The American Ceramic Society

47th International Conference & Exposition on Advanced Ceramics and Composites

ABSTRACT BOOK

January 22–27, 2023
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 23, 2023

Plenary Session

Room: Coquina Salon D (North Tower)
Session Chairs: Palani Balaya, National University of Singapore; Young-Wook Kim, University of Seoul

8:50 AM

(ICACC-PLEN-001-2023) Testing and Design of Ceramic Structural Materials and Components at NASA

J. Salem*[1]

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

Ceramic and glass components are used in manned and unmanned NASA missions. Hardware examples include specialty windows, mirrors, spectrometer components, laser oscillators and amplifiers, and bearing balls. Requirements for missions can include resistance to thermal shock, impact, mechanical loads and chemical attack, while maintaining functions such as excellent optical transmission. Requirements, failure mechanisms, scale effects, design approaches, and test methods will be discussed along with examples of testing difficulties and design challenges.

9:30 AM

(ICACC-PLEN-002-2023) Additive manufacturing of ceramics from liquid feedstocks

P. Colombo*[1]

1. University of Padova, Industrial Engineering, Italy

Additive manufacturing of polymeric materials has reached a far greater maturity with respect to ceramics, the latter being somewhat limited by their high melting temperatures and the processing issues related to handling of feedstocks containing a large volume of particles. Processing slurry-based feedstocks, in fact, poses several challenges: a high amount of powder is required to promote densification and results in high viscosity, scattering and sedimentation phenomena in vat photopolymerization processes, as well as clogging problems at the nozzle for extrusion-based processes. Some of these issues can be solved or mitigated when using all liquid feedstocks. Our research activities have therefore focused on additive manufacturing of ceramics from liquid feedstocks. In particular, we investigated the use of preceramic polymers as well as geopolymers and sol-gel solutions. Despite the many advantages related to their liquid nature, there are also some challenges related to the reactivity of sol-gel systems and to the high amount of solvent usually present. Here, our strategies for producing high quality ceramic components using a variety of liquid feedstocks and different additive manufacturing techniques, from direct ink writing, digital light processing and two photon polymerization to robotic and volumetric additive manufacturing will be presented.

10:40 AM

(ICACC-PLEN-003-2023) Westinghouse fuel innovation leveraging advanced ceramics

R. Baranwal*[1]

1. Westinghouse Electric Company, USA

Energy is central to nearly every major challenge and opportunity the world faces today. Over the next 20 years, the world population is expected to grow 25% and, by 2030, demand for electricity will nearly double. To meet the world population's growing energy demands, Westinghouse is investing in advanced materials and reactors technologies that provide a carbon-free power grid that's always on – here on earth and beyond. Westinghouse has established key reactor and fuel initiatives to support a clean energy future, many of which rely upon advanced ceramic materials. From nuclear fuel to moderators and instrumentation components, many of the technologies within our fuel innovation portfolio are ceramic-based materials. The Westinghouse Encore Accident Tolerant Fuel ProgramTM is developing fuel technologies that provide enhanced safety margins in the event of a severe accident compared to conventional fuels. The program is comprised of both cladding and nuclear fuel pellet technologies. ADOPTTM fuel is a doped large grained UO2 based ceramic pellet, capable of delivering increased economic benefit and safety margin to customers through its higher density and reduced fission gas release. The ATF program includes a revolutionary cladding design using a Siam SiC-SiC composite that provides improved safety margins beyond design basis accidents. This technology has been developed through collaboration with General Atomics.

11:20 AM

(ICACC-PLEN-004-2023) New Challenges for Ceramic Additive Manufacturing

H. Yun*[1]

1. Korea Institute of Materials Science, Republic of Korea

Additive manufacturing (AM) is a fabrication process that uses 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 significant attention in various fields such as medicine, automotive, aerospace, electronics, and other industrial applications. However, the market for ceramic AM is still significantly smaller compared with metals or polymers because there are still technological barriers that limit its adoption. Despite this, ceramic AM is expected to see dramatic market growth, with an expected market value of $4.8 billion by 2030 (SmarTech Analysis) because ceramic AM may eliminate many of the disadvantages associated with traditional ceramic forming techniques. Since 2006, our research group has developed two unique technologies: a multi-ceramic AM technology based on stereolithography including a unique AM system and ceramic AM process without sintering, based on material extrusion. Furthermore, our group has tried to identify the need for ceramic AM based on our new technologies through collaboration with various industrial companies. We believe that these new technologies may provide big turning point not only to overcome limitation of traditional ceramic form process but alsoYu to enlarge the market for ceramic AM.

Special Focused Session on Diversity, Entrepreneurship, and Commercialization

Jubilee Golden Diversity Awards; Entrepreneurship, and Commercialization

Room: Coquina Salon H (North Tower)
Session Chair: Surojit Gupta, University of North Dakota

1:40 PM

(ICACC-DIV-001-2023) Design and Manufacturing of Smart Materials and Structures (Invited)

M. Kim*[1]

1. Sungkyunkwan University, Republic of Korea

Metamaterials are artificially designed structures that can exhibit unconventional properties such as negative mass density or negative bulk modulus. Such unique properties not easily seen in nature can offer a great platform to control optical, acoustic or elastic wave properties and thus broaden its potential applications including robots, sensors, and energy harvestings. In this talk, I will summarize a collection of design advances that can provide a great platform for enhancing voltage and power performance in acoustic and vibration energy harvesting systems. When integrated with piezoelectric materials, one of the representative "Smart" functional materials, these

"Smart" structures (i.e., metamaterials) can lead to a significantly amplified power output. Along with innovative designs, advanced manufacturing techniques such as additive manufacturing are essentials to realize metamaterials. I will share our recent advances in additive manufacturing of piezoelectric fibers and mechanical metamaterials for self-powered sensing applications.

2:20 PM

(ICACC-DIV-002-2023) Understanding Failure Mechanisms in Advanced Ceramic Materials at Elevated Temperatures: From Nuclear Graphite, TRISO to Ceramic-Matrix Composites (Invited)

D. Liu*[1]

1. University of Bristol, United Kingdom

Ceramic materials are a fascinating yet critical class of materials that have multiple length-scale structures with characteristic dimensions spanning from the nano to macro-scale. Nuclear graphite, which is used as the moderator and structural component in operating reactor and future designs, have been found to exhibit an increased strength and toughness at 1000-1100°C compared to room temperature. Using a range of real-time imaging, diffraction and spectroscopy-based techniques, this behaviour was correlated to the relaxation of residual stresses. Related materials, such as low- and high-density Pyrolytic Carbon (PyC), were used to coat spherical fuel kernels in TRISO (tri-structural isotropic) particle. These PyC layers, together with the SiC layer in TRISO, were found to possess notable magnitude of residual stresses (measured by focussed-ion-beam digital-image-correlation method) and impact the particles' high temperature behaviour as well as irradiation-induced dimensional changes. Lastly, ceramic-matrix composites (aerospace components and accident tolerant fuel cladding) have demonstrated very different toughening mechanisms from ambient when fractured at 1100-1250°C. Implications of these observations on the materials design optimisation will be discussed.

3:00 PM

(ICACC-DIV-003-2023) Materials Discovery for Lunar Dust Tolerant Applications (Invited)

V. L. Wiesner*[1]

1. NASA Langley Research Center, Advanced Materials and Processing Branch, USA

Materials capable of withstanding the harsh lunar environment are needed to support long duration, sustainable missions on the Moon's surface. Without an atmosphere, the lunar surface is exposed to the vacuum of space, large temperature fluctuations, and intense radiation, among other factors. Notably, lunar dust significantly threatens durability and reusability of components and vehicles due to its fine, jagged morphology and highly abrasive nature. These characteristics result in particles eroding, adhering, and/or embedding onto component surfaces and into device-confined geometries (e.g., gear housing, interlocking systems, etc.) potentially causing premature failure. A key barrier hindering materials discovery for lunar dust tolerant applications is a lack of standard lab-based methods to evaluate material properties and performance under representative conditions. To select materials resistant to wear by lunar dust, research activities in support of identifying, developing, and characterizing material candidates, such as ceramics, including laser ablation patterning and additive manufacturing, will be presented. Results from testing and evaluation methods devised to mimic aspects of lunar dust degradation, along with development of a custom-built extreme environment wear testbed that simultaneously subjects materials to wear and aspects of the lunar environment will be shared.

12th Global Young Investigator Forum

Global Young Investigator Forum

Room: Coquina Salon C (North Tower)
Session Chairs: Theresa Davey, Tohoku University; Kaline Furlan, Hamburg University of Technology

1:30 PM

(ICACC-GYIF-001-2023) MAX Phases in Extreme Environments (Invited)

C. Wang*[1]

1. Peking University, School of Physics, China

$M_{n+1}AX_n$ phases exhibit unique laminated atomic structures that result in properties typical of both ceramics and metals. Due to their unusual characteristics, these materials have been proposed for use in a wide variety of industrial applications, including in nuclear systems where they will be exposed to severe irradiation conditions, high temperatures, and high pressure. In this talk, I will summarize recent experimental and theoretical work on the behaviors of the $M_{n+1}AX_n$ phases in these extreme environments. I will present the direct observation of irradiation-induced antisite defects in $M_{n+1}AX_n$ phases and chemical disordering at the atomic scale using high-resolution (HR) aberration-corrected STEM HAADF and ABF imaging. An order-to-disorder, hex-to-γ-to-fcc phase transformation and the formation of metastable solid solution phases were observed using synchrotron grazing incidence X-ray diffraction (GIXRD), atom probe tomography (APT), and first-principle calculations. The role of elemental composition and chemical complexity at cation sites in the radiation tolerance of $M_{n+1}AX_n$ phases will be discussed. This talk will provide a comprehensive understanding of the mechanisms of structural modification and defect evolution in $M_{n+1}AX_n$ phases, as well as proposed strategies for designing novel $M_{n+1}AX_n$ phases with enhanced performance under extreme irradiation conditions.

2:00 PM

(ICACC-GYIF-002-2023) Reactive molecular dynamics simulations clarifying the effect of carbon nanotube orientations on mechanical properties of SiC/CNT composites (Invited)

Y. Su*[1]; Y. Asano[1]; Q. Chen[1]; Y. Ootani[1]; N. Ozawa[2]; M. Kubo[1]

1. Tohoku University, Institute for Materials Research, Japan
2. Tohoku University, New Industry Creation Hatchery Center, Japan

SiC is one of the vital advanced ceramics because of its excellent mechanical and electrical properties and is applied in a wide range of industries: from semiconductors to astronomy. However, the brittleness (low fracture toughness) of SiC inhibits its practical use. Carbon nanotube (CNT) is a promising reinforcing fiber for SiC matrix to create a composite material with higher fracture toughness. The reinforcing mechanisms involve various spatiotemporal scale phenomena; microscopic chemical reactions to mesoscale SiC/CNT deformations. Thus, the current issue is to clarify the CNT reinforcing mechanisms by solving the multiscale-multiphysics problem. Despite the orientation of CNT in the SiC matrix being considered the dominant factor of the reinforcing, it is challenging to reveal the detailed mechanisms thus far. A molecular dynamics (MD) simulation helps us to clarify the atomic-scale mechanisms including chemical reactions. In the presented study, the effect of CNT orientations on the mechanical properties of SiC/CNT composites is studied by MD simulations using the in-house reactive MD simulator Laskyo. The results show that the CNT orientation affects the composites' mechanical properties through two competitive mechanisms: loading bearing and crack bridging. The details of the results will be presented at the conference.

2:30 PM

(ICACC-GYIF-003-2023) Surface grafted radical polymerization on spherical clusters of iron oxide nanoparticles (Invited)

L. Klauke*[1]; M. Kampferbeck[1]; C. von Bredow[1]; A. Meyer[1]; T. Vossmeyer[1]

1. University of Hamburg, Physical Chemistry, Germany

Magnetic nanoparticles are interesting materials for various applications, such as information storage, catalysis, and biomedicine. Especially for the latter, superparamagnetic iron oxide nanoparticles (SPIONs) are of interest. However, the response of single SPIONs to external magnetic fields is rather slow. Contrary, a fast response is necessary for the use as magnetic carriers. To overcome this problem, SPIONs can be assembled into superparticles. Herein, we present the synthesis of shape controlled SPIONs via thermolysis. These nanoparticles are assembled into clusters via emulsions. Subsequent encapsulation in a surface grafted, controlled radical polymerization yields the desired composites with sizes of approximately 200-300 nm. To enable the application as magnetic carriers, functional groups (e.g. -COOH, -NH_2) can be introduced into the polymer shell. The incorporation of such monomers is verified by infrared spectroscopy and zeta potential measurements. Electron microscopy and thermogravimetric analysis of the composites confirmed precisely adjustable polymer shell thicknesses. Magnetic measurements showed magnetizations of 10-20 emu/g of the composite material depending on the organic content and the SPION size. The presented clusters are covalently linked to the polymer and are highly promising materials for magnetic carriers due to their fast response to magnetic fields.

3:20 PM

(ICACC-GYIF-004-2023) Reactive spark plasma sintering of the high-entropy diboride $(Hf_{0.2}Nb_{0.2}Ta_{0.2}Ti_{0.2}Zr_{0.2})B_2$ (Invited)

K. Acord*[1]; D. Miracle[1]; L. M. Rueschhoff[1]

1. Air Force Research Lab, USA

The high hardness and promising oxidation resistance of the high-entropy diboride (HEBs), $(Hf_{0.2}Nb_{0.2}Ta_{0.2}Ti_{0.2}Zr_{0.2})B_2$, are of interest for high temperature aerospace applications, however materials processing challenges, including safety and phase purity issues, persist. For instance, synthesis of $(Hf_{0.2}Nb_{0.2}Ta_{0.2}Ti_{0.2}Zr_{0.2})B_2$ from starting metal powders or commercially available diboride powders results in challenges associated with handling air-sensitive, pyrophoric materials. In this study, we overcome these challenges by preparing $(Hf_{0.2}Nb_{0.2}Ta_{0.2}Ti_{0.2}Zr_{0.2})B_2$ samples from oxide-based starting powders using borocarbothermal reduction during reactive spark plasma sintering (R-SPS). Excess carbon is often used to enhance phase purity during borocarbothermal reduction. We demonstrate that carbon content influences microstructural features, secondary phase formation, and mechanical properties using scanning electron microscopy, energy dispersive X-ray spectroscopy/X-ray diffraction, and Vickers hardness testing, respectively. This study unveils the feasibility and challenges of forming bulk high-entropy $(Hf_{0.2}Nb_{0.2}Ta_{0.2}Ti_{0.2}Zr_{0.2})B_2$ samples with high phase purity from oxide powders using R-SPS.

3:50 PM

(ICACC-GYIF-005-2023) Conical Nozzle Levitator Equipped with Dual Lasers for High Temperature Testing (Invited)

F. Thorpe*[1]; S. J. McCormack[2]

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

A levitation device equipped with environmental controls and oxygen mitigation has been used to analyze molten samples using laser heating. This device uses two lasers to enable heating of various high temperature materials: 400W CO2 laser (10.6μm wavelength) and 500W Ytterbium Fiber laser (1.07μm wavelength). The system uses a vacuum pump, gas scrubber, and ultra-high pure Argon to mitigate reactions. The temperature is determined using two single-color laser pyrometers at 0.9μm (750–3500°C) and 0.65μm (750 – 5000°C). This system was used to determine the melting point of ~3mm diameter spherical samples of various refractory metals, group IV/V oxides, and group IV/V diborides. The melting point was determined by analyzing the cooling curve created from heating the samples above the melting point and observing the recalescence as the sample solidifies. Corrections were made for sample emissivity to ensure temperature accuracy. This system can be used in conjunction with in-situ x-ray diffraction to measure the thermal expansion on the atomic level.

4:20 PM

(ICACC-GYIF-006-2023) Microstructural Engineering of Alumina via Magnetic Powder Processing (Invited)

B. Conry*[1]; J. B. Harley[2]; M. R. Tonks[3]; M. S. Kesler[4]; A. Krause[1]

1. Carnegie Mellon University, Materials Science and Engineering, USA
2. University of Florida, Electrical and Computer Engineering, USA
3. University of Florida, Materials Science and Engineering, USA
4. Oak Ridge National Lab, USA

The microstructure of a material dictates its macroscopic properties, and as such, the ability to understand and control microstructural evolution is a crucial component of material processing and design. However, controlling microstructure evolution remains an ongoing challenge because the relationship between grain growth and grain boundary character remains poorly understood. Grain boundary engineering has emerged in recent years as an exciting method by which grain boundary structure and composition can be tailored to control grain growth. In this work, we slip cast Ca-doped alumina, a highly anisotropic material, in a high strength magnetic field to engineer a characteristic grain boundary network structure. As compared to an untextured control specimen, the unique textured microstructural design suppresses abnormal grain growth, demonstrating the potential of this method for engineering applied materials with homogenous, controlled microstructures with tunable grain shapes and orientations. The underlying mechanisms driving this phenomenon will be discussed, including the relationship between grain boundary character and grain growth.

4:50 PM

(ICACC-GYIF-007-2023) Preparation of silicon nitride ceramics with high mechanical strength and thermal conductivity

Y. Nakashima*[1]; Y. Zhou[1]; K. Hirao[1]; M. Fukushima[1]

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

Mechanical strength and thermal conductivity are important factors for the application of silicon nitride (Si_3N_4) as a ceramic insulating substrate. However, those properties generally have a trade-off relationship, and it has been difficult to simultaneously achieve both properties at a high level. In this study, for achieving the both good properties, we prepared the Si_3N_4 ceramics by a sintered reaction-bonded silicon nitride (SRBSN) method, and the effects of the nitridation temperature on their microstructure, bending strength, fracture toughness and thermal conductivity were investigated. Their microstructures had no clear difference, while the ratio of β phase in the nitrided compacts significantly increased with increasing nitridation temperature. The bending strength maintained a high value of 800 MPa regardless of the nitridation temperature. The fracture toughness was almost constant regardless of the temperature. On the other hand, the thermal conductivity clearly improved at the elevated nitridation temperatures. This increase would be related with decrease of a lattice oxygen content in Si_3N_4 because the oxygen content generally decreased with increased ratio of β phase. Therefore, the Si_3N_4 ceramics prepared at the higher nitridation temperature had the high strength and high thermal conductivity, which are generally known to be in a trade-off relationship.

5:10 PM

(ICACC-GYIF-008-2023) Processing of Preceramic Polymer-Based Ceramic Composites (Invited)

M. O'Masta*[1]; E. Stonkevitch[1]; E. Wernick[1]; P. Bui[1]; T. Schaedler[1]

1. HRL Laboratories, USA

Ceramic matrix composites (CMCs) often comprise a silicon-based, ceramic matrix derived from a polymer. Understanding defect formation during polymer-to-ceramic conversion is critical for obtaining the desired properties, shape fidelity and part quality. This talk focuses on the additive manufacturing (AM) of reinforced polymer-derived Si(O)C ceramic matrix. We pair numerical analysis with experimental studies, including in-situ X-ray computed tomography (XCT) of the pyrolysis event, to unveil the necessary conditions for converting the polymer to ceramic without the formation of deleterious cracks and voids. We test the influence of processing conditions on mechanical properties, using printed ceramics reinforced with particles and whiskers. Furthermore, the presentation will demonstrate the applicability of ceramic printing for microelectronics applications.

