI effort currently in its final year. This work has generated a large volume of presentations and publications, and has spawned many similar efforts across the globe. With both computational and experimental elements, this highly successful program has led to a better understanding of the roles of bonding, crystal structure and diffusion on the thermal, mechanical, and chemical properties of these compounds. Separate investments focus on understanding thermal conductivity as a function of dopant level additions, as well as oxidation rate controlling mechanisms in carbides and borides. Additionally, work on additive processing of UHTC structures has started. New directions considered for future investment include chemical synthesis routes for composites processing and testing for combined effects using advanced techniques and diagnostics

9:00 AM

(ICACC-S18-002-2020) Porous ZrB_2 for transpiration cooling of hypersonic vehicles

R. Hedgecock*[1]; L. J. Vandeperre[2]

1. Imperial College, Materials, United Kingdom
2. Imperial College London, Materials, United Kingdom

Ultra-high temperature ceramics (UHTC) are an attractive material for leading edges of hypersonic vehicles due to high melting temperature and thermal conductivity. They are typically limited

in use by oxidation, degrading into oxides with reduced melting temperatures and thermal conductivity. Transpiration cooling has been proposed as a method of protecting the leading edge. By flowing an inert cooling fluid such as He or N_2 through a porous layer of UHTC the temperature can be reduced. As the fluid exits the material, a boundary layer is created which reduces heat flux to the surface and prevents oxidation of the UHTC. This work aims to create porous materials to meet the requirements of such a system. The UHTC layer must be permeable, with the fluid leaving the surface homogenously. Previously studies have shown partially sintered ZrB_2 can provide enough fluid flow for transpiration cooling to prevent oxidation completely below 1800 °C. In this work, the partially sintered ZrB_2 has been shown to densify when heated to 2000 °C, greatly reducing permeability. This prevents further transpiration cooling. This work then shows that densification can be prevented by promoting coarsening of porous ZrB_2. This is done through larger starting powers and increased oxygen contamination. The effect of a coarser microstructure on the strength and thermal conductivity of ZrB_2 is then assessed.

9:20 AM

(ICACC-S18-003-2020) Oxidation of $(Hf_{0.2}Zr_{0.2}Ti_{0.2}Ta_{0.2}Nb_{0.2})C$ and $(Hf_{0.2}Zr_{0.2}Ti_{0.2}Ta_{0.2}Nb_{0.2})B_2$ at 1800°C

L. Backman*[1]; J. Gild[2]; T. J. Harrington[3]; K. Vecchio[3]; J. Luo[2]; E. J. Opila[1]

1. University of Virginia, Materials Science and Engineering, USA
2. University of California, San Diego, Materials Science and Engineering Program, USA
3. University of California, San Diego, Department of NanoEngineering, USA

High entropy ultra-high temperature ceramics (UHTCs) are a class of multi-principal component materials that have expanded the number of known UHTC materials and compositions for use in extreme environments. The oxidation behavior of the high entropy $(Hf_{0.2}Zr_{0.2}Ti_{0.2}Ta_{0.2}Nb_{0.2})C$ and $(Hf_{0.2}Zr_{0.2}Ti_{0.2}Ta_{0.2}Nb_{0.2})B_2$ materials was tested at 1500°C, 1700°C, and 1800°C using a resistive heating apparatus in one atmosphere 0.1% PO_2 oxygen/argon gas mixture for times up to 15 minutes. The results showed an unexpected reduction in material consumption at 1800°C for all times tested, compared to the lower temperatures tested for both compositions. An in-depth analysis of the composition and morphology of the oxide scale and sub-surface regions for the 1800°C case will be presented and compared to the lower temperature cases. It is hypothesized that the oxide scale formed at this temperature is more protective compared to the other conditions tested.

9:40 AM

(ICACC-S18-004-2020) In-Depth Characterization of Selective Oxidation Products of Hafnium Carbide at 1300°C

J. A. Scott*[1]; X. He[2]; D. Lipke[1]

1. Missouri University of Science & Technology, Materials Science and Engineering, USA
2. University of Missouri, Columbia, USA

Graphite and HfO_2 can stably co-exist within certain ranges of oxygen partial pressures at temperatures less than 1700°C at 1 atm total pressure. Conditions conducive to carbon formation during oxidation of HfC can naturally develop at the interface between unoxidized parent material and its solid oxidation products once a sufficiently protective oxide scale forms. Prior work has shown that selective oxidation can result in such a carbon-bearing interphase under conditions consistent with bulk equilibrium thermodynamic predictions and at temperatures exceeding the predicted phase stability limit. Recent nano-scale characterization of the interphase formed upon oxidation of HfC at 1300°C indicates that the first carbon-containing oxidation product is amorphous with overall composition consistent with "HfO_2C". TEM observations reveal that this "HfO_2C" phase separates into graphite and nanocrystalline monoclinic HfO_2. Observations from complementary characterization create a consistent picture of the initial and final stages of formation of the carbon-containing interlayer at 1300°C that may provide clues to the origins of stability of carbon-bearing oxidation products under simulated hypersonic exposures. Elucidation of the chemical nature and properties of the interphase is necessary to understanding and controlling the oxidation behavior of HfC for extreme environment applications.

UHTC Simulation, Composites, and Carbides

Room: Coquina Salon A

Session Chair: Eric Wuchina, Office of Naval Research

10:20 AM

(ICACC-S18-005-2020) Diborides: Modelling the influence of isotope ratio on material properties and in-reactor behaviour for nuclear fuels and beyond (Invited)

S. C. Middleburgh*[1]; L. J. Evitts[1]; F. Martini[1]; I. Ipatova[1]; M. Rushton[1]; W. E. Lee[2]

1. Bangor University, Nuclear Futures Institute, United Kingdom
2. Imperial College London, Materials, United Kingdom

The thermal behaviour of diborides can determine the overall integrity of a component in a given environment - whether in an aerospace, nuclear or other extreme domain. The variation of boron isotope (^{10}B:^{11}B) has been investigated to determine the resulting variation in thermal conductivity in both ZrB_2 (used, for example, as a burnable absorber in nuclear fuel applications) and the advanced fuel candidate material - UB_2. Methods based on density functional theory calculations were used to determine the thermal conductivity as a function of temperature - both lattice vibrations and electronic contributions were considered. Experimental comparison shows good agreements with ZrB_2 values, but significant deviations exist for UB_2 (and are discussed and remedied). The impact of radiation damage and other thermal properties such as thermal expansion are subsequently considered using similar modelling methods and targeted experimental campaigns.

10:50 AM

(ICACC-S18-006-2020) Fabrication and characterization of carbon fibre reinforced UHTC composites (Invited)

A. Vinci*[1]; L. Zoli[1]; D. Sciti[1]

1. ISTEC-CNR, Italy

Ultra-High Temperature Ceramics are a class of materials characterized by very high melting points, high thermal and electrical conductivity and high ablation resistance and are a promising candidate for the use in extreme environments. The major drawback is their low fracture toughness and thermal shock resistance which severely limit their field of application. The ceramic matrix composites (CMCs) currently used in aerospace applications, based on fibre reinforced carbon or silicon carbide matrix, are characterized by excellent mechanical properties but are not suitable for temperatures above 1600°C. To overcome these limits, we are designing and manufacturing a new class of composites which combine the good mechanical properties of CMCs with the oxidation and ablation resistance of UHTCs. In this work we present the possible processes involved in the fabrication of these composites, such as slurry infiltration, hot pressing, spark plasma sintering and reactive melt infiltration, the main thermo-mechanical properties and how these are correlated to the microstructure and fibre matrix interface. Both boride and carbide-based composites are explored. Strength and toughness up to 500 MPa and 10 $MPam^{0.5}$ respectively were obtained, while oxidation resistance was validated in an arc-jet facility. Finally the feasibility of scale-up for the production or large specimens was addressed.