FS1: Bioinspiration, Green Processing, and Related Technologies of Advanced Materials

Bioinspiration, Green Processing, and Related Technologies of Advanced Materials I

Room: Coquina Salon B (North Tower)
Session Chair: Thomas Speck, University of Freiburg

1:30 PM

(ICACC-FS1-001-2023) The Glassomer Technology – a nanocomposite approach to high-resolution glass structuring (Invited)

B. E. Rapp*[1]

1. Freiburg University, Department of Microsystems Technology, Germany

3D printing is the manufacturing revolution of the 21st century. The invention of printing by Johannes Gutenberg over 500 years ago, i.e., the ability to generate, replicate and disseminate artifacts has changed human history significantly. Recent decades have seen printing moving from two-dimensional to three-dimensional. With the development of the Glassomer® Process we have introduced a significant step towards making glass a commodity material accessible via scalable industrial manufacturing technology. This process is based on nanocomposite which can cured to either crosslinked thermosets or non-crosslinked thermoplasts giving the component a precisely controllable three-dimensional shape. Once shaped, the material is debound and sintered to optical-grade fused silica glass. Over the course of the last 5 years we disclosed in contributions in Nature, Nature Materials, Nature Communications, Advanced Materials and Science that this process scales from replication casting, over high-resolution 3D Printing to, more recently, industrial-scale injection molding. The Glassomer® Process closes a significant gap in the material palette of modern manufacturing processes enabling, for the very first time, the free-form generation of highly transparent fused silica glass by a state-of-the-art 3D polymer replication technology.

2:00 PM

(ICACC-FS1-002-2023) High resolution 3D printing of biocomposites for bioinspired materials

D. Böcherer*[1]; Y. Li[1]; B. E. Rapp[1]; D. Helmer[1]

1. University of Freiburg, Department of Microsystems Engineering, Germany

To reduce waste of resources and minimize environmental pollution, the development of sustainable materials is a main task of today's research. Biocomposite materials represent an eco-friendly alternative to conventional petroleum-based materials and show promising material properties such as good mechanical properties, renewability, and biodegradability. However, most of the biocomposites combine a bio-based filler with conventional matrix materials resulting in a low renewability. Here, we report on the combination of a wood filler with photocurable acrylated epoxidized soybean oil (AESO) and isobornyl acrylate (IBOA) resulting in a fully biobased material with a biorenewable carbon content of > 80%. Due to its great photocurable properties and well-tuned viscosity, complex 3D objects of the biobased materials are structured using 3D digital light processing (DLP) printing. Furthermore, two-photon polymerization (2PP) is used to print pure AESO photoresin with a resolution of a few micrometers demonstrating a promising method for additive manufacturing of fully bio-based and bioinspired structures on the microscale.

2:20 PM

(ICACC-FS1-003-2023) Liquid crystalline elastomer photoresists for microrobot 3D printing

B. Rashid Hanif[1]; S. Nocentini[2]; D. Martella[2]; C. Parmeggiani[2]; R. Taboryski[1]; D. Wiersma[2]; A. Bunea*[1]

1. Technical University of Denmark, DTU Nanolab, Denmark
2. European Laboratory for Non-linear Spectroscopy, Italy

Microrobots are extremely suitable for various microscale tasks because of their size. Challenges in microrobotics relate to actuation, and to incoroporating functionality within such tiny objects' bodies. we make use of light as a highly precise and versatile actuator for microrobots. Here, we present a novel liquid crystalline elastomer (LCE) photoresist formulation for micro 3D printing by two-photon polymerization. This fabrication technique was selected because it is enables sub-micrometer resolution and 3D shape freedom. The LCE photoresist contains: 1) a mesogen, which gives it liquid crystal properties, 2) a crosslinker, which forms an elastomer network upon polymerization, 3) a dye, which enables shape morphing of the material in response to green light, 4) a photoinitiator, which ensures a localized polymerization reaction during the micro 3D printing process, and 5) a liquid crystal solvent, commonly known as 5CB. Unlike previous LCE photoresist formulations, which are solid at room temperature, this formulation is liquid at room temperature, which helps streamline the fabrication process Using the novel LCE photoresist, we fabricated simple microrobotic blocks that can be actuated using a green laser in fully reversible contraction / expansion cycles. This should facilitate the fabrication of smart microrobotic components and ultimately enable the development of microrobots with advanced functions.

2:40 PM

(ICACC-FS1-004-2023) Development of a cellular actuator inspired by the motor cells of grass leaves

O. Speck*[1]; A. Mader[2]; M. Langer[3]; J. Knippers[2]

1. University of Freiburg, Cluster of Excellence livMatS, Germany
2. University of Stuittgart, Institute of Building Structures and Structural Design (ITKE), Germany
3. University of Freiburg, Plant Biomechanics Group, Botanic Garden, Germany

Within a biomimetic approach, biologists and engineers developed a pneumatic cellular actuator inspired by the turgor-dependent kinetic motion amplification of grass leaves. The leaf halves fold, when the turgor pressure in the motor cells is low and unfold, when the motor cells become fully turgescent. We incorporated all insights on the leaf movement and the underlying functional principle into a finite element analysis, as a prerequisite for the development of a pneumatic cellular actuator. The first prototype consisted of a row of single cells with compliant hinges positioned on a plate. When increasing the pneumatic pressure applied to each individual cell, the cells become wider at the upper side and the entire structure bends. The actuator was successfully integrated in the facade shading system Flectofold, where the bending of its midrib controls the hoisting of its wings.

Bioinspiration, Green Processing, and Related Technologies of Advanced Materials II

Room: Coquina Salon B (North Tower)
Session Chair: Ada-Ioana Bunea, Technical University of Denmark

3:20 PM

(ICACC-FS1-005-2023) Multi-material artificial Venus flytrap demonstrator combining principles of two snap-trap motions in plants (Invited)

F. J. Tauber*[1]; P. Auth[1]; J. Teichmann[1]; T. Speck[2]

1. University of Freiburg, EXC livMatS / Plant Biomechanics Group Freiburg / Botanical Garden Freiburg, Germany
2. University of Freiburg, Germany

Biological material systems are environmentally adapted systems and developed over 3.8 billion years of evolution in diverse living organisms. These systems serve as concept generators for bioinspired material systems translating functions of living nature into engineering applications, enabling novel functions such as embodied intelligence and embodied energy. Current artificial Venus flytraps are inspired by carnivorous snap-trap plants and enable fast, hingeless movements in plant-inspired robotic systems. We present a novel bioinspired “artificial Venus flytrap” incorporating snap-trap motion principles of two carnivorous plants in one system, exhibiting adaptive responses to various environmental triggers. The presented systems are examples of a successful implementation of several principles based on plant motion and deformation in a versatile, adaptive, and technically compliant multi-material system. The systems and novel motion sequences are characterized in terms of kinematics, energy requirements and overall performance. Our project will use bioinspired technical devices such as an “artificial Venus flytrap” to demonstrate the feasibility of the developed material systems with dynamic, life-like, and non-equilibrium properties. Representing a first step to the future implementation of novel technologies in industrial products and everyday life applications.

3:50 PM

(ICACC-FS1-006-2023) 3D-printed bioinspired devices for micro-filtering and online analysis of washing suds in greener washing machines

T. Speck*[1]; U. Schaumann[2]; T. Kampowski[1]; K. Ulrich[1]; M. Thielen[1]; G. Bold[1]; M. Langer[1]; T. Masselter[1]

1. University of Freiburg, Germany
2. E.G.O. Elektro-Gerätebau Oberderdingen GmbH, Germany

Based on existing biological knowledge of filter feeding in basket sharks and the olfactoric system of hammerhead sharks we developed a microfiber- and microplastic-filter system and a flow channel module suitable for the implementation of diverse state-of-the-art sensor technologies for online analysing of washing suds during the processes in washing machines. Up-to-date simulation methods for the in silico optimization of the bioinspired devices proved to be complicated due to complex microfluidic patterns, Therefore, a considerable part of the optimization of the filtering and flow channel modules took place with real physical 3D-printed models. Following a biomimetic approach, rapid prototyping methods were combined with high-speed videography and 2D particle tracking for characterizing the functionality of the filtering and flow-channel devices. Most of today’s washing machines represent “black boxes” which lack dynamic analysis and filter techniques for an automated and optimized resource management. Therefore, the development of the online control of washing suds and the enhancement of processes for filtration is one solution for stopping the increasing pollution of freshwater and oceans by microplastic and microfibers. Washing machines are among the major contributors to micro-X pollution, but also hold a high technical potential for solving this problem.

4:10 PM

(ICACC-FS1-007-2023) Nanoarchitected Biological Ceramic Composites: Mitigation of Catastrophic Damage (Invited)

D. Kisailus*[1]

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

Over hundreds of millions of years, natural systems have developed well-orchestrated strategies, exemplified in the biological tissues of numerous animal and plant species, to synthesize and construct materials from a limited selection of available starting materials. The resulting structures display multiscale architectures with incredible fidelity and often exhibit properties that are similar, and frequently superior to, mechanical properties exhibited by many engineering materials. In specific instances, comparative analyses of multiscale structures have pinpointed which design principles have arisen convergently; when more than one evolutionary path arrives at the same solution, we have a good indication that it is the best solution. In this work, we describe 3 specific design features: Nanorods, Helicoids, and Nanoparticles. We not only highlight their function in specific organisms, but also describe how interface controlled syntheses and hierarchical assembly using organic (i.e., often chitinous) scaffolds lead to these integrated macroscale structures. Beyond this, we describe their translation to biomimetic materials used for engineering applications, potentially via a process to 3D print these scaffolds, enabling them to provide a scalable template for materials growth.

FS2: Materials for Thermoelectric and Thermionic Energy Conversion

Bulk Thermoelectric Materials I

Room: Flagler A (South Tower)
Session Chair: Michitaka Ohtaki, Kyushu University

1:30 PM

(ICACC-FS2-001-2023) Engineering of Materials Parameters for High-Performance Thermoelectric Materials (Invited)

K. Lee*[1]

1. Yonsei University, Republic of Korea

Thermoelectrics, which can generate electricity from a temperature difference, or vice versa, is a key technology for solid-state cooling and energy harvesting; however, its applications are constrained owing to low efficiency. Since the conversion efficiency of thermoelectric devices is directly obtained via a figure of merit of materials, zT, which is related to the electronic and thermal transport characteristics, the aim here is to elucidate physical parameters that should be considered to understand transport phenomena in thermoelectric semiconducting materials.

2:00 PM

(ICACC-FS2-002-2023) Approach to Determine the Density-of- States Effective Mass with Carrier Concentration-Dependent Seebeck Coefficient (Invited)

K. Lee[2]; S. Kim[1]; J. Lim[3]; J. Cho[4]; H. Yang[3]; H. Kim*[1]

1. University of Seoul, Republic of Korea
2. Yonsei University, Republic of Korea
3. Hongik University, Republic of Korea
4. Korea Institute of Ceramic Engineering and Technology (KICET), Republic of Korea

Band engineering is an effective strategy to improve the electronic transport properties of semiconductors. In thermoelectric materials research, density-of-states effective mass is an undoubted key factor in verifying the band engineering effect and establishing a strategy for enhancing thermoelectric performance. However, estimation of the effective mass is demanding or inaccurate depending on the methods taken. A simple equation is proposed, valid for all degeneracy: $\mathrm{Log}_{10}(m_d^*T/300) = (2/3)\,\mathrm{Log}_{10}(n) - (2/3)\,[20.3 - (0.00508 \times |S|) + (1.58 \times 0.967^{|S|})]$ that utilizes experimentally determined Seebeck coefficient (S) and carrier concentration (n) to determine the effective mass (m_d^*) at a temperature (T). This straightforward equation, which gives an accurate analysis of the band modulation in terms of m_d^*, is indispensable in designing thermoelectric materials of maximized performance.

Bulk Thermoelectric Materials II

Room: Flagler A (South Tower)
Session Chair: Kyu Hyoung Lee, Yonsei University

3:20 PM

(ICACC-FS2-003-2023) Defect chemistry and functionalisation of the conductive network in ternary and quaternary Cu-based sulfides (Invited)

E. Guilmeau*[1]

1. CNRS CRISMAT, France

Research focusing on the interplay between structural features and transport properties of inorganic materials is of paramount importance for the identification, comprehension, and optimisation of functional materials. In this respect, ternary and quaternary copper sulfides have been receiving considerable attention from scientists to discover and optimise their thermoelectric efficiency. The presence of structural defects, non-stoichiometry, the nature of the chemical bonds, order/disorder phenomena in these complex structures are still a matter of debate, which is of capital importance for the optimization of their TE properties. Our investigations on some thermoelectric copper-based sulphides derived from natural minerals (colusite, stannite, enargite, mohite-derivatives...) will be presented. Processing, as well as structural and microstructural features, will be reported, together with electrical, thermal properties. Band structure and vibrational dispersions from first principles calculations will be discussed.

3:50 PM

(ICACC-FS2-004-2023) High-temperature Thermoelectric Properties of Tungsten-based Magnéli Phase Oxide $(W_{1-x}Ti_x)_{18}O_{49}$

M. Ohtaki*[1]; N. Q. Tran[1]; K. Suekuni[1]

1. Kyushu University, Interdisciplinary Graduate School of Engineering Sciences, Japan

High-temperature thermoelectric properties of tungsten-based Magnéli phase oxide $(W_{1-x}Ti_x)_{18}O_{49}$ ($0 \leq x \leq 0.25$) prepared by solid state reaction followed by densification via spark plasma sintering (SPS) were studied. The Ti substitution increased the Seebeck coefficient, the power factor, and decreased both the electronic and lattice thermal conductivity. The synergistic substitution effect on the electrical and thermal properties and inherently low total thermal conductivity of 1.46 ± 0.08 $WK^{-1}m^{-1}$ originating from the tunnel-like crystal structure of the oxide led to a significantly high ZT of 0.50 ± 0.07 at 1073 K for the sample with $x = 0.2$.

FS3: Nanostructures and Low-Dimensional Materials for Chemical Sensors

Nanostructures and Low-Dimensional Materials for Chemical Sensors

Room: Coquina Salon D (North Tower)
Session Chairs: Ho Won Jang, Seoul National University; Koichi Suematsu, Kyushu University; Hyung Gi Byun, Kangwon National University; Kengo Shimanoe, Kyushu University

1:30 PM

(ICACC-FS3-001-2023) A way to standardize odor metadata from Electronic Nose for Olfactory-enhanced multimedia

H. Byun*[1]

1. Kangwon National University, Electronics, Information & Communication Eng., Republic of Korea

The studies regarding olfaction-enhanced multimedia would be divided into two parts in terms of encoding and decoding of odor (scent) metadata (odor label, strength, concentration, harmfulness, etc.). Most of the olfaction metadata coded from Eectronic Nose is transferred to the olfactory display and then the olfactory display generates odor corresponding to the odor recipe. Although many studies have been conducted for harmonizing olfaction with multimedia using the E-Nose and olfactory display, their practical applications have been severely restricted due to their device diversity, non-normative data templates used, and absence of generalized design for application of the devices. The solution coping with above problems is to choose a suitable standard, universally accepted. ISO/IEC 23005, a representative standard related to immersive media, provides normative data templates and commands for the sensory effects of immersive multimedia. In particular, this standard specifies information representation which can be used in controlling olfaction-related sensors (E-Nose) and the actuators (olfactory display). This paper proposes a way of standardize odor metadata obtained from electronic nose system, which has been particularly used to acquire data from different coffee aromas, to transfer for odor display applicable olfactory-enhanced multimedia using ISO/IEC 23005 data template.

1:50 PM

(ICACC-FS3-002-2023) Development of micro gas sensors using new solid electrolyte for detection of oxygen and acidic gases

K. Shimanoe*[1]; S. Ide[2]; K. Watanabe[1]; K. Suematsu[1]

1. Kyushu University, Faculty of Engineering Sciences, Japan
2. MITSUI MINING & SMELTING CO., LTD., R&D Center, Japan

Recently, miniaturization of solid-state gas sensors is investigated and some of these are going to be put to practical use. The typical examples are combustion-type and semiconductor-type gas sensors because they are easy to match a CMOS process. They are called "MEMS-type". On the other hand, it has been thought that it was difficult to miniaturize gas sensors using solid electrolyte. Because there are not many solid electrolytes operative at temperature less than 500°C and suitable for CMOS process. To overcome these problems, we are developing new solid electrolyte, highly c-axis-oriented apatite-type lanthanum borosilicate (c-LSBO), as an oxide-ion conductor. c-LSBO has high oxide-ion conductivity (0.016 S/cm at 400°C) and can be applied for thin film on silicon wafer. By using c-LSBO, we reported some functional devices such as oxygen pumping cell, oxygen, NO_2, CO_2 and CO sensors and so on. In addition, we are trying to miniaturize gas sensors by using the thin film.

2:10 PM

(ICACC-FS3-003-2023) Design of MEMS-type gas sensor using metal oxides semiconductor for super selectivity

K. Shimanoe*[1]; K. Suematsu[1]; K. Watanabe[1]

1. Kyushu University, Japan

In this study, we investigated the combination of receptor function (chemical sensitization) and pulse-heating of MEMS device for super selectivity. MoO_3 was selected as a receptor for ethanol because of acidic oxide that oxygen adsorption doesn't occur. We evaluated the effect of receptor function by paying attention to adsorption and oxidative reaction path of ethanol on MoO_3-loaded SnO_2. In the case of acidic oxides, it is known that oxidative reaction path of alcohol differs depending on the acidity of metal oxide, that is, the oxidation of ethanol on SnO_2 and MoO_3 proceeds through aldehyde and ethylene, respectively. MEMS-type gas sensors using neat-SnO_2 and MoO_3-loaded SnO_2 were operated by double pulse-heating. In the case of SnO_2, the sensor responses were not so high and the significant difference in between ethanol and methanol was not found. On the other hand, the sensor response of MoO_3-loaded SnO_2 to ethanol was 100 times higher than that to methanol. It is presumed that two effects, ethanol adsorption and ethylene formation on MoO_3 at heating-off and heating-on, respectively, worked synergistically.

2:30 PM

(ICACC-FS3-004-2023) Luminescent gold nanoclusters by pulsed laser ablation in water: Modulation of the photoluminescent response by the interaction with heavy metal ions

T. Janqau[2]; G. Concas[2]; Q. Zaman[3]; N. Daldosso*[1]; F. Enrichi[1]; T. Del Rosso[2]

1. University of Verona, Italy
2. Pontificia Universidade Catolica, Fisica, Brazil
3. University of Buner, Physics, Pakistan

In this experimental study, we investigated porous silicon microparticles (pSi MPs) as a delivery system for drugs and Pam3CSK4, a toll-like receptor (TLR) 1 and 2 agonist used as an immunologic adjuvant for immunotherapy. The well-known properties of porous silicon system, such as photoluminescence, large surface to volume ratio and easily-tunable surface, allow it to be an ideal carrier for theranostic purposes. We first performed some preliminary in vitro tests of loading and release of the different drugs and, and the interaction between loaded pSi and dendritic cells (DCs) was studied by live-cell confocal microscopy. The immunologic response was evaluated by ELISA-assay showing a promising enhancement effect resulting from the synergic interaction between pSi MPs and Pam3CSK4. With the aim of increasing the immunologic response, we successfully synthetized a pH responsive polymer, known to promote MHC I and II presentation, and we did some preliminary encapsulation tests.

2:50 PM

(ICACC-FS3-005-2023) Visible light-activated NO_2 gas sensor based on 2-dimensional SnS_2 nanoflowers

G. Nam*[1]; H. Jang[1]

1. Seoul National University, Republic of Korea

Two-dimensional semiconductor SnS_2 has emerged as one of the most eligible candidates for chemoresistive gas sensor applications due to its excellent gas sensing performance. Herein, we propose the room temperature nitrogen dioxide (NO_2) sensing of SnS_2 nanoflowers (NFs) enabled by visible light activation. SnS_2 NFs were prepared by solvothermal synthesis with abundant edge sites. The high absorbance in the visible light region induced the generation of charge carriers resulting in decreased resistance and enhanced gas sensing characteristics. Even under red light and green light with low photon energy, the room temperature NO_2 sensing performance was enhanced. The highest NO_2 sensing performance was achieved under blue light, including the highest response, excellent selectivity towards NO_2 and an extremely low detection limit. Furthermore, the sensor exhibited reliable gas sensing performance in humid conditions and maintained its properties after long-term relaxation. Taking advantage of surface properties, optical properties and gas sensing properties, a light-activated SnS_2 NFs based gas sensor is expected to develop SnS_2 nanostructures for use in an electronic nose.