11:20 AM

(ICACC-S18-007-2020) Effect of Carbon Stoichiometry on Thermal Properties of Zirconium Carbide Ceramics

Y. Zhou*[1]; W. Fahrenholtz[1]; G. Hilmas[1]

1. Missouri University of Science & Technology, Dept. of Materials Science and Engineering, USA

ZrC_x ceramics with different carbon stoichiometries were produced by reactive hot pressing and heat treatment. Precursor powders were reacted at 1600°C for 3 hours and then densified at 2100°C with a pressure of 32 MPa. Heat treatment was used to promote dissolution of carbon into the lattice for some compositions. Archimedes' method was used to measure bulk density while true density was determined from lattice parameters measured by Rietveld refinement of x-ray diffraction patterns. The ordering of carbon vacancies was studied by neutron diffraction and selected area electron diffraction. Measured values of heat capacities, thermal diffusivities and coefficients of thermal expansion were used to calculate the thermal conductivities of ZrC_x ceramics with different carbon stoichiometry. DFT calculations were also performed, and combine with the thermal conductivity results, to aid in determining the intrinsic properties of ZrC_x ceramics.

11:40 AM

(ICACC-S18-008-2020) Mechanical and Thermal Properties of Zeta Phase Tantalum Carbide at Elevated Temperatures

E. C. Schwind*[1]; G. Hilmas[1]; W. Fahrenholtz[1]

1. Missouri University of Science & Technology, Dept. of Materials Science and Engineering, USA

Zeta phase tantalum carbide (ζ-Ta_4C_{3-x}) is an ultra-high temperature ceramic of interest to the ceramic engineering community due to its potential to provide high fracture toughness for extreme environment applications. Highly phase pure zeta phase tantalum carbide was synthesized and densified using reaction hot pressing with tantalum hydride powder and carbon black as precursors. X-ray diffraction with Rietveld refinement was used to ensure high phase purity. The microstructure of the synthesized ζ-Ta_4C_{3-x} was imaged using scanning electron microscopy. Fracture toughness and flexure strength were measured at room and elevated temperatures. Thermal diffusivity and heat capacity were measured at room and elevated temperatures and used, along with density, to calculate thermal conductivity as a function of temperature. Electrical resistivity was also measured at room and elevated temperatures to allow for calculation of the electronic and phonic contributions to thermal conductivity. This study is the first to provide fracture toughness, flexure strength and thermal conductivity at elevated temperatures for ζ-Ta_4C_{3-x}.

UHTC Synthesis and Processing

Room: Coquina Salon A

Session Chairs: Bai Cui, University of Nebraska, Lincoln; Emanuel Ionescu, Technical University Darmstadt

1:30 PM

(ICACC-S18-009-2020) Polymer-Derived Ultra-High Temperature Ceramics (UHTCs) and Related Materials (Invited)

E. Ionescu*[1]

1. Technical University Darmstadt, Materials Science, Germany

In the last few decades, remarkable research efforts and progress were done concerning the physical properties of UHTCs as well as their processing. Moreover, there are vivid research activities related to developing synthetic access pathways to UHTCs and related materials with high purity, tunable composition, nano-scaled morphology, or improved sinterability. Among them, synthesis methods considering preceramic polymers as suitable precursors to UHTCs have received increased attention in the last few years. As these synthesis techniques allow the processing of UHTCs from the liquid phase, they are highly interesting, e.g., for the fabrication of ultra-high temperature ceramic composites (UHT CMCs), additive manufacturing of UHTCs, etc. In the present talk, various synthesis methods using preceramic polymers to access UHTCs and related materials (i.e., (nano)composites thereof with silica former phases) are summarized and critically evaluated.

2:00 PM

(ICACC-S18-010-2020) On the Thermal Decomposition Mechanisms of Differing Polymer-Derived SiBCN Ceramics

K. McGarrity*[1]; H. Shluman[1]; P. Tumurugoti[1]; K. Ning[1]

1. Alfred University, USA

Efficient operation of energy, automotive, and aerospace systems is currently limited by the materials available for related environments exhibiting conditions such as high temperature, chemical corrosion, and mechanical fatigue. In the present work, the authors describe in detail the specific chemical mechanisms for the thermal decomposition of several ultra-high temperature silicoboron carbonitride (SiBCN) amorphous polymer-derived ceramics, which exhibit nanoscale inhomogeneity and consequential preferred bonding conditions. Through thermogravimetric analysis (TGA) coupled with mass spectrometry (MS) it has been determined that these materials, stable to temperatures exceeding 1800 C, depend strongly on their bonding conditions to remain useful at high temperatures. TGA-MS data are correlated with spectroscopic investigations into the specific bonds present in the ceramics in order to determine the effect of ceramic structure, and by extension, polymer precursor architecture, on thermal stability. After heating in air, oxidation resistance and oxide layer thickness are also characterized.

2:20 PM

(ICACC-S18-011-2020) Polymer-Derived (Hf,Ta/Ti)C/SiC Nanocomposites with Excellent High-Temperature Oxidation Resistance

E. Ionescu*[1]

1. Technical University Darmstadt, Materials Science, Germany

In the present work, a synergistic strategy to improve the high-temperature oxidation behavior of HfC/SiC is presented and discussed. The strategy consists of a combination of alloying TaC/TiC to HfC and of applying a passivation step to the ceramic nanocomposites prior to their exposure to high-temperature oxidation conditions. Possible mechanisms involved in the oxidation processes of (Hf,Ta)C/SiC and (Hf,Ti)/SiC ceramic nanocomposites are highlighted and critically assessed. It is shown that the passivation treatment activates the formation of silica, which is sluggish at moderate temperatures; whereas the alloying of HfC with TaC and TiC induces the formation of oxidic phases such as $Hf_6Ta_2O_{17}$ and $HfSiO_4$/$HfTiO_4$, respectively, which are very beneficial for the high-temperature oxidation behavior of the studied ceramic nanocomposites. The solid-solution effect contributes to a significant improvement of the short-term oxidation resistance of the studied nanocomposites, while the passivation of the materials prior exposure of high-temperature oxidation conditions provides a remarkably improved long-term behavior thereof.