3:30 PM

(ICACC-FS3-006-2023) Materials Design for the Thermally Modulated Semiconductor Gas Sensors: Composite of the $Ba_{0.9}La_{0.1}FeO_3$ and SnO_2 for Ethanol Sensing

K. Suematsu*[1]; Y. Hiroyama[1]; K. Watanabe[1]; K. Shimanoe[1]

1. Kyushu University, Japan

We experimentally revealed the amplification of ethanol and O^{2-} adsorption properties of SnO_2 by compositing with BLF, and the effect on the sensor performance under the pulse-driven mode, which is the switching mode of the microheater for sensor driving. We prepared the compositing materials of $Ba_{0.9}La_{0.1}FeO_3$ (BLF) and SnO_2 because BLF plays the role of oxygen and ethanol supplier onto the SnO_2 surface. The oxygen was probably supplied from BLF to SnO_2 less than 250°C, and transferred to the $O^{2-}{}_{ad}$ on the SnO_2 surface. Moreover, ethanol molecules were once preferentially adsorbed on the BLF, and then provided to the SnO_2 surface for ethanol detection in the measurement phase of pulse-driven mode. Therefore, to improve the sensor performance, it is essential to understand the interaction of oxygen and gas molecules on the surface of the particles to obtain the optimal materials design and combine the appropriate material and thermal modulation.

3:50 PM

(ICACC-FS3-007-2023) Chemoresistive Gas Sensing Properties of Tailored Graphene Micropatterns by Wafer-Scale Direct Transfer

S. Kim*[1]; H. Jang[1]

1. Seoul National University, Republic of Korea

Among the alternative materials for gas sensing applications, two-dimensional (2D) materials can offer novel opportunities to realize new types of electronics by providing their unique properties, such as flexibility, transparency, and tunable electrical properties. Graphene is the most representative 2D material and has been studied with great interest. However, embedding graphene into flexible devices remains a challenge due to difficulties in microscale patterning of graphene on flexible substrates, such as a polymer. Herein, a novel strategy to obtain micropatterned graphene on polymer substrates using direct curing process is demonstrated. Employing our method, entirely flexible and transparent, well-defined self-activated graphene sensor arrays, capable of gas discrimination without external heating, are fabricated on four-inch wafer-scale substrates. Finite element method simulations show the potential of this patterning technique to maximise the performance of the sensor devices when the active channels of the 2D material are suspended and nanoscaled. We also successfully discriminated different kinds of gas species with the voltage-dependen sensing data. Our study can contribute considerably to the development of flexible functional electronic devices based on 2D materials.

4:10 PM

(ICACC-FS3-008-2023) Sensitive taste sensors using graphene decorated with metal or nafion for high selectivity to glucose and pH

H. Kim*[1]; C. Lee[1]; H. Jang[1]

1. Seoul National University, Republic of Korea

In recent days, taste sensing have been an important issue in terms of, detecting food quality or sensing various chemical substances from specific materials. This kind of taste sensor mainly uses 2-Dimensional materials such as graphene for sensing rather using lipids or other biological substances. The aim of using 2D materials rather lipids is for longer use; bio-substance based taste sensors have very high selectivity and sensitivity but the life span of the device is short and it is sensitive to external atmosphere. For long term use and daily usage, 2D material based sensors have the advantage to bio-based sensors. Here in, we report graphene based taste sensors graphene decorated with gold for glucose sensing and nafion spin coating on graphene for pH sensing. Graphene is the main source of sensing and the decorated materials help to enhance selectivity by absorbing or penetrating the target material. Gold is well known to have selectivity to glucose and nafion has the ability to selectively penetrate cations. Gold is decorated with e-beam deposition; which thickness is about 2nm. On the other hand, nafion is spin coated onto a graphene transferred substrate.

4:30 PM

(ICACC-FS3-009-2023) Sensitive taste sensors using surface tailored graphene for high selectivity to salt

C. Lee*[1]; H. Kim[1]; H. Jang[1]

1. Seoul National University, Materials Science and Engineering, Republic of Korea

In recent days, taste sensing have been an important issue in terms of, detecting food quality or sensing various chemical substances from specific materials. This kind of taste sensor mainly uses 2-Dimensional materials such as graphene for sensing rather using lipids or other biological substances. The aim of using 2D materials rather lipids is for longer use; bio-substance based taste sensors have very high selectivity and sensitivity but the life span of the device is short and it is sensitive to external atmosphere. For long term use and daily usage, 2D material based sensors have the advantage to bio-based sensors. Here in, we report graphene based taste sensors graphene decorated with calix[4]arene for salt detection and nafion spin coating on graphene to enhance selectivity. Graphene is the main source of sensing and the decorated materials help to enhance selectivity by absorbing or penetrating the target material.

S1: Mechanical Behavior and Performance of Ceramics & Composites

Thermo-mechanical Performance of Ceramic Matrix Composites (CMCs) in Various Environments

Room: Ballroom 5 (South Tower)
Session Chairs: Robert Goldberg, NASA Glenn Research Center; Gerard Vignoles, University Bordeaux; Craig Przybyla, Air Force Research Laboratory

1:30 PM

(ICACC-S1-001-2023) To drill or not to drill? - Creep of an oxide-oxide composite with diamond-drilled effusion holes at elevated temperature (Invited)

M. Harkins[1]; M. Ruggles-Wrenn*[1]

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

Ceramic matrix composites (CMCs) are prime candidates for use in aircraft engines. Even with their high-temperature capabilities, many CMC components will need cooling. Film cooling technique requires rows of small holes within the component surface. Effects of multiple small holes on creep performance of an oxide-oxide CMC consisting of Nextel 720 alumina-mullite fibers in a porous alumina matrix were evaluated. Specimens with 17 diamond-drilled holes of 0.5-mm diameter in the gage section were creep tested at 1200°C in air and in steam. Creep stresses ranged from 40 to 130 MPa. Creep run-out was set to 100 h. Specimens with diamond-drilled holes produced higher creep rates and shorter creep lifetimes than the unnotched composite. An earlier AFIT study found that the presence of rows or laser-drilled holes also considerably lowered the creep resistance of this CMC, albeit via a different microstructural degradation mechanism. Damage to the CMC microstructure caused by these two drilling techniques and implications for mechanical performance are discussed.

2:00 PM

(ICACC-S1-002-2023) Effects of processing parameters and layup techniques on the creep properties of an all Alumina oxide CMCs used for sealing applications at 1100°C (Invited)

T. J. Pirzada*[1]; J. Marrow[1]; M. Galano[1]; L. J. Vandeperre[2]; J. Al-Lami[2]

1. University of Oxford, Materials, United Kingdom
2. Imperial College London, Materials, United Kingdom

The high temperature flexural strength and creep behaviour of Nextel 720/Alumina based composites, fabricated with eight different combinations of two weave patterns and two layups (0°/90° and involute) has been investigated. Beams of 4 x 5 mm cross-section were loaded in flexure to failure at 1100°C with a constant displacement rate of 0.05mm/min. Flexural creep tests were also performed at a constant stress of 25 MPa for 2 hours. The two weaves in involute layup were used to fabricate specimens with a sealing ring geometry (140 mm diameter, 5 x 5 mm cross section), which were subjected to a fixed displacement stress relaxation creep test with an initial nominal flexural stress of 25 MPa for 16 hours at 1100°C. Ex-situ high resolution X-ray tomographs of regions of interest in each beam and ring, obtained before and after the creep tests, were analysed by digital volume correlation to map the internal deformations. In the sealing ring specimens, the matrix cracks introduced during processing were loaded in tension, which resulted in them acting as stress concentrators that caused permanent damage. A unidirectional fabric type sintered at 1250°C, with an involute layup, gave the optimum mechanical properties, whilst the unidirectional fabric with 0/90° layup underperformed in both categories.

2:30 PM

(ICACC-S1-003-2023) Investigation of the high-temperature behavior of oxide-oxide ceramic matrix composites using in situ X-ray tomography and digital volume correlation (Invited)

P. Forna-Kreutzer*[1]; J. Ell[2]; H. Barnard[3]; I. Edmonds[4]; R. O. Ritchie[2]; D. Liu[1]

1. University of Bristol, School of Physics, United Kingdom
2. University of California, Department of Materials Science & Engineering, USA
3. Lawrence Berkeley National Laboratory, Advanced Light Source, USA
4. Rolls-Royce plc, United Kingdom

Two Nextel™610/aluminosilicate oxide-oxide ceramic matrix composites based on 0/90° 1500 denier (D) and 4500 D fabrics were tested in bending at room temperature, 800 °C and 1100 °C with in situ X-ray tomography to examine the evolution of damage under load at temperature. The as-received 1500 D material displayed higher flexural strengths (220 MPa) at elevated temperatures compared to its 4500 D counterpart (190 MPa). A pre-fatigued 1500 D material was also tested at 800 °C, revealing a higher flexural strength (315 MPa). The main fracture mechanism was 0/90° interfacial cracking. 0/0° and 90/90° interfacial cracking and pronounced 90° intra-tow cracking were more prevalent in the 4500 D material and were attributed to increased macro- and microporosity and contact between adjacent 0° fiber tows in the 4500 D material. Fracture in the pre-fatigued 1500 D material was more brittle with thicker, more dominating interfacial cracks. Digital volume correlation revealed the lowest failure strains at 0/90° interfaces and highlighted a profound effect of macropores on fracture. Higher matrix/0° strengths were also found while high strains in the 4500 D material correlated with intra-tow and 0/0° interfacial cracking. The current work demonstrates the impact of reinforcement architectures and pre-fatigue testing on material behavior.

3:20 PM

(ICACC-S1-004-2023) Effects of cooling holes on SiC/SiC CMC strength and durability

C. Smith*[1]; S. Kalluri[2]; R. Bhatt[2]; M. J. Presby[1]

1. NASA Glenn Research Center, USA
2. HX5, USA

Ceramic Matrix Composites (CMCs) such as SiC/SiC are currently operating in select high temperature components of turbine engines. When these materials are implemented in components at higher operating temperatures, film cooling may be necessary. Film cooling requires holes to be fabricated at appropriate locations within these components. This study examines the effect of cooling holes on SiC/SiC composite strength and durability. Mechanical test data on specimens with multiple holes is reported for isothermal conditions at room and elevated temperatures. Tools such as Digital Image Correlation (DIC) and Acoustic Emission (AE) are used to monitor strain and cracking upon loading.

3:40 PM

(ICACC-S1-005-2023) Intermediate Temperature Oxidation of a SiC/SiC Composite with Turbine Cooling Holes

G. C. Ostdiek*[1]; R. J. Kerans[2]; G. Jefferson[3]; J. Pierce[4]

1. US Air Force, AFRL/RQTI, USA
2. Air Force Research Lab, Materials & Manufacturing Dir (emeritus), USA
3. USAF, RXCC, USA
4. University of Dayton, Research Institute, USA

SiC/SiC CMC specimens with holes sized to the cooling holes in turbomachinery were subjected to a furnace dwell and then tensile tested. The furnace temperature range was 500°C - 1100°C, and 100 hour dwells were conducted both with and without steam. Tensile test results showed significant strength degradation for the 500°C - 700°C with steam conditions, with ultimate tensile strength 1/3 to 1/2 that of control specimens. Microscopy and spectrometry of those samples showed significant oxidation of the boron nitride fiber coating in 700°C steam, and volatilization of that coating in 500°C steam and 600°C steam. Porosity was characterized by microscopy and X-ray computed tomography. Extensive degradation was in all cases related to the geometry and extent of porosity; hence, the possibility of extensive and rapid degradation was found to strongly depend upon processing artifacts. The results indicate that process design and control will be key to safe usage of SiC based composites in turbine engines.

4:00 PM

(ICACC-S1-006-2023) Tensile Behavior of 2700°F EBC-CMC System after High Temperature Steam Exposure

A. S. Almansour*[1]; J. D. Kiser[1]; K. Lee[2]; D. Gorican[3]; J. Setlock[4]

1. NASA Glenn Research Center, Ceramic & Polymer Composites Branch, USA
2. NASA Glenn Research Center, Environmental Effects & Coatings Branch, USA
3. HX5, LLC at NASA Glenn, Ceramic & Polymer Composites Branch, USA
4. University of Toledo at NASA Glenn, Environmental Effects & Coatings Branch, USA

In water vapor-rich hot jet engine environments, SiCf/SiC ceramic matrix composite (CMC) components that are coated with 2700°F capable environmental barrier coating (EBC) react with oxygen and water vapor, forming a thermally grown oxide (TGO) silica (SiO_2) layer between the CMC and EBC. When micro cracks or pores in EBC are interconnected, the TGO layer reacts with water vapor to form volatile silicon hydroxide (Si(OH)4) which causes recession of the formed TGO. In this work, several layers of oxide-based EBC were applied on Hi-Nicalon™ Type S SiC fiber-reinforced chemical vapor infiltrated (CVI) SiC minicomposites via slurry spraying. Coated minicomposites were exposed to a steam environment for 50 and 100 hours at temperatures ranging from 2200 to 2600°F without mechanical loading. Minicomposites were then tested in tension at room temperature with the use of acoustic emission to assess surface and subsurface damage mechanisms in the EBC-CMC system. Matrix cracking strength of unexposed coated samples was compared to that of steam-exposed coated samples.

4:20 PM

(ICACC-S1-007-2023) Thermomechanical properties of UHTCMCs produced using the RF-CVI technique (Invited)

V. Venkatachalam*[1]; T. Reimer[2]; J. Binner[1]

1. University of Birmingham, Metallurgy and Materials, United Kingdom
2. Deutsches Zentrum für Luft-und Raumfahrt, DLR, Germany

Ultra-high temperature ceramic composites (UHTCMCs) based on carbon fibre, Cf, preform impregnated with zirconium diboride, ZrB_2, powder and then densified with carbon by radio frequency assisted chemical vapour infiltration, RF-CVI, were mechanically tested to measure the flexural and tensile strengths at room and high temperatures 1600-1800°C. All composites had a degree of delamination during the various mechanical tests and were comparable to literature values. However, despite the oxidation, these UHTCMCs exhibited excellent indications of fibre pullout, possibly due to a weaker fibre-matrix interface resulting from oxidative deterioration. The results also indicated that the 0° orientation plies in the Cf preform structure provided superior resistance to mechanical stresses, indicating that composites can now be engineered to deliver even higher strength levels. Work is currently underway on evaluating various interface coatings in order to enhance their thermomechanical behaviour and these results will hopefully also be presented.

4:50 PM

(ICACC-S1-008-2023) High-Temperature Mechanical Testing of Unidirectional SiC/SiC Composites using a Versatile Lamp Furnace

C. Brockman*[1]; C. Switzer[1]; A. S. Almansour[2]; J. D. Kiser[2]; R. K. Goldberg[2]; P. Sarin[1]

1. Oklahoma State University, Materials Science and Engineering, USA
2. NASA Glenn Research Center, Ceramic and Polymer Composites Branch, USA

Silicon carbide (SiC) based ceramic matrix composites (CMCs) are of interest for high-temperature structural applications, including use in turbine engine components. To understand damage mechanisms and predict durability of these systems, in-depth knowledge of their behavior at ambient and high temperatures is necessary. A method for high temperature testing of unidirectional SiC/SiC composite specimens up to 1500 °C under tensile loading conditions will be presented. This method utilizes a novel radiation heat furnace, equipped with infrared lamps with ellipsoidal reflectors, to heat the specimen at the furnace center in user-defined atmospheres. The housing structure of the lamps allows for observation of the specimen during testing using optical methods and for conducting other experimental studies. The goal is to employ in situ nondestructive health monitoring techniques, including acoustic emission (AE), electrical resistance (ER), and digital image correlation (DIC) to evaluate damage initiation and evolution in the CMC specimens.

5:10 PM

(ICACC-S1-009-2023) Burner Rig Optimization for High Temperature Materials and Coating Systems

C. A. Ferguson*[1]

1. The University of Akron, Mechanical Engineering, USA

Ceramic matrix composites and environmental barrier coatings require high temperature and high velocity tests to approach jet engine and hypersonic conditions for hot section parts. A high velocity oxygen fuel (HVOF) burner rig has been automated with respect to a fixed sample in a horizontally mounted MTS. The two-axis positioner was created using ball screws rotated with two 2.0 Nm stepper motors controlled via a .NET based GUI controller. Through this system, the torch is moved perpendicular from the specimen, changing the distance to the sample and the resulting surface temperature. The closer the torch, the higher the surface temperature as monitored by a FLIR IR camera and a pyrometer. This arrangement allows for thermo-mechanical fatigue tests by changing the position of the torch in a set pattern to create various thermal-mechanical fatigue conditions, e.g., to mimic an aircraft's takeoff, cruise, and landing cycle. Examples of various thermal-mechanical fatigue conditions will be demonstrated and the material response to those conditions.

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

Thermal Barrier Coatings

Room: Flagler C (South Tower)
Session Chairs: Douglas Wolfe, Pennsylvania State University; Eric Jordan, University of Connecticut

1:30 PM

(ICACC-S2-001-2023) Solution Precursor Plasma Sprayed Coatings Applied to Turbine Components with Engine Testing

E. H. Jordan*[1]

1. Solution Spray Technologies, USA

The solution precursor plasma spray (SPPS) has been under consideration for about 2 decades but is now just beginning to be applied to engineering components. Here we will describe the properties of yttrium aluminum garnet SPPS TBC s that led to their demonstration on engine components followed by engine testing at Solar Turbines Inc. The coatings were applied to fuel injector tips, combustor liners and abradable outer air seals. Some issues related to coating complex parts will be presented. The results of subsequent rig and engine testing will be presented. The testing showed promising results. The SPPS YAG coatings show superior performance at higher temperatures compared to YSZ run in parallel to the SPPS YAG coatings. The temperature advantage comes with a lower deposition rate of the SPPS process compared to the APS process.

1:50 PM

(ICACC-S2-002-2023) Effect of laser-structured bondcoats on the furnace cycle lifetime of double-layer Y_2O_3-stabilized ZrO_2/$MgAl_2O_4$ thermal barrier coatings

H. Heyl*[1]; D. Mack[1]; M. Tandler[1]; S. Schrüfer[2]; R. Vassen[1]

1. Forschungszentrum Jülich GmbH, IEK-1, Germany
2. Rolls-Royce Deutschland Ltd & Co KG, Germany

Improving the mechanical interlocking within the layers of a thermal barrier coating (TBC) plays a key role in extending its lifetime. This can be achieved by increasing the surface profile of the bondcoat using laser ablation. To this end, this work analyzes the impact of laser-ablated CoNiCrAlY bondcoats on the furnace cycle lifetime of double-layered Y_2O_3-ZrO_2/$MgAl_2O_4$ TBCs. A laser-ablated bondcoat with a square-shaped grid surface structure and a laser-ablated bondcoat with an additional atmospheric plasma sprayed flash-coat for increased micro-roughness are proposed. As a reference sample, a standard vacuum plasma sprayed bondcoat is used. A detailed analysis of the optimal laser parameters to manufacture homogenous laser-structured bondcoats is presented, while furnace cycle tests at 1100°C are used to identify the performance of the different bondcoat/ceramic topcoat systems. The obtained results show that the cycling lifetime is increased by more than twofold for both structured bondcoats. X-ray diffraction and scanning electron microscope results further reveal that the laser ablation process has no noticeable effect on the material composition. Furthermore, roughness measurements highlight that the bondcoat's macro-roughness has a more pronounced effect on the cycling life than its micro-roughness.

2:10 PM

(ICACC-S2-003-2023) Numerical evaluation of solid particle erosion of EB-PVD TBCs under elevated temperature cycling conditions

K. Chen*[1]

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

The solid particle erosion behavior of electron beam-physical vapor deposition (EB-PVD) thermal barrier coating (TBC) systems was numerically evaluated under thermal cycling conditions. The erosion rates are calculated based on the mechanics-based formulae where the model parameters are fitted to the temperature-process dependent test data. A stochastic approach is applied to simulating erosion behavior close to service conditions. The mechanics-based formulae was validated by experimentally measured temperature and sintering dependent erosion rates. A pseudo-ductile erosion behavior is identified for silica particles EB-PVD topcoat (TC) erosion system above the intermediate temperature (~220°C) due to softening of partial molten silica particles, leading to an increase of cutting wear and decrease of deformation wear. The erosion rates are found to decrease versus temperature but increase versus thermal cycles. Such erosion behavior could be attributed to propagation of sintering cracks developed at elevated temperatures. The parametric study indicates that both erosion parameters and thermal cycling parameters have a profound effect on the erosion mechanism of EB-PVD TC. Two types of erosion mechanisms were evaluated under different thermal cycling conditions.