2:40 PM

(ICACC-S18-012-2020) Low-cost synthesis of $(Hf_{1-x}Zr_x)B_2$ solid solution fine powders via CTR and AMR methods

J. Belisario*[1]; S. Mondal[1]; Z. Cheng[1]; A. Durygin[1]

1. Florida International University, Mechanical and Materials Engineering, USA

Synthesis of $(Hf_{1-x}Zr_x)B_2$ solid solution powder was conducted by two methods. The carbothermal reduction (CTR) method used a solution-based processing of low-cost $HfCl_4$, $ZrCl_4$, sucrose, and H_3BO_3 followed by heat treatment, while the alkali metal reduction

(AMR) method directly heat treated a powder mixture of $HfCl_4$, $ZrCl_4$, and an oxygen-free boron source of $NaBH_4$. The obtained products were characterized for phase purity and micromorphology. It was observed that high purity $(Hf_{0.5}Zr_{0.5})B_2$ and $(Hf_{0.25}Zr_{0.75})B_2$ solid solution fine powders were obtained from recipes with total metal : boron molar ratio of 1 : 3 via either the CTR or the AMR method, both containing a final heat treatment at 1500°C for 1 hour. However, during synthesis of $(Hf_{0.75}Zr_{0.25})B_2$ under the same heat treatment condition and same 1 : 3 total metal to boron molar ratio, HfO_2 and/or ZrO_2 formation were observed when using both methods. Yet, when total metal : boron molar ratio was changed to 1 : 8, no oxides were present. The possible reasons for the observation as well as the influences of processing parameters including temperature, holding times, boron content and molar ratios on single-phase solid solution formation and their microstructures are discussed.

3:20 PM

(ICACC-S18-013-2020) Synthesis of multi-composition UHTC powders using wet or dry process (Invited)

Q. V. Nguyen*[1]; H. Lee[1]; J. Kim[1]; S. Lee[1]; S. McCormack[2]

1. Korea Institute of Materials Science, Republic of Korea
2. University of Illinois at Urbana-Champaign, USA

Among ultra-high temperature ceramics (UHTC), HfC and TaC have received a special attention in the recent years due to the combination and tenability of their chemical and physical properties. Ta-Hf-C system was reported to have extremely high melting points. In particular, $Ta_{0.8}Hf_{0.2}C$ solid solution has attracted strong attention. The synthesis of $Ta_{0.8}Hf_{0.2}C$ powder with high purity, fine size and homogeneous chemical composition is important in order to maintain its intrinsic chemical and physical properties. For this purpose, my talk will focus on the synthesis of tantalum-hafnium oxide nano-powders which have the desired properties using the organic steric entrapment method and plasma method. High purity $Ta_{0.8}Hf_{0.2}C$ nano-powders were successfully synthesized by the carbo-thermal reduction of the oxide powders at 1400-1900°C. The sizes of the synthesized powders were in the range of 20-100nm. The effects of synthesis temperature and carbon contents on the phase composition and microstructures of the synthesized powders were analyzed.

3:50 PM

(ICACC-S18-014-2020) Mechanisms of Laser Shock Processing of Ceramic Materials (Invited)

F. Wang[1]; X. Yan[1]; C. Zhang[1]; L. Deng[1]; Y. Lu[1]; M. Nastasi[1]; B. Cui*[1]

1. University of Nebraska-Lincoln, USA

Laser shock processing (LSP) is a novel advanced manufacturing technique that utilizes a nanosecond pulse laser to generate plasma-driven shock waves, which can induce a high compressive residual stress to a depth of about 1 mm from the surface. It has been widely applied to metals to improve the fatigue and stress corrosion resistance. However, the application of LSP to ceramic materials has been limited and the associated mechanisms are poorly understood. Our research has reveal the fundamental mechanisms underlying the microstructural changes and mechanical properties in ceramic materials (such as SiC) in the LSP process. Transmission electron microscopy (TEM) characterizations revealed significant dislocation activities near the surface and grain boundaries, suggesting that the localized plastic deformation was generated by LSP at room temperature. X-ray diffraction analysis showed that the compressive residual stress in SiC surfaces can extend from the surface to a depth of 750 μm. The LSP-induced localized plasticity can improve the mechanical properties of SiC ceramics, such as the apparent fracture toughness and bending strength.

4:20 PM

(ICACC-S18-015-2020) Hafnium Diboride-Tantalum Diboride Solid Solutions formed by Spark Plasma Sintering

C. Zhang*[1]; A. Agarwal[1]; B. Boesl[1]

1. Florida International University, Mechanical and Materials Engineering, USA

HfB_2-TaB_2 solid solution were sintered by spark plasma sintering (SPS) without any sintering aids. Pure HfB_2 and TaB_2 powders were used as starting powders and mixed using ball milling method. The addition of TaB_2 relieved the poor sinterability of HfB_2. The mixed HfB_2-TaB_2 powder showed complete solid solution formation with much improved densification after SPS as compared to pure HfB_2 and TaB_2. The sintered pellets were subjected to oxidation testing at 3000 °C with gas flow at sonic speed provided by a plasma jet. HfB_2-TaB_2 solid solution sample displayed improved oxidation resistance and formed a dense oxide scale with smaller thickness as compared to pure borides. This study shows that solid solution of ultrahigh temperature ceramics (UHTC) is emerging class of new material displaying improved oxidation properties.

4:40 PM

(ICACC-S18-016-2020) Fabrication and shaping of ZrB_2-SiC Composites by reaction bonding with various precursors and consolidation techniques

T. G. Aguirre[1]; C. L. Cramer*[1]; R. Lowden[2]

1. Oak Ridge National Lab, Energy & Transportation Science Division, Energy and Environmental Sciences Directorate, USA
2. Oak Ridge National Lab, MSTD, USA

The combination of ZrB_2 and SiC has the potential to create ultra-high temperature ceramics (UHTCs) with improved mechanical properties and high oxidation resistance, but the processing of these two materials together is difficult and requires high temperatures. By reaction bonding precursor powders, the fabrication of ZrB_2-SiC composite is possible at lower temperatures. Reaction bonding produces a homogenous dispersion of reacted products. There have been many investigations into reaction bonding of ZrB_2-SiC composites, but there is little work involving volumetric, density, and shaping control. In this work, a lower temperature synthesis route for this UHTC is explored with emphasis on the volumetric, density, and shaping control. Specifically, ZrB_2-SiC composites were fabricated from various reactants. Different precursors were used, and the consolidation techniques included free forming, hot pressing, and spark plasma sintering. Microstructure, net shaping, mechanical properties, and oxidation resistance were investigated.

5:00 PM

(ICACC-S18-017-2020) Synthesis of multicomponent bulk metal nitride $(Nb_{1/3}Ta_{1/3}Ti_{1/3})N$ via reaction flash sintering

S. Mondal*[1]; A. Durygin[1]; J. Belisario[1]; V. Drozd[1]; Z. Cheng[1]

1. Florida International University, Mechanical and Materials Engineering, USA

Single-phase multicomponent metal nitrides (including high entropy metal nitrides) are potential candidates for various applications such as abrasion or corrosion resistant coatings due to their excellent properties like high hardness, chemical inertness, and thermodynamic stability. However, synthesis of bulk phase for those materials has not been reported yet. In this research, a single-phase bulk multicomponent transitional metal nitride $(Nb_{1/3}Ta_{1/3}Ti_{1/3})N$ has been successfully synthesized from mixed powders of NbN, TaN, and TiN via reaction flash sintering technique. This was performed using an inhouse flash sintering setup under a constant electric field of 30V/cm until current density reached a preset limit up to 26.4 A/mm^2.

The flash event, which is the abrupt increase in current and temperature, was achieved at room temperature without sample preheating. The sintered sample appeared to be densified to above 95 % relative density, which was confirmed by SEM. Single phase formation and compositional uniformity were verified by XRD and EDS, respectively. In addition, hardness and oxidation resistance of $(Nb_{1/3}Ta_{1/3}Ti_{1/3})N$ were characterized and compared to ingredient binary nitrides. The oxidation products of $(Nb_{1/3}Ta_{1/3}Ti_{1/3})N$ were also characterized. Implications for this research and directions for future research on these materials would be discussed.

Friday, January 31, 2020

FS3: Molecular-level Processing and Chemical Engineering of Functional Materials

Precursor and Materials Chemistry

Room: Coquina Salon C
Session Chairs: Thomas Fischer, University of Cologne; Sven Barth, Goethe University Frankfurt

8:30 AM

(ICACC-FS3-022-2020) Structure and chemical composition of (0001) inversion boundaries in piezotronic ZnO bicrystals (Invited)

H. Kleebe*[1]; M. Trapp[1]

1. Technical University of Darmstadt, Material Science, Germany

TEM and SEM investigations of ZnO bicrystal interfaces synthesized by epitaxial solid-state transformation revealed a distinctive relationship between dislocation formation and stable incorporation of large dopants such as bismuth. This synthesis method generates a strained meandering interface with a localized network of dislocations, facilitating the dopant incorporation essential for the creation of varistor type potential barriers. This Bi-retaining mechanism is of special interest for the design of piezotronic ZnO bicrystals, which benefit from a (0001)//(0001) alignment regarding the piezoelectric polarization vector, but are simultaneously disadvantageous for dopant segregation along coherent (0001) inversion boundaries. Grain-boundary incoherency is an important parameter determining the segregation of large insoluble dopants such as Pr or Bi to the interface. However, this immanent problem of the (0001)// (0001) orientation is solved by this specific synthesis route; both the piezoelectric response and the nonlinear current voltage characteristics were optimized. An enrichment of Bi along the interface was achieved without the need to deviate from the specified alignment. The findings were cross checked with a reference sample being diffusion bonded with the identical orientation but without the presence of a dislocation network along the bicrystal interface.

9:00 AM

(ICACC-FS3-023-2020) Polymer-derived cobalt-doped amorphous silica with hydrogen affinity (Invited)

S. Tada[1]; S. Ando[1]; Y. Daiko[1]; S. Honda[1]; S. Bernard[2]; Y. Iwamoto*[1]

1. Nagoya Institute of Technology, Japan
2. CNRS, IRCER, France

Co cation-doped amorphous silica materials were designed and synthesized through the Polymer-Derived Ceramics (PDCs) route, and characterized in terms of the oxidation state of Co species within the amorphous silica matrix, and chemical affinity toward hydrogen. Commercially available perhydropolysilazane was modified with $Co(acac)_3$ to afford a single source precursor for Si-Co-C-O-N system. The single source precursor was exposed to air in the presence of ammonia vapor at room temperature, and subsequently pyrolyzed in air at 873 K to afford X-ray amorphous Co-doped silica at the Co/Si atomic ratios ranging from 1/80 to 1/8. XPS, STEM and DRIFTS analysis revealed that the doped Co existed as Co^{2+} which modifying silica matrix and nanoparticles of 2 to 3 nm in size. As an initial study to characterize the hydrogen affinity, the OH/OD conversion behavior under deuterium follow at 773 K of the Co^{2+}-doped amorphous silica was in-situ monitored by the DRIFTS analysis, and it was found that the OH/OD conversion rate increased consistently with the degree of hydrogen-bonded hydroxyl formation, i.e. the amount of the doped Co^{2+} in the silica matrix. At the presentation, the effect of the amount of Co^{2+} doping on the deuterium self-diffusion coefficient will be shown and discussed.

FS4: Green Technologies and Ceramic/ Carbon Reinforced Polymers

Innovative Processing to Minimize Energy Utilization, Recycling, and Reduction of Processing Waste

Room: Halifax A/B
Session Chairs: Steve Bull, Newcastle University; Manabu Fukushima, AIST

8:30 AM

(ICACC-FS4-026-2020) Integrated Additive Manufacturing and Laser Processing for the Fabrication of Protonic Ceramic Electrochemical Cells (Invited)

J. Tong*[1]

1. Clemson University, Materials Science and Engineering, USA

A new integrated additive manufacturing and laser processing (I-AMLP) technique was discovered for producing protonic ceramic electrochemical cells (PCECs). The combination of microextrusion-based 3D printing and picosecond laser cutting/polishing allows fabricating versatile geometries such as tubes, cylinders, cones, lobed tubes, and other complex shapes protonic ceramic parts. With followed dip coating, the tubular PCECs half cells were obtained. The laser cutting of green layer deposited by microextrusion allows manufacturing microchannel membrane reactors. The further combining with spray coating and CO_2 laser sintering allows to deposit multilayers with an individual layer thickness between 5μm to 1000μm for producing components of electrolyte, electrodes and interconnect for PCECs. The laser sintering allows the formation of a component layer with desired phase composition cost-effective raw materials of carbonates and oxides. Furthermore, the laser operating parameters can be adjusted to achieve a fully dense layer for electrolyte and interconnect and highly porous layer for electrodes. The PCEC single cells manufactured by this new I-AMLP technique demonstrated promising performance.

9:00 AM

(ICACC-FS4-027-2020) Additive manufacturing of kaolinite-based clay with electric arc furnace steel dust

E. Ordonez[2]; H. Colorado*[1]

1. Universidad de Antioquia, Colombia
2. Universidad de Antioquia, Mechanical Engineering, Colombia

Electric arc furnace steel dust (EAF dust) is used in this research as a complemented material in the additive manufacturing (AM) of kaolinite-based clays. The AM technique used was the direct ink writing method. The addition of steel dust waste in the form of powder to the clay is beneficial to the environment because the residues can be immobilized and thus can reduce the contamination of water. EAF dust is a hazardous waste available in millions of tons

generated by the metallurgical industry worldwide. Therefore, the current investigation shows the possibility of using EAF dust not only as admixture with clays ceramics after sintering, but also in 3D printed parts. Different samples were built with different water to clay ratios (W/C), and with waste between 0 and 20% contents. Cylinders for compression tests were printed, and after a sintering process to 1100°C for 2 hours in air atmosphere, were tested in compression. Other materials characterization included scanning electron microscopy, density, Weibull statistics, rheology on the green body and creep analysis.

9:20 AM

(ICACC-FS4-028-2020) Carbon rich-polymer derived ceramic fibers and mats for energy applications (Invited)

G. Singh*[1]; Z. Ren[1]; C. Gervais[2]

1. Kansas State University, Mechanical and Nuclear Engineering Dept., USA
2. UPMC, LCMCP, France

Polymer-Derived Ceramic (PDC) fibers have gained attention for the intrinsic thermal stability and mechanical strength with simple and cost-effective synthesis techniques. Here, carbon-rich SiOCN fibers were synthesized via hand-drawing and electrospinning followed by pyrolysis of a hybrid precursor of cyclotetrasilazane, cyclosiloxane and organic polymers. The type of silazane and siloxane are considered as a major precursor for SiCNO fibers, although these preceramic polymers are unspinnable if used pure, due to their unfavorable physical properties (low viscosity) and chemical structure (cyclic rather than linear structure). Investigations on structural and compositional development of the fibers were mainly conducted via Raman spectroscopy, Fourier-Transform InfraRed spectroscopy (FTIR), Scanning Electron Microscopy (SEM), X-ray Photoelectron Spectroscopy (XPS), Nuclear Magnetic Resonance (NMR) and ThermoGravimetric Analysis (TGA) to determine spinnability, free carbon content, crosslinking and pyrolysis behavior of the fibers, respectively.