2:30 PM

(ICACC-S2-004-2023) Thermodynamic investigations at high temperatures of the ZrO_2-$YTaO_4$ quasibinary for thermal barrier coating applications

M. Lepple*[1]; S. V. Ushakov[2]; K. Lilova[2]; C. A. Macauley[4]; C. G. Levi[3]; A. Navrostky[2]

1. Justus-Liebig-University Giessen, Institute of Inorganic and Analytical Chemistry, Germany
2. Arizona State University, School of Molecular Sciences, USA
3. University of California, Santa Barbara, Materials Department, USA
4. Friedrich-Alexander-University Erlangen-Neurnberg, Department of Materials Science and Engineering, Germany

Compositions at the ZrO_2-rich and the $YTaO_4$-rich end of the ZrO_2-$YTaO_4$ quasibinary have been proposed as candidate materials for next generation thermal barrier coatings owing to their attractive high temperature properties. For high temperature applications, it is essential to have a rigorous understanding of the materials system, including phase stabilities and relations as well as quantification of the energies and driving forces for forming the stable and metastable phases. However, investigations of high temperature phase stable materials require measurement temperatures > 1500 °C leading to experimental challenges, such as severe side reactions or temperature limits of the equipment. In this work, transition temperatures, enthalpies and phase stabilities were explored by several thermal analysis techniques, e.g. differential thermal analysis, drop-and-catch calorimetry and in-situ X-ray diffraction. The obtained data and insights are invaluable for CALPHAD modeling to obtain a self-consistent database and comprehensive understanding of the thermochemistry of the quasibinary.

2:50 PM

(ICACC-S2-005-2023) Highly tough, dense zirconia coatings

S. Badie*[1]; S. Conze[2]; L. Berger[2]; R. Vassen[1]

1. Forschungszentrum Juelich, IEK-1, Germany
2. Fraunhofer Institute for Ceramic Technologies and Systems IKTS, Germany

The use of ceramic materials for wear-resistant applications is often limited by their brittleness. Amongst oxide ceramics though, partially stabilized zirconia shows superior performance as a bulk ceramic, originating from the transformation toughening due to a tetragonal-to-monoclinic phase transformation. The objective of the present work is to transfer these properties also to plasma-sprayed zirconia coatings offering numerous new applications. Zirconia coatings were produced by atmospheric plasma spraying of 3YSZ powders. Spraying parameters such as arc current, standoff distance, torch traverse speed, plasma mixture and coating cycles were varied separately in order to achieve the highest densities possible. High currents of up to 540 A, as well as short standoff distances of 65 mm and torch traverse speeds of 400-600 $mm.s^{-1}$ led to a residual porosity of less than 2%, with a 100% content in transformable tetragonal t-ZrO_2. A reduction of the cycle number led to an increase in coating density, as single passes avoided interlaminar porosity. The denser coatings presented segmentation cracks propagating through all of their thickness, but less delamination cracking occurring in transversal direction, likely correlated to the anisotropic grain growth upon cooling and the good intersplat cohesion. The wear-resistance and toughness of the coatings was qualitatively deduced by impact and erosion testing.

3:30 PM

(ICACC-S2-006-2023) GAP-GZO Composite Thermal Barrier Coatings for Superior Thermomechanical Properties and Resistance to Calcium-Magnesium-Aluminosilicate Degradation

D. E. Wolfe*[1]; J. Reiss[1]; M. Schmitt[3]; A. K. Rai[2]; P. Albert[1]; C. DeSalle[1]

1. Pennsylvania State University, USA
2. UES Inc., USA
3. HAMR Industries LLC, USA

State-of-the-art thermal barrier coating (TBC) topcoats comprised of yttria-stabalized zirconia (YSZ) are susceptible to calcium-magnesium-aluminosilicate (CMAS) infiltration and subsequent spallation and failure. The leading alternative to YSZ, gadolinium zirconate (GZO), offers improved resistance to CMAS degradation, but is limited in application due to decreased erosion resistance. Recently, gadolinium aluminum perovskite (GAP) has demonstrated resistance to CMAS degradation while providing improved mechanical durability compared to GZO. However, studies to date have yet to explore the impact of coating microstructure on CMAS infiltration in GAP based topcoats. This work evaluates the mechanical and thermochemical resilience of GAP-GZO composite TBCs deposited via electron beam physical vapor deposition, and correlates the effects of coating composition and microstructure on CMAS and erosion degradation. GAP-GZO composite TBCs are exposed to Eyjafjallajokull volcanic ash infiltration, and evaluated for phase evolution utilizing XRD, SEM, TEM, and EDS. Coatings are also exposed to sand erosion and correlated to relative mass loss. These results are compared to air plasma spray (APS) to establish the process-structure-property-performance relationships associated with leading compositions and deposition techniques.

3:50 PM

(ICACC-S2-007-2023) Thermochemical Stability of High Entropy Rare Earth Oxides (HERO) as Thermal Environmental Barrier Coatings for Refractory Alloys

K. D. Ardrey*[1]; M. Ridley[2]; P. Balachandran[1]; B. Zhou[3]; P. E. Hopkins[3]; E. Opila[3]

1. University of Virginia, Materials Science and Engineering, USA
2. Oak Ridge National Lab, USA
3. University of Virginia, USA

High entropy rare earth oxide (HERO) coatings are under investigation for application on Nb-base refractory alloys to increase operating temperatures in turbine engines beyond the capability of Ni-base superalloys. However, Nb-base refractory alloys lack the ability to form a protective oxide layer, making an environmental barrier coating (EBC) essential for their performance. Currently used coatings for Nb-base alloys do not have sufficient life for long-term turbine engine applications, due to limited thermochemical stability in combustion environments. Potential alternative coating candidates that can provide optimal environmental protection are rare earth oxides. Rare earth oxides have a similar thermal expansion with Nb-base alloys and provide thermochemical stability in a turbine engine environment, giving them the ability to act as an environmental barrier coating (EBC). Furthermore, with the high entropy approach, thermal conductivity may be reduced by mixing multiple rare earth oxides into a single HERO coating, providing dual thermal/environmental barrier coating (T/EBC) capability. This presentation will focus on the effects of HERO coating compositions with simulated combustion engine environmental exposures, and interactions with the underlying Nb-base alloys subjected to high temperature testing.

4:10 PM

(ICACC-S2-008-2023) Investigating Thermal and Optical Properties in Novel Rare Earth Zirconates for Radiative Barrier Coatings

W. Riffe*[1]; H. B. Schonfeld[3]; M. Milich[3]; J. Deijkers[1]; V. Champagne[4]; H. Wadley[1]; D. Clarke[4]; P. Balachandran[1]; P. E. Hopkins[2]

1. University of Virginia, Department of Materials Science and Engineering, USA
2. University of Virginia, USA
3. University of Virginia, Mechanical and Aerospace Engineering, USA
4. Harvard University, USA

Rare earth zirconates show promising thermal and optical properties required of next generation radiative barrier coatings (RBCs) for ultrahigh temperature (1500K+) applications. A fundamental mode of heat transfer that has been neglected in current barrier coatings is radiative heating between hot components. New research aims to mitigate both conductive and radiative heating by doping standard barrier coating materials with rare earth metals that are optically opaque in the spectral range of the emitted radiation. In this work, we perform a series of thermal and optical studies via pump-probe thermoreflectance techniques and ellipsometry to elucidate temperature-dependent thermal conductivity, wavelength-resolved ultrafast electronic transitions, and optical constants of rare earth zirconates. Understanding the trends and thresholds of thermal conductivity is of utmost importance in choosing RBCs that can endure cycling to ultrahigh temperatures. Additionally, we directly measure electronic excitations as a function of wavelength and incident fluence to relate non-equilibrium relaxation of electrons to the optical constants at photon energies associated with peak blackbody emission at ultrahigh temperatures. By investigating pertinent physical scattering mechanisms, we deconvolute key design considerations for next generation RBCs.

4:30 PM

(ICACC-S2-009-2023) Influence of composition on structural evolution and material properties of High Entropy Zirconates

P. Hutterer*[1]; M. Lepple[2]

1. DECHEMA Forschungsinstutut, Germany
2. Justus-Liebig-University Giessen, Institute of Inorganic and Analytical Chemistry, Germany

High Entropy Oxides (HEOs) have attracted great interest in recent years due to their unique properties. Analogous to high entropy alloys (HEAs), they consist of five or more different cations on one or more cation sublattices in approximately same amount. HEOs are considered as promising TBC materials since the high configurational entropy results in improved phase stability at high temperatures and low thermal conductivity is obtained due to increased phonon scattering. However, little is known about the structural evolution of these materials as well as the structure-properties relationships. In this work, HEOs with the general formula $A_2Zr_2O_7$ and up to 10 different cations on the A-site have been successfully synthesized by using reverse co-precipitation. The composition has been varied systematically to evaluate its influence on crystal structure and material properties. Chemical and structural characterization was performed using X-ray diffraction, scanning electron microscopy, electron backscatter diffraction and electron microprobe. Differential scanning calorimetry, HT-XRD and long-term annealing experiments were conducted to assess the thermal stability as well as thermodynamic and thermophysical properties of the compounds. The results were compared to the single element counterparts and an easy indicator for single phase prediction of High Entropy Zirconates is given.

4:50 PM

(ICACC-S2-010-2023) Increased Scattering Coefficient in the Thermal Barrier Coating for High-Temperature Gas Turbine Operations

Y. Wang*[1]; P. Hsu[2]

1. Florida Institute of Technology, USA
2. Florida Institute of Technology, Mechanical Engineering, USA

Thermal barrier coatings (TBCs) are wildly used to protect hot components in the gas turbine engines. With higher turbine inlet temperature, the thermal radiative transfer cannot be ignored in the overall heat transfer in TBC. It has been found that the radiative properties are closely related to the coating's microstructure. By changing the microstructure, the thermal radiative resistance of TBC can be changed effectively. A new approach is developed to control the microstructure of air plasma spayed top coat by mixing the polyester powder with the yttria-stabilized zirconia (YSZ) powder before the thermal spray. By changing the volumetric mixing ratio of YSZ vs. polyester and the size distribution of the polyester powder, different 8YSZ films are fabricated by the air plasma spray process. The room-temperature, spectral transmittance and reflectance data are collected over the near infrared wavelength range and the radiative properties are reduced from spectral measurement. Base on the SEM images of the YSZ films' cross sections, the morphology-based pore size distribution (MPSD) is obtained and the relationship between the MPSD and radiative properties is investigated. The results show that mixing polyester into YSZ powder is a very effective way to modify the microstructure of the top coat, which, in turn, can increase the scattering coefficient.

S3: 20th International Symposium on Solid Oxide Cells (SOC): Materials, Science and Technology

System Design and Demonstration

Room: Ponce de Leon (North Tower)
Session Chair: Mihails Kusnezoff, Fraunhofer IKTS

1:30 PM

(ICACC-S3-001-2023) Overview of U.S. DOE's Solid Oxide Fuel Cell Program (Invited)

P. Burke*[1]

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

The U.S. DOE's Solid Oxide Fuel Cell (SOFC) Program, managed by DOE's National Energy Technology Laboratory (NETL), is focused on achieving significant technological advancements towards the Program's goal of commercialization of SOFC technology via partnerships with National Laboratories, academic institutions, and industrial partners. The Program is also developing synergistic solid oxide electrolysis cell (SOEC) technology. SOEC systems offer a potentially attractive option for producing hydrogen because of high efficiency and system flexibility. In addition to the development of standalone SOEC systems, developers are exploring the potential to use both SOFCs and SOECs in a single hybrid device in order to produce electricity during times of high demand and to produce hydrogen during times of off-peak demand. The Program's near-term objectives include development of small-scale SOFC systems for DG applications validation, development of efficient and cost-effective electrolyzers for hydrogen production, and conducting R&D to make progress towards low cost, high efficiency power and hydrogen generation. The status of the Program and the projects awarded in FY21 to develop SOEC technology and improve understanding of degradation mechanisms in SOECs for efficient and cost-effective production of hydrogen will be presented.

2:00 PM

(ICACC-S3-002-2023) Solid Oxide Cells Stacks Manufacturing for Power-to-gas and Gas-to-power Applications (Invited)

D. Montinaro*[1]

1. SOLIDpower SpA, Italy

SOLIDpower SpA is an Italy-based industry that has acquired HTceramix SA in Yverdon (Switzerland) in 2007 and the assets and team of Ceramic Fuel Cells Germany GmbH in Heinsberg in 2015, and operates also an office in Melbourne, Australia. During more than 20 years experience, SOLIDpower validated competitive manufacturing technologies by extensive lab testing and demonstration in a pilot line, scaling the production up to an industrial automated plant of 25MW per year. Currently, SOLIDpower commercializes a small scale kW cogenerator, the BlueGEN BG-15, for the residential and small commercial market in Europe. The mCHP fleet installed up to date has reached a cumulative size of ca. 3000 units and more than 60 Million operating hours. In order to diversify its offer and be ready to the fast-changing regulation and market trends, a Hydrogen-ready version has been developed. Besides the afore mentioned Gas-to-Power solutions, applications in the field of electrolysis and co-electrolysis have been considered to leverage the large potential of core technology. Particularly, a larger stack with a nominal power of 8 kW, corresponding to 25 kW in electrolysis mode, has been successfully developed and is currently in the industrialization phase.

2:30 PM

(ICACC-S3-003-2023) Elcogen SOC technology: Status and perspectives (Invited)

S. Pylypko*[1]; E. Ounpuu[1]; M. Noponen[1]; H. Grano-Fabritius[1]

1. Elcogen, Estonia

Elcogen is one of the leading European manufacturers of solid oxide cell (SOC) technology. Elcogen products are a unique combination of best-in-class performance and low-cost product structure. The unit cell and stack products are designed for low temperature operation enabling major cost reductions on the system level. Elcogen's operations are located in the Nordic-Baltic technology cluster of Estonia and Finland. This article summarizes the recent activities and development trends of Elcogen. Elcogen solid oxide technology can be used both in electrolysis and fuel cell mode. In electrolysis mode, steam is converted with renewable electricity into green hydrogen. The electrolysis technology can also be used to produce syngas mixture for e-fuel production by combining steam and carbon dioxide. In the fuel cell mode, a variety of fuels can be used for power generation, including various hydrocarbons and carbon-free fuels. Elcogen technology can be operated at high conversion efficiency level for both modes as well as in reversible operation, enabling coupling with renewable power sources for massive energy storage. Elcogen is currently focused on ramping up its unit cell and stack production with increased customer demand. The goal is to set-up of a manufacturing plant in Estonia for the unit cells and stacks with annual capacity of 50 MW in fuel cell mode equaling to 200 MW capacity in electrolysis mode.

Electrolysis and Applications

Room: Ponce de Leon (North Tower)
Session Chair: Albert Tarancón, IREC / ICREA

3:20 PM

(ICACC-S3-004-2023) Power-to-X and Solid Oxide Electrolysis offerings at Topsoe (Invited)

S. D. Ebbesen*[1]; P. Blennow[1]; T. Heiredal-Clausen[1]; J. Rass-Hansen[1]; J. Bøgild Hansen[1]; P. Moses[1]

1. Topsoe, Power to X, Denmark

Topsoe's strategic vision is to be recognized as the global leader in carbon emission reduction technologies by 2024. As part of this strategy Topsoe has established a focused Power-to-X division to accelerate our electrolysis and Power-to-X business. Topsoe's dedicated ceramic based high-temperature electrolysis technology is designed to integrate seamlessly with downstream processes, so green hydrogen can be processed into ammonia, for use in chemical applications or energy storage. With decades of experience as a world-leading provider for a wide range of Power-to-X solutions, Topsoe is one of the very few companies possessing expertise and technologies along the entire value chain to transform renewable electricity into hydrogen, ammonia, chemicals and eFuels for zero-emission fuels and chemicals. Topsoe is committed to meet the growing need for production of green hydrogen and have taken final investment decision to upscale the electrolysis production capacity and begin construction of one of the world's largest SOEC electrolyser manufacturing plants. Further Topsoe and First Ammonia have launched the green ammonia production with the world's largest reservation of electrolyser capacity for production of green ammonia by solid oxide electrolysis. Topsoe's various stepping-stone projects on the road to accelerate and upscale the Power-to-X and Solid Oxide Electrolysis offerings will be presented.

3:50 PM

(ICACC-S3-005-2023) Solid Oxide Technolgy Development and Demonstration for Terrestrial and Space Applications - Materials to Device Challenges (Invited)

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

1. OxEon Energy, LLC, USA

High temperature electrolysis using solid oxide cells provide an efficient way of storing renewable energy in the form of hydrogen by electrolyzing steam. Co-electrolysis of steam and carbon dioxide allows energy storage in the form of hydrocarbon fuels, both synthetic methane and liquid hydrocarbon fuels. The use of electrolysis to make fuel and oxygen for lunar and Martian applications is another avenue that OxEon Energy is actively pursuing, building upon the successful demonstration of oxygen production on Mars by electrolyzing Mars atmosphere carbon dioxide. While broader materials selection is common to all applications, making them robust for space applications requires additional modifications to the materials set. Furthermore, the operating conditions for each application add additional requirements to the balance of plant design that need to be customized. OxEon Energy is also expanding the application to reversible solid oxide system, which introduces complexity to the thermal management strategy. Key challenges and OxEon's approach will be discussed.

4:20 PM

(ICACC-S3-006-2023) Optimization of solid oxide electrolyzers – through modified microstructure to enhanced macro-level performance (Invited)

J. Kupecki*[1]; A. Niemczyk[1]; S. Jagielski[1]; M. Kosiorek[1]; R. Kluczowski[1]; D. Katla[1]

1. Institute of Power Engineering, Center for Hydrogen Technologies (CTH2), Poland

The rapid development of hydrogen value chains worldwide is leading to the scaling up of electrolyzers and advances in materials, design, and manufacturing processes. Of the three generic types of electrochemical hydrogen generators - alkaline (AWE), proton conducting membrane (PEM) and solid oxide (SOE) electrolyzers - the last is attracting much research effort aimed at optimizing performance, durability, and production cost. SOEs are currently in the phase of subsidized demonstrations, with initial deployments in the range 200 – 1,000 kW. While greater efficiency of hydrogen generation can be achieved through improved system integration of high temperature electrolysis and optimization of working conditions, microstructural modifications may be beneficial for the kinetics of electrode reactions, and could limit degradation and broaden the operating envelope. This work focuses on modifications - implemented at the level of SOE electrodes - which lead to reductions in operating voltage and in the energy required to produce hydrogen in solid oxide electrolyzers designed for operation as a part of power-to-X systems i.e. for production of synthetic fuels or as standalone hydrogen generators. The presented research was financially supported by the National Centre for Research and Development, Poland, under project no. LIDER/1/0003/L-12/20/NCBR/2021.

4:50 PM

(ICACC-S3-007-2023) Upscaling of proton conducting ceramic cells and stack components (Invited)

R. Kiebach*[1]; F. Palmerini[1]; X. Georgolamprou[1]; S. Pirou[1]; P. V. Hendriksen[1]

1. Technical University Denmark, DTU Energy, Denmark

Ceramics based on proton-conducting oxides are promising for various applications, including fuel cells, electrolysis, hydrogen pumping, or chemical synthesis, but up-scaling of cells and stack remains a challenge. This presentation will give an overview of ongoing activities at the Technical University of Denmark (DTU Energy) to address this challenge. The latest results on upscaling planar proton-conducting ceramic cells (PCCs) based on Ba(Ce, Zr)O_3 will be presented. Symmetrical and asymmetrical cells with an area of <140 cm^2 can be produced, and the geometry and composition of the electrolyte and electrodes can be varied/optimized for different applications. Also, the development of auxiliary components like interconnects and glass ceramic sealants needed to build stacks will be discussed. Furthermore, potential stacks design, which can circumvent challenges arising from the thermal expansion mismatches of the different materials used in PCC stacks, will be highlighted.