9:50 AM

(ICACC-FS4-029-2020) Nano-Lignin - A Unique Material System for Designing High Performance Materials (Invited)

S. C. Borrillo[1]; S. Javaid[1]; M. Dey[1]; S. Gupta*[1]

1. University of North Dakota, Mechanical Engineering, USA

There is a urgent need for environmentally conscience products which can directly allign with the principles of circular economy. Natural polymers derived from agricultural byproducts have emerged as source. In this study, we report the effectiveness of a novel process to form lignin nanoparticles (LNP) by using different types of lignin. As a part of this study, a novel ultrasonication spray method was developed to produce a solid nano-powder useable in additional applications. Applications of these LNP may include antimicrobial food packaging, mechanical strengtheners in composite materials, and can be used to design high performance composites. As a case study, the synthesis and characterization of cellulose acetate (CA) - lignin composites will be presented.

10:20 AM

(ICACC-FS4-030-2020) Continuous Forming and Secondary Processing Technology for Long Composite Materials (Invited)

A. Nakai*[1]

1. GIFU University, Faculty of Engineering, Japan

Replacing metals with fiber reinforced composites is attractive to the field of civil and construction engineering. For example, L-shaped angles utilized as skeleton frames and internal reinforcement of a building require lightweight and high durability to improve workability and safety at the construction site. In this study, pultrusion was first selected for the manufacturing method of L-shaped angles because of a continuous process with high productivity. The development of pultrusion technology for manufacture of composite materials leads to an increase in production rate, reduction in labor cost, and improvement in processing accuracy. Since thermoplastic composites are faster in molding speed than thermoset composites, thermoplastic composites are suitable for pultrusion. Next, roll forming processing was examined. A long molded plate having been completely impregnated was prepared, reheated locally, and processed into an L-shape using a roll-shaped multi-stage mold. As a result, they have found the possibility of achieving higher productivity than pultrusion. On the other hand, the drawback of this processing technique is that it is necessary to prepare a long shaped plate which has been impregnated. Finally, by combining the above-mentioned pultrusion and roll forming technology, we developed a new processing technology that compensates for each other's shortcomings.

S9: Porous Ceramics: Novel Developments and Applications

Membranes and High SSA Ceramics

Room: Coquina Salon F

Session Chair: Thomas Konegger, TU Wien - Vienna University of Technology

8:30 AM

(ICACC-S9-027-2020) Development of Thermally Stable Aerogels for Aerospace Applications

N. S. Olson*[1]; F. I. Hurwitz[2]; J. A. Krogstad[1]

1. University of Illinois at Urbana-Champaign, Materials Science and Engineering, USA
2. NASA Glenn Research Center, USA

Aerogels demonstrate extraordinarily low thermal conductivity and low density, presenting a promising form of lightweight insulation for aerospace applications. The primary challenge is stabilization of the highly porous structure at temperatures to 1200 °C. Upon collapse of the pore structure, the favorable thermal properties and low density are diminished. Through investigation of novel syntheses and aerogel formulations starting with yttria-stabilized zirconia (YSZ), high temperature aerogels are identified. YSZ has long been the gold standard for a low thermal conductivity material used in thermal barrier coatings. However, there is a growing body of literature on alternative rare earth dopant schemes that demonstrate lower thermal conductivity and improved thermal stability. By employing YSZ and related compositions in a highly porous aerogel, the thermal conductivity can be markedly decreased. Several rare earth-doped zirconia aerogels are synthesized to investigate the effect of composition on morphology, phase behavior, and thermal stability. To develop insight on aerogel synthesis, the effect of water content, solids loading, and precursor on aerogel structure are studied by physical properties, surface analysis, and microscopy. With a better understanding of aerogel structure and performance, metal oxide aerogels can be developed to withstand the extreme environments of space exploration.

8:50 AM

(ICACC-S9-028-2020) Dynamic Hysteresis Scanning of SiCO Aerogels

P. Taheri*[1]; P. Kroll[1]; J. Kenvin[2]; J. Lang[1]

1. University of Texas, Arlington, USA
2. Micromeritics, USA

We study impact of solvents on porosity and pore morphology of two aerogels by Differential Hysteresis Scanning (DHS). Polymeric silicon oxycarbide (SiCO) aerogels are synthesized from PMHS and

DVB in either acetone or cyclohexane and dried using supercritical CO2. Standard porosity analysis is augmented by high-resolution argon sorption experiments, systematically scanning the hysteresis loop of the sorption-desorption curve at intermediate pressures. Results of DHS are analyzed using an advanced modeling framework that allows to map and quantify geometry, size, and connectivity of pores in the material. BET analysis of the aerogel synthesized in acetone (A-AG) yields a Specific Surface Area (SSA) twice as large as the SSA of the sample synthesized in cyclohexane (C-AG). BJH analysis indicates that the average pore size of A-AG is also twice as large as that of C-AG. However, DHS reveals that pores in C-AG are constricted: wide pores are connected to each other through small windows. Thus, BJH analysis captures the small window size only, while the much large diameter of the pore behind the window is only revealed by DHS. Similar analysis of A-AG indicates the persistence of pyramidal pores, with a pore window diameter agreeing with pore sizes provided by BJH analysis. Overall, we demonstrate that DHS is an indispensable tool to analyze pore hierarchy in porous materials.

9:10 AM

(ICACC-S9-029-2020) Preparation and Gas Separation study of Ceramic supported Membrane Reactor for Hydrogen Production from Syngas

C. D[1]; S. K L*[2]; M. C D[3]

1. M V J College of Engineering, Chemical Engineering, India
2. Siddaganga Institute of Technology, Chemical Engineering, India
3. Bharat Heavy Electricals Ltd, Ceramic Technological Institute, India

Membrane reactor is a process intensification system where reaction and separation take place in one unit resulting in enhanced reaction rates. One of the useful applications is for the production and purification of H_2 from syngas using water gas shift reaction. Dense Pd-based membranes are commonly used because of high H_2 selectivity, thermal stability and mechanical resistance. Supported membranes prepared by incorporating a thin Pd layer on the surface of a porous material avoids the issue of thick Pd layer hindering hydrogen permeate rate and reduces membrane cost. Ceramic supports provide a smoother surface with control on porosity and narrow pore size distributions. In this work, Membrane reactor was designed, fabricated and evaluated using Pd membranes. Pd membranes were prepared on Alumina ceramic supports by Electro-less plating method and evaluated for H_2/N_2 selectivity and separation factor at 400ºC and compared with commercial Pd membranes. The membrane reactor was filled with ceria based catalysts for water gas shift reaction and was experimented at 400ºC with feed gas containing 20% CO. The membrane reactor showed CO conversion efficiency of 88% and H_2 purity of 75%. These experiments demonstrate the membrane reactor designed using a Ceramic supported Pd membrane for efficient and purer H_2 production from syngas can be used for industrial applications.

9:30 AM

(ICACC-S9-030-2020) Studies on effect of various parameters that affect the effective waste water treatment using submerged ceramic membrane bioreactor

S. Surappanahalli Rajanna*[1]; G. Madhu[1]; S. Easwaran[1]; C. Madhusoodana[2]

1. M S Ramaiah Institute of Technology, Bangalore, Chemical Engineering, India
2. BHEL, Ceramic Division, India

It is very important to control the water pollution in order to avoid water crisis. The present work focuses on treating waste water using an efficient alternative to conventional sedimentation tanks, namely, submerged ceramic membrane bioreactor (SCMBR). The main focus of the present study is to understand the effect of various parameters such as temperature, pressure, pH and sludge retention time. It is found that with increase in operating pressure, there is an innocuous reduction in operating time, thereby reducing the operation cost. The quality of effluent is found to be better at higher temperature, increased sludge concentration and lower pH. Thus the present study signifies the optimum conditions at which the SCMBR can be used so as to reduce the operating costs without affecting the efficacy of the process.