5:20 PM

(ICACC-S3-008-2023) AVL's SOEC Portfolio for highly efficient Hydrogen and eFuels production

R. Schauperl*[1]

1. AVL List GmbH, Research & Innovation, Austria

This work presents AVL's most recent development activities in the field of SOEC based hydrogen and fuel production. The main highlights will be the development of a Power-to-Liquid plant. A thermodynamic analysis as well as the detailed plant concept of a fully integrated SOEC-Fischer-Tropsch Power-to-Liquid plant for a highly efficient production of synthetic aviation fuel was carried out. The project provides the unique possibility to design the plant by optimizing and integrating both processes, SOEC and Fischer-Tropsch, in the most efficient manner. Especially, heat integration between both processes as well as product tail gas recycling will be the key for overall efficiencies of 50-55 %. Eventually, estimates of diesel prices in the range of 1-3 EUR/L are discussed as an alternative to fossil based diesel. Furthermore, AVL currently focuses on the transition towards the use of blended CNG/H_2 and pure H_2 mixtures for H_2-ready SOC solutions. An outlook of potential design changes caused by hydrogen requirements and its impact on system performance in terms of electricity and heat production will be shown. Furthermore, a Levelized Cost sensitivity analysis for electricity and hydrogen production will be provided as a basis for an economic assessment of the SOC technology.

5:40 PM

(ICACC-S3-009-2023) Onboard Desulfurization of Jet Fuel for Fuel Cell Applications

Y. Du*[2]; D. Panthi[1]; H. Feng[2]; S. K. Sahu[2]; I. Soliman[2]

1. Kent State University, Engineering Technology, USA
2. Kent State University, USA

One of the major benefits of solid oxide fuel cells (SOFCs) is their fuel flexibility and tolerance toward fuel impurities. However, the presence of sulfur, a common contaminant in hydrocarbon fuels, is a concern because it can poison the SOFC anode at concentrations as low as a few ppm. In this work, we developed a lightweight and compact desulfurization reactor for onboard sulfur removal from liquid hydrocarbon fuels such as jet fuel for SOFC applications. The 3D printed desulfurization reactor was tested under different operating conditions and with different sorbent materials. We observed a strong influence of operating parameters such as temperature and fuel flow rate as well as temperature distribution inside the reactor on the desulfurization performance. Our results confirm that the developed desulfurization system is capable of reducing the sulfur content in jet fuel to a level suitable for fuel cell applications. Moreover, the regeneration of metal oxide sorbents for their repeated application was demonstrated. The scaling up of the developed reactor for practical SOFC systems and other related applications will be discussed.

S5: Next-Generation Bioceramics and Biocomposites

Biomimetic and Bioactive Ceramics

Room: Ballroom 1-2 (South Tower)
Session Chair: Katalin Balazsi, Centre for Energy Research HAS

1:30 PM

(ICACC-S5-001-2023) Real-time monitoring of early-stage dissolution of bioactive glasses in simulated body fluids (Invited)

D. Galuskova[1]; H. Kankova[1]; L. Bunova[1]; D. Galusek*[1]

1. Alexander Dubcek Univeristy of Trencin, FunGlass, Slovakia

Doping of bioactive glasses with therapeutic ions was reported to promote angiogenesis, osseoconductivity, or their antibacterial properties. The structural role of the dopant affects dissolution of glass, controling its bioactivity, expressed by the formation of hydroxyapatite. Leaching of therapeutic ions is usually monitored in-vitro, in a variety of liquids simulating human body environment. The results are then related to biological properties of glass, such as antibacterial activity, cytocompatibility, or cytotoxicity, often with controversial results. The controversy can be attributed to improper selection of body fluid, or unsuitable conditions of the test, with the content of leached elelements monitored under static conditions after a long time (1 day and longer), with limited knowledge of the processes in the initial phases of the contact of glass with SBFs. This problem was addressed in this work, by real-time monitoring of the early-stage dissolution and ion leaching kinetics of bioactive glasses with different therapeutic ions in various simulated body fluids (SBF, MEMS, Tris) by an in-line ICP OES. The results confirmed burst release of some therapeutic ions in the initial period of dissolution, with the concentrations exceeding cytotoxicity limits. This attitude contributes to better explanation of the results of biological testing of bioactive glasses.

2:00 PM

(ICACC-S5-002-2023) Synthesis and in vitro biocompatibility of nanofibrous TiO_2 materials (Invited)

Y. Wang[1]; S. Chen[1]; W. Chen[1]; A. Osaka*[2]

1. Taiyuan University of Technology, College of Biomedical Engineering, China
2. Okayama University, Faculty of Engineering, Japan

TiO_2 is one of the materials with good biocompatibility, while their nanowires are examples of the representative nanofibrous materials and possess extracellular matrix-like fibrous feature. We hydrothermally treated two types of supportive substrates, i.e., 2-dimensional compacts and 3-dimensional microspheres of TiO_2 nanoparticle, in the concentrated sodium hydroxide solution. With the alkali hydrothermal treatment, we were successful to transform in situ those TiO_2 nanoparticles to TiO_2 nanowires. According to the cell culture tests, TiO_2 nanowires grown on both types of substrates were biocompatible and favored cell attachment and proliferation.

2:30 PM

(ICACC-S5-003-2023) Novel borosilicate bioactive glass material for bone implants

A. Szczodra*[1]; A. Houaoui[2]; J. Massera[2]

1. Tampere University, Finland
2. Tampere University, Faculty of Medical Sciences and Technology, Finland

Commercial bioactive glasses, S53P4 and 1393, demonstrate crystallization tendencies during sintering. Thus, manufacturing of these glasses into 3D porous scaffolds without their bioactivity being reduced is challenging. Therefore, bioactive glass bone graft products are limited to powders, granules, putties. Yet, 3D scaffolds with large pores and highly interconnected porosity are necessary for osteoconductivity, tissue infiltration and regeneration. To overcome these problems borosilicate glasses were developed. Glasses with high boron content show fast conversion into hydroxyapatite and thermal properties allowing sintering into 3D scaffolds. Moreover, boron substitution for SiO_2 showed strong ability to stimulate osteogenic commitment and upregulate endothelial markers. The aim of the study was to develop new borosilicate bioactive glasses 3D printed scaffolds. The addition of strontium has been shown to stimulate osteogenic response from human bone marrow mesenchymal stem cells. Magnesium broadens the sintering window and therefore suppress crystallization tendency of glass during processing. Firstly, mechanical properties of these scaffolds will be measured. Moreover, the human fat stem cells viability will be assessed after 7 days of culturing with scaffolds. Finally, cell behavior will be explained using ion release profiles from in vitro dissolution in TRIS and SBF buffer solutions.

Biomimetic and Bio-inspired Ceramics

Room: Ballroom 1-2 (South Tower)
Session Chair: Hui-suk Yun, Korea Institute of Materials Science

3:20 PM

(ICACC-S5-004-2023) Multifunctional scaffolds based on Sr,Mg,Ag-substituted octacalcium phosphate and carboxymethyl chitosan (Invited)

A. Ressler*[1]; M. M. Marić[1]; H. Ivanković[1]; M. Ivanković[1]

1. Faculty of Chemical Engineering and Technology, University of Zagreb, Croatia

One of the used approaches to improve the biological and physicochemical properties of scaffolds is ionic substitutions with key role ions. Octacalcium phosphate (OCP) is considered a precursor of biological apatite and has shown remarkable regenerative properties. In this study, Sr,Mg,Ag-substituted OCP powders were prepared by precipitation method with varying substitution content (0, 1 and 2.5 mol%). Prepared powder samples were composed of OCP and amorphous calcium phosphate, while lattice parameters of OCP changed depending on the size of Sr^{2+} (0.113 nm), Mg^{2+} (0.072 nm) and Ag^{+} (0.0128 nm) ions. Substitution with Sr^{2+} and Ag^{+} ions increased, while with Mg^{2+} ion decreased the thermal stability of OCP. Substituted OCP crystals have shown characteristic plate-like morphology with homogeneously distributed ions. To obtain multifunctional scaffolds, Sr,Mg,Ag-substituted OCP powders have been mixed to determine the synergic properties of substituents. The substituted ions enhanced protein adsorption capacity and antibacterial activity towards Gram-positive Staphylococcus aureus and Gram-negative Escherichia coli. Highly porous scaffolds based on obtained powders and carboxymethyl chitosan have been prepared by the feeze-gelation technique. The scaffolds have shown a highly porous structure, with interconnected pores and high stability in simulated body conditions.

3:50 PM

(ICACC-S5-005-2023) Bringing Light into darkness: Biophotonic scaffolds (Invited)

J. Massera*[1]

1. Tampere University, Faculty of Medical Sciences and Technology, Finland

Since the discovery of bioactive glasses by L.L. Hench, several glass compositions have found space in the clinics (45S5, S53P4, 13-93, nanoborate fibers) in a wide range of applications. More recently researchers have focused on adding bio-functionality such as antimicrobial or pro-angiogenic properties, by doping the glass with specific ions (Ag, Cu, B etc…). However, some drawbacks, inherent to the traditional silicate bioactive glasses, still remains. For example, traditional bioactive glasses are prone to crystalize upon sintering, thus preventing their manufacturing into porous scaffold. Furthermore, bioactive glasses are radio transparent, thereby tracking the material degradation and osteointegration is difficult in-vivo. Here, the development a borosilicate glass enabling 3D robocasting will be presented. These scaffolds were further processed with persistent luminescent particles. The impact of particles degradation, as well as light emission, on cell fate has been assessed. These new luminescent bioactive glasses open the path to in-vivo bioimaging or photo-drug release.

4:20 PM

(ICACC-S5-006-2023) Development of peptide synthesized hydrogel with loaded calcium phosphate as bi-layered osteochondral structure

J. Kim*[2]; H. Park[1]; H. Yun[1]

1. Korea Institute of Materials Science, Republic of Korea
2. University of Science and Technology, Republic of Korea

Osteoarthritis is the most common chronic degenerative joint disease which affected world widely. Through the limitations from surgical treatments, development of multi-functional materials is required to improve each differentiation function of osteochondral tissue. For that reason, in this study, we tried to develop the multi-functional bi-layered hydrogel through integration of each layer. First as articular cartilage part, N-Cadherin peptide was synthesized to methacrylate modified alginate (MA-alginate). The peptide synthesized hydrogel showed enhancement in cell proliferation and chondrogenesis, either way. Secondly, for subchondral bone part, RGD peptide was synthesized to MA-alginate and alpha-tricalcium phosphate (α-TCP) was added in matrix. The peptide synthesized and α-TCP loaded, simultaneously, hydrogel showed improvement in osteogenesis. Also, α-TCP cement reaction significantly improved the mechanical property and osteogenesis without significant damage to encapsulated cells during the reaction. After in vitro cellular activities, possibility of fabrication as integrated structure through two-step crosslinking was investigated. Consequently, we expected this multi-functional bi-layered hydrogel developed in this study, can potentially be used for osteochondral tissue regeneration.

4:40 PM

(ICACC-S5-007-2023) Bioinspired, osseoconductive calcium phosphate coatings by Electrostatic Spray Deposition

M. Veronica[2]; L. Gremillard*[1]; S. Tadier[1]; C. Gaillard[1]; E. Djurado[2]

1. INSA, Materials, Engineering and Science, France
2. CNRS LEPMI, France

For an optimal efficiency of orthopaedic implants such as hip and knee total replacement, their good integration in the surrounding bone is necessary. Osseointegration is often favoured by a calcium-phosphate coating on the implants surface. Traditionally, these coatings are fabricated by plasma-spray, a process that involves very high temperature and fast thermal cycles, leading to well crystallized films mostly composed of hydroxyapatite but sometimes also containing other, out-of equilibrium phases. Even though these plasma-sprayed coatings are efficient, their use is sometimes subject to controversy because of several drawbacks such as excessive thickness, possible delamination leading to local inflammations, and the overly stable nature of the constitutive materials that do not favour reactivity. We present here an alternative process, Electrostatic Spray Deposition, that enables the fabrication of thin, nanostructured hydroxyapatite coatings with tunable morphology (from dense to coral-like) through a cheap and low-temperature route. The effects of precursors and processing parameters on the composition, morphology and biologival activity of the coatings will be described.

5:00 PM

(ICACC-S5-008-2023) Investigation of rice husk-derived silica toxicities depending on synthesizing methods

J. Lee*[1]; R. J. Mitchell[2]; J. Park[1]; W. Mun[2]

1. Korea Institute of Ceramic Engineering and Technology (KICET), Biomaterials & Processing Center, Republic of Korea
2. Ulsan National Institute of Science and Technology, Republic of Korea

Rice husks are an ever-growing renewable biomass, with global rice husk output was an estimated 194 million tons in 2020. Rice husks are a feedstock of biogenic silica because of their high silica content. As interest in natural biogenic silica nanoparticles (NPs) from rice husks grows, it is important to know how their preparation impacts their biocompatibility. Within this study, three methods (combustion, acid leaching or alkali extraction) to purify silica NPs from rice husks were explored, with an emphasis on their inherent toxicity towards human cell cultures. Whereas acid leaching and alkali extraction both generated highly pure silica NPs (>99.1% SiO_2 vs. 93.1% with combustion), toxicity tests performed with human and mouse cell lines found the alkali-extracted silica NPs to be much more biocompatible. We explored the toxicity further by incorporating polyethylene glycol (PEG) in the alkali-extracted silica NPs prior to the calcination step to mimic the presence of organic carbon during this step. The significantly increased their toxicities were found in PEG incorporated silica NPs and was exacerbated further when calcination was performed at a lower temperature and time. These results show that the biocompatibility of rice husk-derived silica NPs is negatively affected by the presence of residual carbon during calcination.

S6: Advanced Materials and Technologies for Rechargeable Energy Storage

All-solid-state Batteries I

Room: Coquina Salon E (North Tower)
Session Chairs: Palani Balaya, National University of Singapore; Olivier Guillon, Forschungszentrum Juelich

1:30 PM

(ICACC-S6-001-2023) Dynamic stability design for fast charging solid state batteries (Invited)

X. Li*[1]

1. Harvard University, SEAS, USA

Solid-state batteries pose new challenges to the battery design due to the unique solid-solid interfaces at battery cathode and anode. However, these interfaces, upon critical understanding and design, also form the new opportunity to unlock advanced battery performances. We design solid state batteries based on our unique mechanical constriction principle, demonstrating a stable cycling against the lithium dendrite penetration at high current densities up to 40 mA/cm^2.

2:00 PM

(ICACC-S6-002-2023) Interfacial stability of LLZO/LCO and LLTO/LNMO systems in oxides-based all-solid-state Li batteries (Invited)

S. Lin*[1]; C. Lin[1]

1. National Cheng Kung University, Materials Science and Engineering, Taiwan

Interfacial issues have been the bottleneck of all-solid-state batteries (ASSB) development. Among the issues, interfacial instability is one of the main reasons causing high interfacial impedance upon cycling in ASSB. $Li_7La_3Zr_2O_{12}$ (LLZO) and $Li_{0.33}La_{0.56}TiO_3$ (LLTO) are promising oxide-based solid electrolytes for ASSB. LLZO possesses good stability against Li metal anode, and LLTO has high bulk ionic conductivity. In the development of LLZO-based ASSB, $LiCoO_2$(LCO), which is the most common layered cathode material, is often used as the cathode material for composite cathode. Although well sintered LLZO/LCO composite cathode can be obtained, the batteries degrade rapidly. In this presentation, we performed ab initio thermodynamic calculations to clarify the reason of capacity fading for Al-doped LLZO/LCO ASSB. Combining with experimental analyses, the degradation mechanism of LCO/Al-doped LLZO was revealed. For LLTO, to improve the interfacial stability of LLTO-based ASSB, we designed the interface between LLTO and $LiNi_{0.5}Mn_{1.5}O_4$ (LNMO) high voltage cathode material based on computation. According to our calculation, dopants that can improve the LLTO/LNMO interfacial stability are proposed. The interfacial stability of LLZO/LCO and LLTO/LNMO systems in ASSB is discussed.

2:30 PM

(ICACC-S6-003-2023) Interface Engineering of $Li_7La_3Zr_2O_{12}$ for all-solid-state-lithium battery

K. K. Halankar*[1]; S. Mathur[1]

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

Lithium-ion batteries using solid-state electrolytes are considered to be the most promising direction to achieve safe higher energy density secondary batteries. $Li_7La_3Zr_2O_{12}$ (LLZO) possesses high mechanical properties but poor interface compatibility. This research work focuses on challenges to compensate for the disadvantages of LLZO electrolyte material and engineer membrane electrolyte, which has good flexibility and can be easily mass-produced. The LLZO-based engineered electrolyte is electrochemically stable in the wide voltage range of 0–5.0 V vs. Li/Li^+ due to the good oxidative stability of LLZO. A simple and low-cost approach to fabricate the all-solid-state lithium battery (ASSLB), by constructing an atomic wet coating and hot pressing electrolyte to form a continuous and homogeneous electrolyte layer on a cathode layer. The integrated preparation process improves interface contact by reducing the resistance between the cathode layer and the electrolyte layer. The $Li/LiNi_{0.8}Mn_{0.1}Co_{0.1}O_2$ batteries show marked improvements in performance, in terms of electrolyte physical and electrochemical properties, as well as coulombic efficiency, interfacial compatibility, and cyclebility of solid-state cells.

All-solid-state Batteries II

Room: Coquina Salon E (North Tower)

Session Chairs: Valerie Pralong, CNRS ENSICAEN; Xin Li, Harvard University

3:10 PM

(ICACC-S6-004-2023) Protecting solid-state batteries from failure by using pulsed current waveform and ion implantation

D. Rettenwander*[1]

1. NTNU Norwegian University of Technology, DMSE, Norway

Solid-state Li batteries (SSLB) belong to the most promising concepts for boosting the transformation of current transportation to low-emission modes. High theoretical energy density of SSB makes them a powerful tool for improving the efficiency of current transportation systems. However, to reach the promised performance, SSLB must reversibly plate a large amount of Li at a high current rate (>5 mA/cm^2) for at least 1000 cycles. Today, under such conditions, SSLB invariably fail due to Li dendrites. The formation and growth of these dendritic structures are believed to be driven by stress accumulation stemming from the deposited Li itself. Upon reaching a critical pressure, the solid-state electrolyte starts to crack, which propagates until the cell short-circuits. Herein, I will show that pulsed current waveforms and ion implantation are promising strategies to mitigate Li dendrites. For example, I will demonstrate that pulsed current waveforms can be used to increase the current density by a factor of six, reaching values as high as 6.6 mA/cm^2 without forming Li dendrites. Moreover, I will highlight ion implantation as a tool to tune the surface-near mechanical properties of solid-state electrolytes by introducing high compressive residual stresses, which allows the deflection of cracks with the aim to stop Li dendrites from propagating.

3:40 PM

(ICACC-S6-005-2023) Optimization and sintering of LATP-based solid-state battery (Invited)

M. Kusnezoff*[1]; K. Waetzig[1]; J. P. Beaupain[1]; H. Auer[1]; K. Nikolowski[1]; M. Partsch[1]; A. Michaelis[1]

1. Fraunhofer IKTS, Germany

All solid-state batteries (ASSB) working at ambient temperature are very promising but simultaneously most challenging approach for electrochemical storage devices. Especially oxide / phosphate electrolytes offer unique opportunities for in air manufacturing, intrinsic safety, and high energy density. Among Li^+-ion conducting ceramic electrolytes $Li_{1+x}Al_xTi_{2-x}(PO_4)_3$ (LATP) is the material, which offers the possibility for reduced temperature sintering and processing and therefore the ability to build and demonstrate proof of concept for ASSB. In the presentation the overview of our research on sintering of LATP based electrolyte as function of particle size, sintering aids and temperature will be provided. The main challenge for ASSB is the realization of composite cathode with acceptable performance. The influence of sintering temperature (down to cold sintering at temperatures below 250°C) on performance of different composite cathodes will be shown and electrochemical performance of sintered cathodes and its relationship to electrode microstructure discussed. Due to the poor ionic conductivity of sintered LATP backbone the reasonable performance can be only demonstrated using liquid or polymer electrolyte infiltration. The best performance of composite electrode with polymer electrolyte infiltration was achieved for composite cathodes sintered at 700 °C and was 140 mAh/g.