S10: Modeling, Genome, Informatics, and Machine Learning

Prediction of Crystal Structure and Related Properties I

Room: Coquina Salon G
Session Chair: Jingyang Wang, Shenyang National Laboratory for Materials Science, IMR

8:30 AM

(ICACC-S10-027-2020) Creation of predictive models of 4f-5d transition energy of Ce^{3+} in garnet-type oxides using only structural parameters of host crystals as attributes (Invited)

K. Ogasawara*[1]

1. Kwansei Gakuin University, Department of Chemistry, Japan

The 4f-5d transitions of Ce^{3+} in crystals are utilized in various optical materials such as solid-state lasers, scintillators and phosphors. Therefore it is important to predict the 4f-5d transition energy of Ce^{3+} in crystals for theoretical design of novel optical materials. In order to establish an efficient method for prediction, a combina-tion of first-principles molecular orbital (MO) calculations and machine learning is useful. Using this approach, we have recently created predictive models of the 4f-5d transition energy of Ce^{3+} in garnet-type oxides using the structural parameters and the electronic parameters obtained by MO calculations using simple CeO_8 clus-ters as attributes. In this work, in order to establish a more efficient method, systematic MO calculations for Ce^{3+} in fictitious garnet-type oxides with various combinations of structural parameters were performed. Using these results as the training data, the predic-tive models of the electronic parameters were created by machine learning. As a result, by combining the predictive models of the elec-tronic parameters based on structural parameters with the predictive models of the 4f-5d transition energy based on the electronic and structural parameters, efficient predictive models of the 4f-5d transition energy based on only structural parameters were created.

9:00 AM

(ICACC-S10-028-2020) Predicting the phase stability of high entropy pyrochlore oxides (Invited)

K. Pitike*[1]

1. Oak Ridge National Laboratory, Materials Science and Technology Division, USA

High entropy, multicomponent systems are interesting due to the role that cation disorder may play in defining their mechanical, magnetic, reversible energy storage properties, etc. Within the class of high entropy oxides; rocksalt, fluorite, spinel and perovskite phases have all been recently synthesized. Complementing these endeavors, the current work explores the stability of high entropy oxides in the pyrochlore (HEPy) A_2 B_2O_7$ phase – where the B-site is randomly and equally occupied by five chemical species from the set of {Hf, Nb, Sc, Sn, Ta, Ti, Zr} – through a collaborative computational and experimental efforts. We use density functional theory (DFT) calculations to analyze the phase stability of the indi-vidual $AB O_x and binary $A(BB')$O_x$ oxides. Furthermore, the enthalpies of mixing – calculated using DFT for binary oxides in their potential respective phases – are used to construct a nearest neighbor (NN) model. The candidates for the HEPy oxides are predicted from the configurational landscapes of the five component

oxides, estimated through Monte Carlo simulations using the NN model. Our approach allows us to evaluate potential impurity phases thereby making realistic predictions of novel multicomponent oxides that can be synthesized. This work was supported by LDRD Program of ORNL, managed by UT-Battelle, LLC, for the U. S. DOE using computational resources at at OLCF, CADES at ORNL and NERSC.

9:30 AM

(ICACC-S10-029-2020) Light-Illumination Dependent Electronic and Atomic Structures of Glide Dislocations in Inorganic Semiconductors (Invited)

K. Matsunaga*[1]

1. Nagoya University, Materials Physics, Japan

It was recently found that zinc sulfide single crystals having the zinc blend structure can undergo significantly large plastic deformation even at room temperature in complete darkness (Oshima, Nakamura, and Matsunaga, Science (2018)). This result is closely related to changes in mobility of glide dislocations in ZnS due to light illumination. In order to clarify such a light-illumination effect, DFT calculations were performed for glide dislocations in ZnS. It was found that glide dislocations in ZnS are not perfect dislocations but dissociate into Shockley partial dislocations on the {111} planes. These partial dislocations cores tend to have smaller band gaps and excess electrostatic charges, so that extra electrons and holes excited by light illumination can be favorably trapped at the partial-dislocation cores. It was also then found that the partial dislocations can undergo bond reconstructions at the cores in carrier trapping. Since the reconstructed atomic structures should be broken on the onset of dislocation glide, the core reconstruction should decrease mobilities of the glide partial dislocations. This may explain the decreased partial-dislocation mobilities, namely hardening of ZnS, under light illumination, which was suggested by experiment.

Prediction of Crystal Structure and Related Properties II

Room: Coquina Salon G

Session Chairs: Katsuyuki Matsunaga, Nagoya University; Krishna Chaitanya Pitike, Oak Ridge National Lab

10:20 AM

(ICACC-S10-030-2020) Modeling General Grain Boundaries (GBs): From Computing GB Diagrams to Understanding GB Superstructures and an Electric Field Induced GB Transition (Invited)

C. Hu[1]; J. Nie[1]; Z. Yu[2]; J. Luo*[1]

1. University of California, San Diego, USA
2. Fuzhou University, China

Grain boundaries (GBs) can be considered as 2D interfacial phases that are called "complexions" to differentiate them from thin layers of bulk (3D) Gibbs phases. They can exhibit structures and compositions that are distinct from any 3D phase, and they often control the materials processing, microstructure evolution, and various mechanical and physical properties. Here, "general" GBs are of particular importance for the real polycrystalline materials, but they are much less understood. This talk will first review and discuss our on-going studies of computing GB "phase" (complexion) diagrams as a new and generally useful materials science tool via three approaches: thermodynamic models, atomistic simulations, and machine learning. Subsequently, two recent studies will also be presented and discussed. On the one hand, a discovery of a GB superstructure with highly asymmetric and off-the-center segregation in WC, along with interfacial disordering and symmetry change on the opposite sides, extends our knowledge of GB segregation and complexions. On the other hand, an electric field induced GB structural transition has been observed in ZnO, which results in enhanced grain growth. In both cases, first-principles based calculations can justify and explain all observations. All studies are focused on general GBs.

10:50 AM

(ICACC-S10-031-2020) Transition Metal Diborides Investigated by X-ray Spectroscopy and Ab-Initio Electronic-Structure Calculations (Invited)

M. Magnuson*[1]

1. Linkoping University, Sweden

Transition metal-borides are known to exhibit interesting materials properties from superhardness to superconductivity with high thermal and chemical stability. These materials have potential to be used for the next generation of hard, wear- and corrosion-resistant coatings. In particular, this is the case for borides with an AlB_2 type of structure (Strukturbericht notation C32), where the B atoms form honeycombed, graphite-like sheets that are interleaved between hexagonal close packed Zr atoms. From a combination of ab-initio electronic structure calculations and analysis with XPS, XRD, XANES, and EXAFS, the local chemical bonding structure and structural properties with atomic distances are investigated in epitaxial ZrB_2 films and compared to properties to those of the ZrB_2 compound target from which the film was synthesized as well as a bulk a-Zr reference. Epitaxial films are shown to be promising for property determination of borides and future exploration also of other material classes. Trends in transition-metal diborides are compared and dicussed with respect to chemical bonding, orbital overlap, and electronic structure.

11:20 AM

(ICACC-S10-032-2020) DFT Study of the Cleavage Planes in Σ9 {122} SiC -3C Grain Boundary

J. S. Dunn*[1]; S. P. Coleman[1]; M. C. Guziewski[1]; C. M. Carlin[1]

1. U.S. Army Research Laboratory, USA

A DFT study of interfacial energies and cleavage planes in the Σ9 {122} SiC -3C tilt grain boundary (GB) was performed to determine the lowest energy fracture path. This boundary has three unique reconstructions: (1) Symmetric non-polar interface composed of 5 and 7 member rings containing either a single C-C or Si-Si 'wrong bonding' (i.e. different from bulk Si-C bonding) (2) Non-symmetric polar interface composed of rings containing one C-C wrong bond, and (3) Non-symmetric polar interface composed of rings containing one Si-Si wrong bond. The interfacial energy for the nonpolar and polar interfaces are 1.346 eV and 1.336 eV indicating that the polar interface is more stable. However, it should be noted that due to periodic boundary conditions, the interfacial energy calculated for the polar interface is an average of the two unique polar ('C-C' and 'Si-Si') interfaces, whereas the nonpolar interface is symmetric. In this study, we calculated the cleavage energy and analyze the electronic structure for eight fracture planes for each of the nonpolar, polar 'C-C' and polar 'Si-Si' interfaces and compare the results to the lowest energy cleavage plane as determined using a machine learning optimization algorithm. Machine learning allows a more efficient search of the interfacial configuration space, which becomes increasing important for grain boundaries with a high degree of chemical and structural complexity.

S18: Ultra-High Temperature Ceramics

UHTCs: High Entropy Materials

Room: Coquina Salon A
Session Chair: William Fahrenholtz, Missouri University of Science & Technology

8:30 AM

(ICACC-S18-018-2020) Hardness and Young's Modulus Anisotropy in High Entropy Boride Ceramics

A. Stanfield*[2]; L. Feng[2]; F. Monteverde[1]; G. Hilmas[2]; W. Fahrenholtz[2]

1. CNR-ISTEC, Italy
2. Missouri University of Science & Technology, Dept. of Materials Science and Engineering, USA

The purpose of this study is to quantify the anisotropy in hardness and Young's modulus for high entropy boride ceramics using nanoindentation. Nominally phase-pure $(Ti_{0.25},Y_{0.25},Nb_{0.25},Hf_{0.25})B_2$ and high purity ZrB_2 were densified by direct current sintering. Grains were grown to ~10 μm to mitigate grain boundary effects during testing. Hardness and Young's modulus were measured by nano-indentation of the polycrystalline specimen surface. An array of indents tested at least 100 grains of varying crystallographic orientations at loads from 5 mN to 50 mN. During indentation, force and penetration depth were recorded at 0.2 s intervals and used to calculate H and E. Data were catalogued and compared to electron backscatter diffraction maps of the indented area. Phase information was generated for electron backscatter diffraction by Rietveld refinement of x-ray diffraction patterns. Euler angles of each indented grain were calculated and correlated to the measured mechanical properties. The crystallographic orientation dependence of hardness and Young's modulus will be discussed for high entropy borides and compared to high purity ZrB_2.

8:50 AM

(ICACC-S18-019-2020) A High Entropy Route to Tough Ceramics

M. Hossain*[1]; T. M. Borman[1]; D. Brenner[2]; J. Maria[1]

1. Pennsylvania State University, Materials Science and Engineering, USA
2. North Carolina State University, Materials Science and Engineering, USA

High entropy carbide (HEC) ceramics present great promise and opportunity to synthesize new ultra-high temperature ceramics with exceptional mechanical and chemical properties. In the current study, an ab-initio approach was implemented to predict tough HEC ceramic compositions which were subsequently synthesized using physical vapor deposition. Nanoindentation experiments were performed to characterize the ductile vs brittle fracture. The HEC shows no brittle fracture under the load limit of the instrument whereas binary and ternary carbides constituents fail via formation and propagation of cracks at 20% of the load limit. Evaluation of the electronic structure for the HEC composition reveals that the valence electron concentration regulates the Fermi energy which alters the shear and bulk modulus of the HEC. Thoughtful compositional design can adjust the Fermi energy to obtain a certain modulus ratio which should lead to ductile characteristics. Based on the theoretical and experimental findings a universal descriptor is proposed to synthesize numerous tough HEC compositions.

9:10 AM

(ICACC-S18-020-2020) Asynchronously Patterned Pulsed Sputtering (APPS) for Rapid UHTC Compositional Exploration

T. M. Borman*[1]; M. Hossain[2]; J. Maria[1]

1. Pennsylvania State University, Materials Science and Engineering, USA
2. Pennsylvania State University, USA

The authors describe a new technique, asynchronously patterned pulsed sputtering (APPS), for deposition of ternary and quaternary UHTCs using multiple High Power Impulse Magnetron Sputtering (HiPIMS) sources. A key advantage of HiPIMS (relative to DC or RF sputtering) is the decoupling of deposition rate from energetics allowing increased film quality. However, when co-sputtering with multiple HiPIMS sources, some of this freedom may be lost to control flux. APPS consists of one source running at a given frequency for every pulse while the second source runs asynchronously at the same frequency, following a pattern of pulses and skips. By changing the ratio of pulses to skips, the relative fluxes of the two sources can be controlled without impacting the energetics critical for quality UHTC film deposition. APPS enables deposition within a few atomic percent across the entire composition range from just two deposition rate (flux) measurements, with more precise control from further measurements in regions of interest. By using APPS a wide range of UHTC ternary and quaternary compositions can be rapidly synthesized and tested for properties of interest such as thermal conductivity, hardness, and ductility.

9:30 AM

(ICACC-S18-021-2020) Synthesis, densification, and characterization of high-entropy carbide ceramics

L. Feng*[1]; W. Fahrenholtz[1]; G. Hilmas[1]

1. Missouri University of Science & Technology, Dept. of Materials Science and Engineering, USA

High-entropy carbide powders were synthesized by a two-step process consisting of carbothermal reduction followed by solid solution formation. Nominally pure (Hf,Zr,Ti,Ta,Nb)C had a single-phase rock salt structure with an average particle size of about 550 nm and an oxygen content of 0.2 wt%. The fine particle size was due to the use of high-energy ball milling prior to carbothermal reduction at the relatively low synthesis temperature of 1600°C. The powders synthesized by carbothermal reduction were hot pressed at 1900°C to produce nominally pure and dense (Hf,Zr,Ti,Ta,Nb)C ceramics with an average grain size of 1.2 μm. Low sintering temperature, high relative densities, and fine grain sizes were achieved due to the use of fine starting powders. Dense ceramics had Young's modulus of 452 GPa, Vickers hardness of 24.8±0.8 GPa, fracture toughness of 3.5±0.3 $MPa \bullet m^{1/2}$, and flexural strength of 421±27 MPa at room temperature. The flexural strength remained above~400 MPa up to 1800°C, then decreased to 318±21 MPa at 2000°C and 93±10 MPa at 2300°C. The presentation will discuss the synthesis, densification, and mechanical properties of (Hf,Zr,Ti,Ta,Nb)C ceramics that were systematically investigated in the present work.