4:10 PM

(ICACC-S6-006-2023) Scalable Fabrication of Composite Solid-State Electrolytes with Improved Stability (Invited)

Z. Chen*[1]

1. University of California, San Diego, Department of NanoEngineering, USA

All solid-state batteries (ASSBs) have the potential to deliver higher energy densities, wider operating temperature range and improved safety compared with today's liquid electrolyte-based batteries. However, of the various solid-state electrolyte (SSE) classes: polymers, sulfides or oxides, none alone can deliver the combined properties of ionic conductivity, mechanical and chemical stability needed to address scalability and commercialization challenges. While promising strategies to overcome these include the use of polymer / oxide or sulfide composites, there is still a lack of fundamental understanding between different SSE-polymer-solvent systems and its selection criteria, which prevents the scalable manufacturing using industry compatible processes. In this talk, we will isolate various SSE-polymer-solvent systems and study their molecular level interactions by combining various characterization tools. With these findings, we will introduce a suitable sulfide SSE - SEBS polymer - solvent combination that significantly reduces SSE thickness (<50 μm), with improved uniformity and ionic conductivity obtained under the guidance of machine learning (ML). In addition, we will also discuss the chemical evolution of a sulfide electrolyte during air exposure and evaluate its dry room compatibility. These results can provide some design implication for future ASSB manufacturing processes.

4:40 PM

(ICACC-S6-007-2023) Wet-Chemical Spray Drying of Coatings on Cathode Active Materials for Solid-State Batteries

J. P. Beaupain*[1]; K. Waetzig[1]; S. Yanev[1]; H. Auer[1]; K. Nikolowski[1]; M. Partsch[1]; M. Kusnezoff[1]

1. Fraunhofer IKTS, Germany

Cathode active materials (CAM) with high Ni content ($LiNi_xMn_yCo_zO_2$ $x \geq 0.8$) are used in conventional and solid-state batteries due to their high energy density, capacity and low costs. However, the high nickel content induces unwanted reactions at the interface between active material and the solid electrolyte (SE), which limit the electrochemical performance and fast-charging ability. A promising concept to optimize the interface is the introduction of thin protective coatings to prevent direct contact between CAM and SE. The coating requires mixed ionic and electronic conductivity to minimize oxidative decomposition of the SE. In addition, thickness of < 10 nm must be achieved for optimal performance, leading to high requirements of the coating homogeneity. Different technologies investigated up to date include dry, wet-chemical and vapor phase coating deposition, which have differences in terms of their coating homogeneity, scalability, and costs. In this study, spray drying is used to investigate wet-chemical coatings (Li-M-O with M = Si, Zr, Nb, W) of CAM. The coating homogeneity of the materials is characterized by imaging electron- and He-microscopy. Further analysis regarding interactions between CAM and coating are investigated by XPS spectroscopy and XRD. Finally, the electrochemical properties of the coated CAM are demonstrated with liquid and solid state (sulfide) electrolyte.

5:00 PM

(ICACC-S6-008-2023) High performance solid-state batteries with garnet scaffolds prepared by phase inversion

F. Shen*[1]; M. Tucker[1]

1. Lawrence Berkeley National Laboratory, USA

Garnet material is one of the most promising electrolytes due to its negligible electron conduction, wide potential window range, and good chemical stability to lithium metal. However, the high interfacial resistance between the garnet electrolyte and cathode, a couple orders of magnitude higher than the anode interfacial resistance and electrolyte resistance, is still a challenge. In addition, inhomogeneous current distribution resulting from poor physical contact can lead to short circuiting at a low current density. A small amount of liquid electrolyte or ionic liquid is often added at the cathode to reduce interfacial resistance, while leading to safety and other concerns. In this study, porous scaffolds are prepared by phase inversion, which creates finger-like pores when the solvent in a slurry counter-diffuses with a non-solvent liquid in contact with the slurry surface. Surface pore access is improved via a sacrificial layer, and the pore size is controllable. The scaffold serves as an electrode support with a thin electrolyte layer. The apparent interfacial resistance on the cathode side is reduced by the large interfacial surface area within the scaffold, compared to cells with a planar structure. This improves energy density as thinner garnet electrolyte is feasible.

S8: 17th International Symposium on Advanced Processing and Manufacturing Technologies for Structural and Multifunctional Materials and Systems (APMT17)

Design-oriented Manufacturing and Processing

Room: Coquina Salon F (North Tower)
Session Chair: Hisayuki Suematsu, Nagaoka University of Technology

1:30 PM

(ICACC-S8-001-2023) Advanced Thermal Diffusion Protective Coatings: Major Processing Principles and Applications (Invited)

E. Medvedovski*[1]

1. Endurance Technologies Inc., Canada

The major principles of the coatings' formation and manufacturing through thermal diffusion CVD-based processes are discussed and analyzed. The advanced thermal diffusion coatings on steels and alloys with multi-layered architectures consist of 2-3 coating layers, which can be composed of borides, aluminides, chromides and some other compounds. They also may have an additional top layer of a few-micron thickness based on selected oxides or non-oxides forming composite coatings. The major processing steps and features are outlined. The coating architectures and total thickness varied from ~25 to 250 µm or greater depend on the metallic substrate, selected starting compositions providing desirable coating phase composition and structure, process parameters, as well as the application requirements. These coatings are successfully produced to protect complex shape components and long tubing for wear and/or high-temperature corrosion applications. Multi-layered structure and diffusion-induced bonding between the protective layers and the substrate promote the coatings' integrity and delay micro-crack propagation under stress conditions occurring in service.

2:00 PM

(ICACC-S8-002-2023) Materials design to minimize the water absorption and pyroplastic deformation of alumina-strengthened porcelain

D. Hao*[1]; T. Akatsu[1]; N. Kamochi[2]

1. Saga University, Japan
2. Saga Ceramics Research Laboratory, Japan

Pyroplastic deformation is one of the most critical problems that should be solved to improve the productivity of porcelain. However, because of the contradictory relationship between pyroplastic deformation and water absorption, it is difficult to fabricate the porcelain with both small pyroplastic deformation and small water absorption in a wide firing temperature range. In our study, we found that minimization could be achieved by controlling the balance of densification and high-temperature crystallization, which could be controlled by using IA or IIA oxides and by using the high-temperature crystallization precursor, respectively. Unlike most traditional porcelains, our newly designed porcelains exhibit both small water absorption and small pyroplastic deformation in a wide firing temperature range, which is important to reduce production loss. In this presentation, strategies for minimizing pyropalstic deformation and water absorption will be introduced from the materials design viewpoint. This research will provide feasible support for the fabrication of tableware, sanitary ware, insulators, and other ceramics.

2:20 PM

(ICACC-S8-003-2023) Control of forming time and magnetic flux density in colloidal forming in high magnetic field (Invited)

S. Tanaka*[1]

1. Nagaoka University of Technology, Materials Science and Technology, Japan

Crystal orientation is effective for improving the performance of functional ceramics with crystal anisotropy. In colloidal forming in a high magnetic field, the crystal particles are oriented due to the magnetic anisotropy of the crystals. The forming process in a high magnetic flux density also needs to improve the problems such as molding time. A method for shortening the manufacturing time in a magnetic field and lowering the magnetic flux density was investigated. Casting and gel casting in a magnetic field take a long time to solidify. Therefore, we tried ultraviolet curing molding in a magnetic field. After being held in the magnetic field for about 10 seconds, it could be solidified by irradiating with ultraviolet light, shortening the time to less than 1/10. In $(SrCa)_2NaNb_5O_{15}$, it was shown that a sheet with a thickness of 0.1 mm can be produced and laminated. Even in reducing the magnetic flux density, it is effective to devise a particle side. In the case of zeolite particles, by ion exchange with transition elements, etc., and high orientation at 2T is possible.

Green Manufacturing, Global Environmental Issues and Standards

Room: Coquina Salon F (North Tower)
Session Chair: Eugene Medvedovski, Consultant

3:20 PM

(ICACC-S8-004-2023) Chemisorption of molybdenum oxide on the surface of stainless steel 304 at high temperatures

T. Do*[1]; T. Nakayama[2]; H. Suematsu[2]

1. Nagaoka University of Technology, Nuclear System Safety Engineering, Japan
2. Nagaoka University of Technology, Japan

In nuclear severe accidents, cesium is released to the reactor coolant system in the form of cesium hydroxyl (CsOH) or cesium molybdate (Cs_2MoO_4). Under the steam condition at high temperatures (T > 1200 °C), a part of Cs_2MoO_4 would be decomposed to form molybdenum trioxide (MoO_3) and cesium oxide (Cs_2O) which later would react to steam to form CsOH. The behavior of Cs_2MoO_4 and CsOH has been focused for a time, however, MoO_3 does not seem to have been focused. In the result of Phebus Test 1, up to 23 % of molybdenum inventory was released to the containment vessel. Besides, MoO_3 gaseous could deposit on the structure material such as stainless steel from about 750 °C, and the reaction between iron oxide and MoO_3 may occur. Therefore, it is necessary to understand the behavior of MoO_3 during severe accidents. In this study, the possible reaction between Cs_2MoO_4 (gas) and SUS 304 at 1200 °C was studied. It is found that hematite (Fe_2O_3) outside the spinel $(Fe, Cr)_3O_4$ layer contained Mo-enriched precipitates. In the literature, the thermodynamics calculation for the Fe-Mo-O system has shown that forming $FeMoO_4$ and its compounds is possible. Finally, the ternary isotherms at 1200 °C as well as the stability diagrams for the Fe-Mo-O at 1200 °C were calculated.

3:40 PM

(ICACC-S8-005-2023) Design of Biofoams by Using different types of Biomasses

J. Zhang*[1]; G. Ngige[1]; S. Gupta[1]

1. University of North Dakota, Mechanical Engineering, USA

Biomass is an important source of raw materials for designing novel materials. In this paper, we will report latest development in the design of biofoam fabrication by using different types of biomasses (Lignin, Hemp and DDGS) reinforced with different additives. We will report the microstructure, mechanical strength, and pore structure development of these solids during the pyrolysis process at different temperatures. In addition, the wettability was also performed using contact angle analysis which showed hydrophobic behavior. Scanning electron microscope (SEM) study and X-ray Computed tomography study show that these compositions formed porous cellular structure after the pyrolysis process. With the increase of the Hemp and the DDGS content, the material became dense and therefore had a stronger mechanical strength. Biofoam possesses low cost, low density, and hydrophobic behavior. These properties make biofoams useful for different types of applications. Oil adsorption test indicated the synthesized material has the potential for oil spill removal.

4:00 PM

(ICACC-S8-006-2023) Color centers in K-Na-Cl crystals at low temperatures for remote sensing of Europa

H. Suematsu*[1]; Y. Namioka[1]; Y. Goto[1]; T. Kikuchi[3]; T. Do[1]; T. Nakayama[1]; G. Thorogood[2]

1. Nagaoka University of Technology, Extreme Energy-Density Research Institute, Japan
2. Australian Nuclear Science and Technology Organisation, Australia
3. Nagaoka University of Technology, Nuclear Technology, Japan

Europa is a satellite of Jupitar and has been known to be covered with ice and unknown non-ice component. Recently, a hypothesis of the non-ice component being of NaCl based crystals irradiated by electrons in the magnetosphere was proposed. The irradiation induces point defects in the NaCl crystal to show broad absorption peaks in the UV-visible light spectrum, which are called color centers. Under the ice, salt water exists and NaCl with small amount of K is precipitated. The absorption peak wavelength shift can estimate the K content and/or the temperature under the ice in Europa, however, there are no data on color centers in K-Na-Cl crystals at low temperatures. In this research, growth of K-Na-Cl crystals and its electron irradiations were carried out. The transparent K-Na-Cl crystals were grown by cooling from 850 °C in air. The crystals were cooled in liquid nitrogen and irradiated in a pulsed electron beam accelerator, ETIGO-III, at a peak voltage of 2 MeV, a current of 1 kA in 100 ns for one to four shots. After the irradiation, the optical transmittance was measured in liquid nitrogen. With decreasing the temperature and K content, the peak wavelength was shortened, which was explained by the shrinkage of the lattice constant.

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: Ballroom 4 (South Tower)
Session Chair: Takaaki Koyanagi, Oak Ridge National Laboratory

1:30 PM

(ICACC-S13-001-2023) New Approaches for Actinide Containing Waste Forms (Invited)

J. Amoroso*[1]

1. Savannah River National Laboratory, USA

Technologies that significantly reduce the burden of nuclear waste disposal are critical to the successful deployment of advanced reactor designs and sustainable nuclear energy production in the future. This realization has led to renewed interest in the development of novel actinide-containing materials as alternative waste forms. Hierarchical materials with multiple layers of structural modularity present a unique opportunity to develop element specific motifs. The Center for Hierarchical Waste Form Materials (CHWM) has been researching direct and indirect synthetic approaches for actinide immobilization in a variety of hierarchical materials including inorganic framework structures, tunnel structures, and metal organic frameworks. These material systems are chemically diverse and can host a variety of ion sizes and valences including actinide elements while maintaining structural integrity, a prerequisite for a radionuclide waste form. The synthesis, characterization, and performance testing of more than a dozen novel structures containing Pu, Np, Am, and U will be discussed. The results from this research provide the foundational knowledge to design tailored waste forms and materials with functionalities to facilitate the disposition of wastes arising from nuclear energy systems.

2:00 PM

(ICACC-S13-002-2023) Phosphate Glass Waste Forms for Salt Waste Stream (Invited)

M. Tang*[1]; M. Page[1]; A. Gootgeld[1]; K. Brinkman[1]; J. George[2]; B. Riley[2]; S. Stariha[3]; W. Ebert[3]

1. Clemson University, Department of Materials Science & Engineering, USA
2. Pacific Northwest National Lab, USA
3. Argonne National Lab, USA

Glass waste forms are currently used to stabilize legacy high-level waste (HLW) materials, and glass is the baseline technology for treatment of HLW that would result from reprocessing commercial used nuclear fuel (UNF). Electrochemical reprocessing is one option for the United States to reprocess UNF in advanced nuclear cycles. During reprocessing, UNF is dissolved in molten salt. Due to the low solubilities of the chloride/fluoride ions and evolution of Cl_2/F_2 gas, it is not suitable to employ the borosilicate glasses as the host for salt waste streams. Phosphate glass, especially iron phosphate glass, was investigated as waste form candidates for HLW. However, there are few studies on iron phosphate glass waste forms to immobilize salt waste streams. In this study, we intend to develop and optimize highly durable and easily processable phosphate glass waste form to immobilize the salt streams from advanced nuclear fuel cycle. Different Fe:P ratioes vs waste loading in iron phosphate glass waste forms are fabricated; various glass modifiers are added into iron phosphate glass compositions to improve chemical durability and reduce melting temperature. Further, thermal stability, chemical durability, radiation stability tests are performed on these iron phosphate glass and glass waste form samples. The preliminary result shows promising for iron phosphate glass waste form being an improvement over other candidate waste forms.

2:30 PM

(ICACC-S13-003-2023) Processing and properties of titanium beryllides ($Be_{12}Ti$) as a replacement material in future fusion applications

D. Bhardwaj*[1]; D. Sprouster[1]; B. Cheng[1]; J. Trelewicz[1]; L. Snead[1]

1. Stony Brook University, Material Science and Chemical Engineering, USA

Beryllium intermetallic (beryllides) have been of great interest among researchers, due to its ability to be used as advanced neutron multipliers. They have lower chemical reactivity, lower swelling against He, higher compatibility with structure materials, and reduced tritium retention. A plethora of literature is available for different beryllide systems (Be-Ti, Be-V, Be-Zr, etc.). The most studied system is Be-Ti. Be-Ti intermetallic stabilizes in different phases depending upon the Ti concentration and temperature. $Be_{12}Ti$ has the most desirable thermomechanical properties, and optimal Be content. Here we study the processing of pure $Be_{12}Ti$ along with mixed Be:Beryllide materials which may have superior mechanical properties. $Be_{12}Ti$ and Be: $Be_{12}Ti$ composites were synthesized in situ, using SPS, and optimizing the parameters for powder processing of Be and Ti. XRD results confirmed the presence of $Be_{12}Ti$ with trace Be, BeO and unreacted Ti. The density of the samples was >98%. Upon increasing Be concentration, complete reaction of Ti was observed, confirmed via XCT. Morphological results show an increase in Be channels at grain boundaries when Be concentration is increased from 5 to 20 vol%. Hardness value also decreases upon addition of Be. These results show direct current sintering is a potential fabrication route for these important beryllide materials.

2:50 PM

(ICACC-S13-004-2023) Application of spray drying processes for advanced uranium fuel preparation

P. Makurunje*[1]; J. T. Prabhakar[1]; S. C. Middleburgh[1]

1. Nuclear Futures Institute, Bangor University, United Kingdom

The spray drying process comprises the atomisation of a liquid stream into a chamber and contact with a hot stream of air for moisture evaporation. In the present project, wet colloidal techniques of spray drying were highlighted for advanced uranium fuel preparation, from the upstream processes of leaching uranium concentrate out of the uranium ore to forming fuel rods-ready pellets for reactor use, to the downstream process of containing spent fuel. Spray drying facilitates the drying of leached and separated concentrates from the ore at temperature ranges of 100-400 °C, either reactively or non-reactively. The technique enables agglomeration and consolidation for minimising air-borne during dry powder handling. Leveraging the droplet formation process during atomisation facilitates the shaping into fuel kernels of uranium oxides, carbides, nitrides and boride forms. The spray drying manufacturing route presents a scalable, and facile processing technique across advanced uranium fuel cycles.

Radiation Effects and Advanced Characterization

Room: Ballroom 4 (South Tower)
Session Chair: Jose Arregui-Mena, Oak Ridge National Lab

3:30 PM

(ICACC-S13-006-2023) Materials science aspects of space propulsion reactor fuel development (Invited)

S. J. Zinkle*[1]

1. University of Tennessee, USA

Nuclear energy offers the potential for dramatic improvements in space propulsion capabilities compared to conventional chemical propulsion technology. There are two major options for space nuclear propulsion (particularly considering crewed interplanetary missions): nuclear electric propulsion and nuclear thermal propulsion. Nuclear electric propulsion (NEP) involves reactor operation at moderate to high operating temperatures for extended time periods, with the reactor thermal power converted to electrical power via standard power conversion methods such as Rankine or Brayton turbine systems. Conversely, nuclear thermal propulsion (NTP) is based on the expansion of cryogenic hydrogen flowing through a nuclear reactor and exiting through a nozzle. The NTP reactor operates at extremely high temperatures (>2700 K) for relatively short time periods and short fuel burnups, e.g., <10 h for two sets of acceleration and deceleration for a crewed mission to Mars). The wide difference in operating conditions for the NEP and NTP reactor systems lead to the different fuel systems to be best suited for the different propulsion systems. This presentation will summarize the operating conditions and discuss the materials science rationale for selecting different fuel systems for NEP and NTP propulsion systems. Unresolved challenges in the development of the fuels for these two propulsion options will be summarized.