UHTC Phase Equilibria and Properties

Room: Coquina Salon A
Session Chair: William Fahrenholtz, Missouri University of Science & Technology

10:10 AM

(ICACC-S18-022-2020) Phase Equilibria and Symmetry relations in the HfO_2-Ta_2O_5-TiO_2 system up to 3000C (Invited)

S. J. McCormack*[1]; K. Tseng[4]; R. Weber[2]; S. Ushakov[3]; A. Navrotsky[3]; W. M. Kriven[4]

1. University of California, Davis, Materials Science and Engineering, USA
2. MDI, USA
3. University of California, Davis, Peter A. Rock Thermolab and NEAT ORU, USA
4. University of Illinois at Urbana-Champaign, USA

Most material applications rely on a foundation of knowledge of phase equilibria. Here a systematic approach to the rapid production of the high-temperature HfO_2-Ta_2O_5-TiO_2 phase diagrams is presented that highlights the combined use of: (i) in-situ high-temperature X-ray diffraction (up to 3000C) and the (ii) extraction of atomic motifs with associated material symmetry analysis. The tools and methodologies developed herein are essential for the development of high-temperature materials. The extreme temperatures were achieved by utilizing a quadrupole lamp furnace (QLF)

(200 – 2000C) and a conical nozzle levitator system equipped with a CO_2 laser (CNL) (700 – 3000C) in conjunction with synchrotron X-ray powder diffraction. These devices allow for (i) high-temperature phase identification, (ii) crystal structure determination using the charge flipping algorithm, (iii) extraction of anisotropic coefficients of thermal expansion, (iv) measurement of lattice variant deformation during phase transformations and (v) identification of atomic motifs within material systems. This intersection of phase equilibria and crystallographic symmetry analysis is an innovation methodology that aims to extend our fundamental understanding of material systems. Here we describe how this data is used to build symmetry relationships within the traditional representation of phase equilibria for the HfO_2-Ta_2O_5-TiO_2 system.

10:40 AM

(ICACC-S18-023-2020) Direct consideration of point defects in CALPHAD modelling of zirconium carbide (Invited)

T. Davey*[1]; Y. Chen[1]

1. Tohoku University, School of Engineering, Japan

Zirconium carbide is of interest in nuclear and aerospace industries due to its stability at extremely high temperatures. Its properties are strongly affected by the presence of significant structural vacancies. Conventional CALPHAD-type phase diagram models do not directly consider such defects; instead they are implicitly considered via thermodynamic data relating to non-stoichiometric compounds. The widely-used C-Zr phase diagram from Fernandez-Guillermet has been previously shown to be intrinsically incompatible with our physical understanding of structural vacancies. Defect-related properties such as formation energies and defect-defect interaction energies are challenging to obtain experimentally with the required accuracy. In this work, state-of-the-art first-principles calculations of defect-related properties are used to inform development of Gibbs energy models that may be used in cases where many structural point defects are present. This is done both by incorporating such information directly into existing thermodynamic databases with conventional Gibbs energy descriptions, and through development of new Gibbs energy models. Directly considering defect-related properties in the development of the thermodynamic database produces a more physically consistent description and may allow further predictive ability of the phase diagram in regions where experimental information may be scarce.

11:10 AM

(ICACC-S18-024-2020) Solute Characterization and Mechanical Properties of (Zr,Ta)B2 Ceramics

A. N. Dorner*[1]; D. J. Barton[2]; G. B. Thompson[2]; G. Hilmas[1]; W. Fahrenholtz[1]

1. Missouri University of Science & Technology, Materials Science and Engineering, USA
2. University of Alabama, Metallurgical and Materials Engineering, USA

Zirconium diboride ceramics containing solid solution additions of tantalum ranging from 0 to 6 at% were synthesized by reactive hot pressing of zirconium and tantalum hydrides and amorphous boron. Microstructural analysis demonstrated that the (Zr,Ta)B2 ceramics reached nearly full relative density and were nominally phase pure. Analysis of x-ray diffraction data revealed that tantalum was fully incorporated into the ZrB2 structure and that the lattice parameters decreased with increasing tantalum addition. Electron microscopy and atom probe tomography were used to characterize Ta distribu-tion and segregation in doped materials. The mechanical properties that were measured included Vickers hardness, flexure strength, fracture toughness, and elastic moduli. The elastic moduli were measured by impulse excitation and resonant ultrasound spectros-copy. Properties were tested from room temperature up to 2200° C. Changes in properties were analyzed to determine the effects of tantalum content on ZrB2 and to guide future studies into the effects of additives and impurities on the processing and properties of diboride-based ultra-high temperature ceramics.

LATE ADDITIONS

Monday, January 27, 2020

1:30 PM

(ICACC-S7-031-2020) Possible continuous (successive) fabrication of nano-structured ceramics via soft solution processing (Invited)

M. Yoshimura*, National Cheng Kung University, Taiwan

Practical devices would be better to be fabricated via continuous and/or successive Processes. Presently, however, they have generally been fabricated artificially and/or industrially by so-called high-technology, where high temperature, high pressure, vacuum, molecule, atom, ion, plasma, etc. using expensive equipments thus they consumed huge amount of resources and energies thus exhausted huge amounts of wastes: materials, heats and entropy. The major reasons might be 1) The reactants should be nano-sized species, 2) high-energy reaction might be required, thus 3) They cost economically and environmentally. To save this tragedy, a) we must consider "Cascade use of Heats", and b) "Low energy Production of advanced materials via solution-based technologies." c) Continuous (Successive) Fabrication will be possible in solution process(es).
We proposed in 1995 an innovative concept and technology, "Soft Processing" or "Soft Solution Processing," which aims low energetic (=environmentally friendly) fabrication of shaped, sized, located, and oriented inorganic materials in/from solutions. When we have activated/stimulated interfacial reactions locally and/or moved the reaction point dynamically, we can get patterned ceramic films directly in solution without any firing, masking nor etching. Direct Patterning of CdS, PbS and CaWO4 on papers by Ink-Jet Reaction method.

Thursday, January 30, 2020
11:40 AM

(ICACC-S10-033-2020) 2040 Vision Study: NASA's TTT Implementation activities (Invited)

S. Arnold*, NASA Glenn Research Center, Materials and Structures Division, USA

Over the last few decades, advances in high-performance computing, new materials characterization methods, and, more recently, an emphasis on integrated computational materials engineering (ICME) and additive manufacturing have been a catalyst for multiscale modeling and simulation-based design of materials and structures in the aerospace industry. As a result, NASA's Transformational Tools and Technology (TTT) Project sponsored a study to define the potential 25-year future state required for integrated multiscale modeling of materials and systems to accelerate the pace and reduce the expense of innovation in future aerospace and aeronautical systems. This talk will briefly review the findings of this 2040 Vision study and discuss NASA's TTT implementation activities; with special emphasis on composite applications. The study, NASA CR 2018- 219771, envisions the development of a cyber-physical-social ecosystem comprised of experimentally verified and validated computational models, tools, and techniques, along with the associated digital tapestry, that impacts the entire supply chain to enable cost-effective, rapid, and revolutionary design of fit-for-purpose materials, components, and systems. Although the vision focused on aeronautics and space applications, it is believed that other engineering communities can benefit as well from the proposed framework with only minor modifications.

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*** Denotes Presenter**

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