4:00 PM

(ICACC-S13-007-2023) 3D tomography imaging-based study of PYCASSO TRISO fuel particles

D. Liu*[1]; S. Knol[2]; M. Jiang[1]; E. White[1]; M. Davies[3]; A. Vreeling[2]; M. Jordan[4]; N. Tzelepi[4]; D. Goddard[4]

1. University of Bristol, United Kingdom
2. NRG, Netherlands
3. USNC, USA
4. UK National Nuclear Laboratory, United Kingdom

PYCASSO (Pyrocarbon irradiation for creep and swelling/shrinkage of objects) experiment was designed to study the impact of high temperature (900–1100 °C) fast neutron irradiation on the

thermo-mechanical properties of various coating materials in coated fuel particles. The PYCASSO fuel particles were all fabricated with surrogate kernels to exclude the influence of fuel. In the present work, three types of CEA PYCASSO (Pyrocarbon irradiation for creep and swelling/shrinkage of objects) particles including Buffer-1 (Kernel/Buffer), PyC-1 (Kernel/Buffer/PyC) and PyC-2 (Kernel/Buffer/SiC/PyC) were studied using focused ion beam (FIB) tomography and X-ray micro-tomography (XCT). Two sets of FIB-tomography data were collected on each sample and were used to investigate the porosity of the low-density buffer layer and its evolution with irradiation. An artificial intelligence-based method was developed for the FIB-tomography pore analysis. The total volume of porosity and the pore size distribution will be presented. The XCT data collected on PYCASSO particles were analyzed to relate the change of layer thickness to the neutron fluences. The PyC layer in particles with and without SiC layer showed opposing thickness changes and potential cause of this will be discussed.

4:20 PM

(ICACC-S13-008-2023) TRISO Analysis Capability in Fuel Performance Code BISON

G. Singh*[1]; W. Jiang[1]; A. Toptan[1]; S. Dhulipala[1]; Y. Che[1]; K. Gamble[1]; J. Hales[1]; S. Novascone[1]

1. Idaho National Lab, USA

TRISO fuels are robust and structurally resistant to irradiation, corrosion, oxidation and high temperature. These fuel forms are foundational to many Gen-IV high temperature gas-cooled and flouride salt-cooled reactor concepts. Evaluating the performance of these fuels under normal operating conditions and accident conditions is an essential step on the qualification path of these Gen-IV reactors. BISON is a fuel performance code that is developed at multiple national laboratories of USA and has continually expanding capability for evaluating the performance of TRISO fuel. The presentation will focus on the overview of BISON's existing capability and ongoing developement regarding TRISO fuel analysis. Results from validation cases will be discussed for demonstration purpose.

S17: Advanced Ceramic Materials and Processing for Photonics and Energy

Advanced and Nanostructured Materials for Photonics, Electronics and Sensing I

Room: Coquina Salon G (North Tower)
Session Chairs: Marina Leite, UC Davis; Nicola Pinna, Humboldt-Universität zu Berlin

1:30 PM

(ICACC-S17-002-2023) Material issues for sustainable plasmonics (Invited)

J. Plain*[1]

1. Université de technologie de Troyes, France

For more than twenty years, noble metal nanoparticles have been of first interest due to their varied and complex optical properties. Such very intense nanosource paves the way for numerous applications: controlling, manipulating and amplifying the light at the nanoscale. Nevertheless, if numerous applications have already been developed, the finite stocks of Au and Ag impose us to question some material issues. Is that possible to find abundant materials presenting similar optical properties in order to replace gold and silver? Very recently, new materials have been proposed as suitable materials to tackle emerging applications of nano-photonics, e.g. high-temperature applications, nanochemistry, sensing, or active plasmonics where gold and silver do not possess all the required properties, such as high-temperature sustainability, or catalytic activity. For these reasons, gold and silver are currently reducing their predominance in plasmonics to the benefit of other materials that constitute a new emerging branch of research. Following this burgeoning variety of optical nanomaterials, it becomes useful to conduct a comprehensive and comparative study in order to clearly establish the relative efficiencies of these new materials. I will first present all the nanofabrication techniques developed in our group to obtain nanostructures made from materials chosen because of their abundance on earth and optical properties.

2:00 PM

(ICACC-S17-003-2023) Effect of selenium integration on the electrochemical properties of Ni-hexacyanoferrate-based nanocubes: the oxygen evolution reaction (Invited)

F. Polo*[1]; E. Lushaj[1]; E. Moretti[1]; A. Vomiero[2]

1. Ca' Foscari University of Venice, Molecular Sciences and Nanosystems, Italy
2. Lulea University of Technology, Engineering Sciences & Mathematics, Sweden

In recent years, extensive efforts have been devoted to design and synthesize highly active, earth-abundant, and low-cost alternative catalysts for oxygen evolution reaction (OER). Prussian-blue analogue (PBA) materials have attracted research interests owing to their intriguing stimuli-responsive electron transfer properties. Moreover, the development of hierarchical nanostructures with controllable composition proved to be a new pathway to tune their physico-chemical properties. Unfortunately, several steps and elaborate techniques are required to obtain complex structures. Therefore, achieving both structural complexity and reliable performances is still a challenging task. In this presentation, we describe a two-step protocol to synthesize composite nanocubes, constituted of nickel hexacyanoferrate (Ni-HCF) and trigonal selenium, in which the characteristics of PBAs and chalcogenide-based materials are merged. The electronic, structural, and optical properties have been fully characterized and their performances have been tested toward OER.

2:30 PM

(ICACC-S17-004-2023) Transparent Lutetium-based, Sesquioxide Ceramic Scintillators

Y. Wang*[1]; U. Shirwadkar[1]; S. Onorato[1]; J. Nam[1]; C. Brecher[1]; K. Shah[1]; J. Glodo[1]

1. Radiation Monitoring Devices, Inc., Research, USA

Lu_2O_3 is an excellent scintillator host for gamma-ray detection due to its high density (9.4 g/cm^3), high effective atomic number (Z_{eff}=68), excellent chemical stability, and radiation hardness. Its cubic structure and isotropic optical properties allow the fabrication of fully transparent ceramic scintillators at lower temperatures and costs than their single-crystal peers. Lu_2O_3 can easily lend itself to multiple activator rare earth ions such as Yb^{3+} which has been demonstrated as a promising ultra-fast scintillator for Positron Emission Tomography (PET), High Energy Physics (HEP), or high-count rate radiography. This scintillator exhibits an ultra-fast (1.7 ns decay time) emission with a light yield of about 2,000 ph/MeV. Recently, La^{3+} doped Lu_2O_3 has been shown to have very promising properties with a higher light yield up to 20,000 ph/MeV and good energy resolution. In this study, we will discuss the development and synthesis of the transparent Lu_2O_3 ceramic with La^{3+} and/or Yb^{3+} doping. The phase structure and microstructure evolution of the Lu_2O_3 ceramics with different amounts of La^{3+} and Yb^{3+} dopants under different synthesis conditions were also explored. It is found that by adjusting the La and Yb dopant concentration, we can effectively tailor the microstructure. decay time and light yield of the Lu_2O_3:Yb, La ceramics, and makes them promising candidates for future PET applications.

3:10 PM

(ICACC-S17-005-2023) Cubic boron arsenide (c-BAs): A promising semiconductor for next generation electronics (Invited)

J. Bao*[1]

1. University of Houston, USA

Cubic boron arsenide (c-BAs) is a unique semiconductor because it possesses both high thermal conductivity and high carrier mobility, however, a lot of challenges still remain in order for c-BAs to realize its promise for next generation electronics. Besides the challenge of synthesis of large size c-BAs wafers, techniques that can rapidly characterize and screen high quality c-BAs are still lacking. In this talk, I will first describe basic properties of c-BAs, including photoluminescence and Raman spectroscopy, recent verifications of simultaneous high thermal conductivity and mobility, I will then discuss the challenges of using optical techniques to identify high quality c-BAs.

3:40 PM

(ICACC-S17-006-2023) Photonics and energy applications of advanced nanomaterials (Invited)

D. Ban*[1]

1. University of Waterloo, Electrical and Computer Engineering, Canada

Abstract: Advance nanomaterials such as perovskite materials have been widely used in photonic applications because of their facile manufacture and excellent optoelectronic performance, including high optical absorption for solar cells, high color purity and spectral stability for light-emitting diodes, large Stokes shift or small absorption-emission overlap (low reabsorption loss) for luminescent solar concentrators, to name a few. Recently, perovskites have been adopted for energy harvesting applications because of their large piezoelectric coefficients. In this talk, I will present the latest progress in perovskite research ranging from photonics to energy-related applications. The research discoveries include efficient solar cells based on monolayer perovskite bridges that enabled strong quantum dot coupling; bright, narrowband, and stable blue emitting perovskite diodes enabled by chloride insertion-immobilization; attachable luminescent solar concentrators; and high-performance nanogenerators based on perovskite materials.

4:10 PM

(ICACC-S17-007-2023) YAG-based transparent ceramics: Study of nanopowders synthesis by batch or continuous co-precipitation

F. Delaunay*[1]; R. Boulesteix[1]; A. Maitre[1]

1. IRCER, France

In this study, YAG ($Y_3Al_5O_{12}$) nanopowders have been synthetized by a reverse co-precipitation synthesis method with two designs of reactors (batch or continuous). The effects of parameters such as the concentration of metallic ions, the precipitating agent used, the temperature, the pH and the maturation time have been studied in regards to the chemical, structural and morphological features of powders. It has been shown that the temperature and the concentration of the metallic ions were able to control the precipitation kinetics of Y^{3+} and Al^{3+} and thus have a strong effect on both the stoichiometry and morphology of the powder. Also, the temperature promotes the precipitation kinetics and consequently reduces the grain size. It was also demonstrated that nanopowders obtained with the continuous reactor were more homogeneous and reproducible than those obtained by batch. Finally, the effect of the powder's morphology on their sintering ability was investigated in order to produce transparent ceramics.

4:30 PM

(ICACC-S17-008-2023) Ge-based Nanowires of Metastable Composition: Hyper-Doping and Alloy Formation

S. Barth*[1]; V. Krause[1]; R. Boeckle[2]; M. Sistani[2]

1. Goethe University Frankfurt, Germany
2. TU Wien, Institute of Solid State Electronics, Austria

This contribution will address the formation of nanowires and nanorods of metastable composition and demonstrate the significant changes in physical properties associated with their composition. All the growth studies are carried out either in solution or by gas phase techniques using the metal to be in corporate in the Ge host lattice as a growth seed. This approach allowed the formation of highly crystalline, metastable $Ge_{1-y}Ga_y$ (y~0.03%) as well as $Ge_{1-x}Sn_x$ (x=0.13-0.28) nanowires and nanorods at low temperatures. The materials have been characterized by different analytical methods including TEM, EDX, Raman spectroscopy as well as XRD. Generally, a homogeneous incorporation of unusually high contents of Sn and Ga in the Ge lattice has been observed. The high incorporation efficiency also alters the physical properties significantly. While hyperdoped $Ge_{0.97}Ga_{0.03}$ shows quasi-metallic behavior in temperature dependent transfer characteristics, $Ge_{0.81}Sn_{0.19}$ reveals still semiconducting behavior in equivalent experiments. Moreover, the formation of $Ge_{1-x}Sn_x$ with x>0.09 causes the transformation to a direct bandgap material, while the solid solubility limit according to the binary phase diagram (~ 1 at% Sn in Ge) has to be overcome. Moreover, molecule-based approaches also allow to form other binary and ternary compositions of interest for electronics and optics.

S18: Ultra-High Temperature Ceramics

Compositionally Complex UHTCs I

Room: Coquina Salon A (North Tower)
Session Chair: William Fahrenholtz, Missouri University of Science & Technology

1:50 PM

(ICACC-S18-001-2023) High Entropy UHTCs: Are they better than conventional UHTCs? (Invited)

W. Fahrenholtz*[1]

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

High entropy ultra-high temperature ceramics have become a hot research topic. This presentation will discuss several controversial aspects of HE-UHTCs. First, the use of the term "high entropy" will be discussed in the context of whether it is appropriate to use for these materials. The thermodynamic stability of HE-UHTCs will also be analyzed to determine whether the high entropy effect can possibly increase melting temperature. Next, oxidation behavior will be discussed in the context of its potential effect on scale formation and stability. Finally, the potential effects of the presence of multiple transition metals in solid solution on mechanical response will be considered. The proposal will draw conclusions about the benefits, or lack of benefits, of incorporating multiple transition metals into a single ceramic phase.

2:20 PM

(ICACC-S18-002-2023) High-temperature oxidation and ablation behavior of high-entropy borides

A. C. Feltrin*[1]; F. Akhtar[1]

1. Luleå University of Technology, Materials Engineering, Sweden

Ultra-high temperature ceramics (UHTCs) are used under severe chemical and thermal environments for extended times due to their chemical and physical stability at high temperatures. High-entropy UHTC borides are based on the entropy stabilization of multicomponent borides offering unique compositions, microstructures, and properties. We report oxidation properties at 1000 and 1500°C and ablation properties under oxyacetylene torch of multicomponent metastable and high-entropy borides, synthesized from HfB_2, ZrB_2, VB_2, TiB_2. The metastable boride showed a dual-phase boride (DPB) microstructure with Hf-Zr-rich and a V-Ti-rich phases, whereas the high entropy boride showed a chemically homogeneous single-phase high-entropy boride (HEB) $(Hf_{0.25}Zr_{0.25}V_{0.25}Ti_{0.25})B_2$ phase. The oxidation behavior of these borides showed para-linear behavior at 1000°C and the oxidation mechanism was controlled by oxygen diffusion into B_2O_3 protective layer and linear behavior at 1500°C, where the metallic oxide layer formed offered a poor barrier to oxidation. The mass and linear ablation rates were -0.18 mg/s and -0.46 μm/s for DPB and -0.14mg/s and -0.33μm/s for HEB, respectively, and mechanical denudation was the main ablation mechanism. The ablation and oxidation mechanisms were the same for DPB and HEB, however, the oxidation and ablation rates were lower for the HEB in all cases, attributable to the high-entropy effect.

2:40 PM

(ICACC-S18-003-2023) Interdiffusion of High-Entropy Borides and Carbides

S. M. Smith*[1]; W. Fahrenholtz[1]; G. Hilmas[1]; T. Huang[2]

1. Missouri University of Science & Technology, Dept. of Materials Science and Engineering, USA
2. Honeywell Federal Manufacturing and Technologies, USA

Dual phase high-entropy boride-carbide ceramics can be produced using various synthesis methods. Regardless of the synthesis method, the transition metals in the phases can segregate preferentially to either the boride or carbide phase. Typically, Ti segregates to the boride phase and Ta segregates to the carbide phase. While some reasons for this segregation have been proposed, no studies have investigated the diffusion behavior of the metals in these environments. The present research aims to characterize the behavior of the metals in dual phase high-entropy ceramics through heat treatments of high-entropy boride-carbide diffusion couples at temperatures ranging from 1900-2100°C. This presentation will focus on microstructural characterization of the boride-carbide interface and will identify trends in metal behavior across the interface. Honeywell Federal Manufacturing & Technologies, LLC operates the Kansas City National Security Campus for the United States Department of Energy/ National Nuclear Security Administration under Contract Number DE-NA0002839

Novel Processing Methods I

Room: Coquina Salon A (North Tower)
Session Chairs: Lisa Rueschhoff, Air Force Research Lab; Diletta Sciti, ISTEC-CNR

3:20 PM

(ICACC-S18-004-2023) Novel processing and Compositions of fiber-reinforced UHTCs (Invited)

L. M. Rueschhoff*[1]; B. Lam[1]; C. Kassner[1]; C. Wyckoff[1]; K. Acord[1]; Z. D. Apostolov[1]; M. Cinibulk[1]

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

While UHTCs are re-gaining traction as materials of interest for use in harsh environments encountered by next generation aerospace systems, implementation is still limited by brittle monolithic behavior and difficulty in processing into complex shapes. Fiber reinforcement can be used to improve on the limiting mechanical performance in the form of either continuous or discontinuous (i.e. chopped) fiber to yield UHTC ceramic matrix composites (UHTCMCs). Here, we present the use of the additive manufacturing technique of direct ink writing (DIW) with newly developed chopped carbon fiber reinforced zirconium diboride inks. Along with the development of similar reinforced silicon carbide inks, dual in-line mixing is used to create compositions with functional compositional gradients. High densities (>95%) are achieved via pressureless sintering, making this a promising 1-step processing route towards chopped fiber UHTCMCs. Microstructural, mechanical and thermal oxidative performance will be presented for structures of varying compositions and fiber loadings. In addition, brief mention will be given to highlight other work in the team on conventional processing of continuous fiber reinforced UHTCs as a more robust and industry relevant option towards similar compositions.

3:50 PM

(ICACC-S18-005-2023) Layered UHTCMCs with variable matrix type to enhance lightness and oxidation resistance (Invited)

D. Sciti*[1]; A. Vinci[2]; M. Mor[1]; L. Zoli[3]

1. ISTEC-CNR, Italy
2. ISTEC-CNR, DSCTM, Italy
3. CNR ISTEC, ISTEC, Italy

Ceramic matrix composites (CMC) with an ultra-high temperature ceramic (UTHC) matrix are very promising materials for aerospace and defense applications due to high oxidation and ablation resistance coupled with elevated temperature mechanical properties. The target of this work was to maximize lightness in the material core and ablation resistance in the external surface through different approaches. Using continuous Cf preforms, composites with a SiC-rich core coupled with UHTC-rich surface either containing ZrB_2 or HfB_2, were produced by slurry impregnation and sintering. In parallel, a new technique was developed allowing the fabrication of thin (~ 100 μm), sheets made of randomly distributed short carbon fibres suitable for the manufacturing of layered structures consolidated by sintering. A wide range of compositions, in terms of matrix type or fibre volumetric content was possible. Planar graded structures with crack free, sharp interfaces were manufactured overlapping sheets with fixed carbon fibre content and different matrix type from the interior to the exterior. Otherwise, layered structures were obtained overlapping sheets with fixed matrix type and variable fibre composition. Mechanical properties, short oxidation tests up to 1650°C in conventional air furnace and arc-jet tests over 2000°C were carried out for selected materials.

4:20 PM

(ICACC-S18-006-2023) $Ti_3C_2T_x$ MXene-Zirconium Diboride Based Spark Plasma Sintered Ultra-High Temperature Ceramic Composites

S. Nemani*[1]; Y. Im[1]; B. Anasori[1]

1. Indiana University--Purdue University, Mechanical Engineering, USA

$Ti_3C_2T_x$ MXene is a two-dimensional (2D), solution-processable, high aspect ratio, with reported stiffness (333 GPa) highest among solution-processable 2D materials. In this project, we have investigated a one-pot, surfactant-free, aqueous mixing method to develop homogeneous ZrB_2-Ti_3C_2 MXene green bodies and evaluated their sintering behavior. We present the phase transformation of Ti_3C_2 MXene to TiC_y at the grain boundaries, the interface interactions between the ZrB_2-TiC_y grains and their densification mechanism. A nominal relative density of ~96% is achieved when 0.5 wt.% Ti_3C_2 MXene in ZrB_2 was sintered at 1900C with 50 MPa pressure in inert atmospheres. We discuss the intermediate phase formation and evolution and their effect on the mechanical properties of the resulting UHTCs. This study lays the groundwork for 2D MXenes as compatible materials for UHTC applications that can be used as precursors for carbides in ultra-high temperature applications such as hypersonics, nuclear, and extraterrestrial travel.

4:40 PM

(ICACC-S18-007-2023) Fabrication of W-ZrC Composites by Using ZrO_2 and WC

S. Kim*[1]; Y. Han[2]; S. Ryu[1]; S. Lee[2]

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

W-ZrC is one of the most commonly investigated UHTCs(Ultra-high Temperature Ceramics) due to its hardness and high melting temperature. While W is a stable refractory metal with high Young's modulus, flexural strength, fracture toughness and a high melting temperature over 3400°C, ZrC also has a high melting temperature similar to that of W, great hardness as well as good resistance to wear or corrosion. Mixture of ZrC with a refractory metal such as W, requires high sintering temperature and pressure, so a variety of reaction methods have been explored. In this study, W-ZrC composites are fabricated by hot pressing as well as vacuum sintering by using WC and ZrO_2 starting materials. With different starting composition and fabricating condition, the phase formation behavior and thermal properties are examined. The relation between phase formation and those properties is discussed as well.

5:00 PM

(ICACC-S18-008-2023) Joining of HfB_2-ZrB_2 based composites with and without Ni filler layer

S. Bajpai*[1]; A. Bhadauria[1]; T. Venkateswaran[2]; K. Balani[1]

1. Indian Institute of Technology Kanpur, Materials Science and Engineering, India
2. Vikram Sarabhai Space Centre, India

Hafnium and Zirconium di-borides are considered as viable choices for ultra-high temperature applications, owing to their high melting point (3380 °C and 3245 °C, respectively), high thermal conductivity, and chemical inertness. Difficulties associated with the manufacturing of large and complex shaped parts mandates the joining of diborides based UHTCs for use in extreme thermal conditions. Herein, spark plasma sintering is utilized to join pre-sintered HfB_2-ZrB_2-SiC-B_4C-CNT (HZSBC) based composites with and without Ni interlayer at 1100 °C and 1800 °C, respectively. In HZSBC-Ni-HZSBC joint, microstructural analysis has elicited formation of Ni diffused reaction zone (150-200 μm) and unaffected HZSBC composite resulting in overall reduction of ~18 % in hardness and ~14% in elastic modulus from parent UHTC to interaction zone. However, seamless interface and formation of homogeneous microstructure in HZSBC-HZSBC joint has resulted in uniform hardness and elastic modulus (~22 GPa and 397 GPa), along with higher bending strength (~341 MPa) and shear strength (~80 MPa). HZSBC-HZSBC joints with a thinner oxide layer (relative to those with Ni-interlayer) and the presence of (Zr, Hf)O2 crystals at the surface exhibit improved oxidation resistance at 1500 °C, making them more appropriate for applications involving complicated shapes and severe conditions.

S19: Molecular-level Processing and Chemical Engineering of Functional Materials

Chemical Approaches to Energy-related Functional Materials

Room: Ballroom 3 (South Tower)
Session Chair: Emanuel Ionescu, Technical University Darmstadt

1:30 PM

(ICACC-S19-001-2023) Atomic Scale Control Strategies to Boost Catalytic Activities toward High Performance Fuel Cells (Invited)

J. Baek[1]; M. Jung[1]; K. Kim[1]; S. Kwon*[1]

1. Pusan National University, School of Materials Science and Engineering, Republic of Korea

The performance of fuel cells strongly depends on the controlled size, dispersion, density, and surface properties of catalytic nanoparticles on carbon supports. Therefore, a proper selection of synthesis method and design strategy for catalytic nanoparticles is very important to obtain a high performance fuel cells. One of the important strategy for engineering such catalytic nanoparticles is to functionalize the surface of them for obtaining improve performance and stability. And, atomic layer deposition (ALD) has its inherent merits to precisely control the textual properties as well as surface properties of catalytic nanoparticles in atomic scale. Herein, we will introduce an atomic scale control strategy for realizing a high-performance catalytic nanoparticles for fuel cells using ALD method. The favorable surface characteristics of the functionalized supporting materials for nucleation produced catalytic nanoparticles with an increased uniformity and density and a narrow size range, which led to a higher electrochemical surface area than that of commercial Pt/C. Also, an effective matching of the supporting material and an optimization of the membrane electrode assembly showed many beneficial points in terms of the performance and stability. These merits can mark a step toward a modern high-performing catalytic nanoparticles for enhanced fuel cell technology.

2:00 PM

(ICACC-S19-002-2023) On the performance of polymer derived ceramic nano-composites in sodium and potassium-ion half cells (Invited)

S. Dey[2]; S. Mujib[3]; G. Singh*[1]

1. Kansas State University, Mechanical and Nuclear Engineering Dept., USA
2. Kansas State University, Mechanical Engineering, USA
3. Kansas State University, Mechanical & Nuclear Engineering, USA

Silicon containing polymer-derived ceramics (PDC) fibers, because of their intrinsic thermal stability, had enormous commercial success in the form of high-damage tolerant ceramic matrix composites. Another interesting aspect of PDCs is their amorphous microstructure which imparts them with electrochemical properties suitable for battery electrodes—nanodomains of various constituents in PDCs such as SiC, SiO or the disordered carbon phase renders PDCs with ability to cycle alkali metal-ions at room temperature without degradation or chemical corrosion for prolonged times. Here, carbon rich SiOC composite fibers were fabricated via electrospinning and pyrolysis of cyclosiloxane precursor loaded with a variety of high capacity nanofillers such as 2-D nanosheets and nanoparticles of transition metal sulfides. Investigations on structural and compositional development of the fibers were mainly conducted via Raman spectroscopy, Fourier-Transform InfraRed spectroscopy, Scanning Electron Microscopy, and X-ray Photoelectron Spectroscopy to determine free carbon content, crosslinking, pyrolysis behavior and morphology of the fibers. Performance as electrode material in sodium and potassium ion rechargeable metal ion batteries will be discussed.

2:30 PM

(ICACC-S19-003-2023) Electroactive low dimensional materials for Water-Splitting Electrocatalysts (Invited)

M. Siaj*[1]

1. University of Quebec, Montreal, Faculty of Science, Canada

This presentation focuses on multifunctional 2D and 3D materials and structure/property relationships to control graphene-like and transition metal dichalcogenides nanostructure to be used as three-dimensional hybrid electrodes for hydrogen evolution reaction, sensors and supercapacitors fabrication. We developed a platinum-free electrocatalyst (3D $MoSe_2$ rose-like structure) for the hydrogen evolution reaction (HER) as a first step for large-scale production and application of water splitting devices. The hybrid membrane exhibits excellent performance for HER. In the same endeavor, we showed a novel strategy to synthesize vertically aligned porous $MoSe_2$. This designed architecture based on an oriented vertical structure possesses fully exposed active edges and open structures for fast ion/electron transfer, leads to remarkable HER activity with a low onset potential and a 3D binder-free without the need for a transferring step or a conductive additive to build the electrodes. This work opened a new class of nanomaterials in my lab, in which we start the fabrication of photoanodes via in-situ reduction–graphitization approach toward efficient solar hydrogen evolution. Recent emphasis is on controllable synthesis and development of one-dimensional oriented CdS-based nanoarray photoanodes for efficient solar water splitting / solar hydrogen.

Solution-processing of Functional Oxides

Room: Ballroom 3 (South Tower)
Session Chair: Eva Hemmer, University of Ottawa

3:20 PM

(ICACC-S19-004-2023) Synthesis of faceted metal oxides with unique properties in catalysis and carbon capture (Invited)

R. M. Richards*[1]

1. Colorado School of Mines, Chemistry, USA

The preparation of nanoscale materials is one of the most exciting areas of modern science and is at the forefront of the quest for a sustainable future. The Richards' group is working on new synthetic methods to control the size, shape and composition of nanoscale materials and applying them in systems integral to alternative energy technologies, pharmaceuticals, biomass upgrading, batteries, petrochemicals and environmental cleanup. The ability to manipulate earth abundant metal oxides presents an important potential technology to develop sustainable materials with novel properties. These materials are of interest due to the coordination environments that can be achieved and to the high degree of control over properties that can result from tailoring the exposed facets and from mixing metal oxides. The initial synthesis of MgO and NiO with (111) facets as the primary surface has been followed by recent work utilizing a multiscale characterization platform to discover the underlying phenomena associated with the electrolytic properties of NiO(111) for Li ion batteries and electrochromics. Joint experimental-theoretical work with collaborators, has led to a report unravelling the potential of (111) polar surfaces for carbon capture. Most recently, in-situ microscopy studies have revealed insights into the NiO active sites and the effects of Fe doping for electrolysis.

3:50 PM

(ICACC-S19-005-2023) Tale of Two Bismuth Alkylthiolate Precursors' Bifurcating Paths in Chemical Vapor Deposition

T. Fischer*[1]; U. Atamtürk[1]; S. Mathur[1]

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

Chemical Vapor Deposition (CVD) is a versatile gas phase processing technique for the deposition of homogeneous thin films used in various applications ranging from encapsulation for microelectronics, functional layers in sensors, as well as hard coatings for tools. Therefore, well defined molecular precursors are essential for achieving stable process conditions and reproducible coatings. We report on the application of two bismuth alkylthiolates, $Bi(SR)_3$, differing only with respect to the alkyl substituents (R = $-Bu^t$; $-Pr^i$) as single-source precursors in the CVD processing for both Bi_2S_3 and Bi. Despite similar structural chemistry and chemical composition, the minor differences in the polarity of the bonds in Bi-S-C units result in strikingly different results in their low-temperature (250°C) decomposition behaviour. Whereas the CVD of $Bi(SBu^t)_3$ produced single-crystalline Bi_2S_3, elemental Bi was obtained from $Bi(SPr^i)_3$. The structure and composition of resulting CVD deposits were analyzed using XRD, XPS, SEM, TEM, SAED and Raman spectroscopy.

4:10 PM

(ICACC-S19-006-2023) Synthesis of amorphous and crystalline oxides from $MAl_3(O^iPr)_{12}$ (M = Ln, Cr) precursors (Invited)

G. Westin*[1]; M. Ek[1]

1. Uppsala University, Sweden

Alumina is of high importance for a wide range of application due to its combination of extreme properties including low cost, low toxicity, wide range of optical transparency, high redox stability, and hardness and temperature resistance. A drawback in some mechanical applications is its brittleness, which may be improved by a second oxide as a solution or second phase in nano-composite. Doping with optically active ions such as lanthanides or transition metals is of interest for e.g. high temperature sensing applications. The strong bonds formed in alumina's make synthesis and sintering based on migration of atoms difficult. This also lead to a dearth in detailed knowledge of the mechanisms that lead to the final oxide. This makes advanced, molecular based synthesis of interest as it allows for atomic scale mixing of the component oxides. Here synthesis of $Cr:Al_2O_3$ and $Ln:Al_2O_3$ (Ln = Y and Eu) using the heterobimetallic alkoxides $CrAl_3(OiPr)_{12}$ and $(Y/Eu)Al_3(OiPr)_{12}$ are reported. The phase development from all-inorganic gels, via amorphous, homogenous oxides to the phase separated nano-crystalline products is discussed based on TG-DSC, IR and Raman spectroscopy, XRD, SEM, and TEM studies.

4:40 PM

(ICACC-S19-007-2023) Flexible and Scalable Thermoelectric Device and its Application as a Self-Sufficient Power Supply for Wearable Electronic Devices (Invited)

D. Madan*[1]

1. University of Maryland Baltimore County, Mechanical Engineering, USA

This work demonstrates a low-energy input manufacturing method for controlling the micro and nanostructures of p-type and n-type composites by varying the film thickness and understanding its effect on thermoelectric properties. The lower number of grains, grain-binder interfaces, and micro-scale defects in the thinner films (170 μm) manifested higher electrical conductivities than in the thicker films (300 μm). The high electrical conductivities of p-type thin films (660 S/cm) and n-type thin films (286 S/cm) achieved best ZT of 0.89 and 0.50 respectively. The best p and n inks were stencil printed to manufacture the 6-couple thermoelectric devices. A power output of 357.6 μW and power density of 5.0 was achieved at a temperature difference of 40 K. The device was successfully bent for 1000 cycles at a radius of 3 cm and a 4.3% increase in resistance was observed, demonstrating its high flexibility. Moreover, the integration of TEG and step-up voltage circuits demonstrated the capacitor charging (upto 3 V) and discharging. These demonstrations confirm the TEG device's great potential to be used as a self-sufficient battery for wearable devices and health monitoring sensors

5:10 PM

(ICACC-S19-008-2023) Microwave-assisted synthesis of lanthanide-based nanoparticles for applications from biomedicine to printing (Invited)

E. Hemmer*[1]

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

Based on their optical properties, lanthanide-based compounds have been suggested for applications including the fields of biomedicine, optoelectronics, and solar energy conversion. Our favourite nanomaterials are lanthanide-based fluorides ($MLnF_4$), and our research addresses challenges in their synthesis as well as the establishment of structure-property relationships. The growing attention toward such optically active materials has prompted the development of novel synthesis methods for a more reliable and efficient access to these systems. In this regard, microwave-assisted approaches provide unique advantages over traditional solvothermal methods reliant on convectional heating: namely, significantly shorter reaction durations, more rigid reaction conditions, and thus a higher degree of reproducibility. The developed approach allows to control the material's crystalline phase and doping of various Ln^{3+} ions into core/shell architectures. Additional surface modification with biopolymers renders the nanomaterials dispersible in various solvents and allows for their assembly into multipurpose micro-carriers. The resultant emission colour tuneable upconverting nanoparticles are promising candidates for versatile applications, ranging from multiplexed imaging and light-induced therapy to inks for printing of micropatterns for optoelectronic devices.

Tuesday, January 24, 2023

Emerging Materials and Sustainable Manufacturing Technologies in a Global Landscape: Symposium in Honor of Dr. Tatsuki Ohji

Tatsuki Ohji Honorary Symposium I

Room: Coquina Salon D (North Tower)
Session Chairs: Mrityunjay Singh, Ohio Aerospace Institute; Stuart Hampshire, University of Limerick

8:40 AM

(ICACC-HS-001-2023) Recent progress of porous ceramics to achieve sustainable development goals (Invited)

M. Fukushima[1]; T. Ohji*[1]

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

Sustainable development goals (SDGs) including 17 goals and 169 targets have been adopted unanimously in Sustainable Development Summit held at the United Nations (UN) Headquarters in New York in September 2015. The use of macro-porous ceramics is definitely one of the solutions to achieve the goals, because that can cover almost all technological issues. In order to achieve the goals, human beings must effectively utilize, recycle and reuse global resources such as 1) water from ocean, river and lake, 2) land and soil for livestocks and agricultural crops grown by the water, 3) wood and plant grown on the land and soil, and 4) fossil fuel from those petrified plants and animals, because overpopulation is getting more serious problem in this twenty-first century. Ceramic materials, especially oxides are natural abundances given, and macro-porous ceramics are the key reliable components in numerous functional and structural devices described above, in which strategically engineered porosity can become one of most important technologies to accomplish sustainable developments. In this presentation, pore morphologies, volume fraction and size will be discussed in terms of the necessary features required to achieve the ideal sustainable society for the intended applications.

9:00 AM

(ICACC-HS-002-2023) Review of Mechanical Properties of Porous Silicon Nitride Ceramics with Controlled Microstructures (Invited)

S. Hampshire*[1]

1. University of Limerick, The Bernal Institute, Ireland

This paper reviews investigations of porous silicon nitrides with volume fraction porosities up to 0.5. These materials can be fabricated by a range of techniques including partial hot-pressing, partial sintering or by inclusion of fugitive additives such as starches and other organics. Irrespective of the porosity, samples have been shown to exhibit almost the same microstructural features including grain size, grain aspect ratio, and pore size. Porosity dependences of Young's modulus, flexural strength, and fracture resistance have been investigated and as expected, these properties decrease with increasing porosity. For partial hot-pressed silicon nitrides, because of the high aspect ratio β-Si_3N_4 grains, decreases of flexural strength and fracture toughness have been shown to be moderate compared with the much greater decrease of Young's modulus. Thus, the strain tolerance (fracture strength/Young's modulus) increases with increasing porosity. For silicon nitrides containing large pores with volume fractions of ~0 to 0.25, values of relative fracture energy in the range (1-P) = 0.85 to 1 have been shown to be higher by a factor of up to 1.2 than for the fully dense reference silicon nitride.

9:30 AM

(ICACC-HS-003-2023) How Computational Thermodynamics, Lattice Engineering, and Data Science Work Together to Design Commensurate Martensitic Transformations in Zirconia (Invited)

E. Pang[1]; G. B. Olson[1]; C. A. Schuh*[1]

1. Massachusetts Institute of Technology, Department of Materials Science and Engineering, USA

The martensitic transformation in zirconia is very widely studied, most usually for the damage it causes upon cooling from the high temperature tetragonal phase to the low temperature monoclinic one. That transformation can be made "gentler" through lattice engineering, i.e., doping to change the lattice parameters such that the transformation occurs in a more crystallographically commensurate manner. However, it is not clear what dopants should be added, in which proportions, to achieve such commensurate transformation. Computational thermodynamics gives insight on the solubility limits of various alloying options, as well as their effects on the transformation temperature, but not the lattice parameters in the two phases of the martensitic transformation. Accordingly, in this work we augment a process of computational materials design with elements of data science that allow us to cover previously unexplored regions of composition space with reasonable predictions of phase crystallography. The result is a new suite of zirconia alloys with near ideal commensurability of the transformation, and a record low transformation hysteresis.

10:20 AM

(ICACC-HS-004-2023) Silicon carbide ceramics with improved specific stiffness (Invited)

Y. Kim*[1]; G. Kim[1]; S. Yong[2]; W. Jung[2]

1. University of Seoul, Dept. of Materials Science & Engineering, Republic of Korea
2. Agency for Defense Development, Republic of Korea

The addition of B_4C was very effective for improving the specific stiffness of SiC ceramics because (1) the mass density of B_4C is lower than that of SiC, (2) its Young's modulus is higher than that of SiC, and (3) B_4C is an effective additive for sintering SiC ceramics. An increase in the B_4C content from 0.7 wt% to 25 wt% leads to an increase in the specific stiffness from ~142 x 10^6 m^2s^{-2} to ~153 x 10^6 m^2s^{-2}, due to the increase in the Young's modulus from ~454 GPa to ~460 GPa and the decrease in the sintered density from 3.204 gcm^{-3} to 3.010 gcm^{-3}. The strength of the SiC ceramics was maximal with the incorporation of 10 wt% B_4C (755 MPa), and the thermal conductivity decreased linearly from ~183 to ~81 $Wm^{-1}K^{-1}$ when the B_4C content was increased from 0.7 to 30 wt%. The flexural strength, thermal conductivity, and specific stiffness of the developed SiC ceramic containing 25 wt% B_4C were ~690 MPa, ~95 $Wm^{-1}K^{-1}$, and ~153 x 10^6 m^2s^{-2}, respectively.

10:50 AM

(ICACC-HS-005-2023) Ultra-high Creep Resistant SiC Ceramics (Invited)

P. Sajgalik*[1]

1. Institute of Inorganic Chemistry, Slovak Academy of Sciences, Ceramic Department, Slovakia

The additive-free β/α silicon carbide ceramics prepared by rapid hot pressing was crept in vacuum at temperatures of 1500 °C to 1750 °C and compressive loads of 200 MPa to 400 MPa. The results showed that this way prepared ceramics had the lowest creep rate reported in the literature. The observed strain rates increased from 2.5×10^{-9} s^{-1} at 1500 °C and a load of 275 MPa to 1.05×10^{-7} s^{-1} at 1750 °C and a highest load of 400 MPa. The average creep activation energy taken from all creep tests was 315 ± 20 $kJ.mol^{-1}$, and the stress exponent was 2.22 ± 0.17. The suggested creep mechanism is GB sliding accommodated by GB diffusion and β-α SiC phase transformation.

11:20 AM

(ICACC-HS-006-2023) Nanocarbon added silicon nitrides (Invited)

C. Balazsi*[1]; K. Balazsi[2]

1. ELKH Centre for Energy Research, Hungary
2. Centre for Energy Research HAS, Thin Film Physics, Hungary

Silicon nitride (Si3N4) based ceramics are well-known as low density materials with high strength and toughness. Silicon nitride, known as a typical dielectric material, is an ideal candidate for several structural applications, even at high temperatures. The addition of graphene or carbon nanotubes to silicon nitride to create ceramic nanocomposites gives rise to promising applications in a wide range of fields such as electronics, biomedical aids, membranes, flexible wearable sensors and actuators. The presentation shows how the use of different reinforcing phases and sintering methods affects microstructure and as a result, mechanical properties, electrical conductivity and friction coefficients of the final silicon nitride nanocomposites.

11:50 AM

(ICACC-HS-007-2023) Strength and plastic deformation of Si_3N_4 at mesoscale measured using microcantilever specimens (Invited)

J. Tatami*[1]; M. Tanabe[1]; T. Ohji[2]; H. Nakano[3]; M. Iijima[1]; T. Takahashi[4]; T. Yahagi[4]

1. Yokohama National University, Japan
2. National Institute of Advanced Industrial Science and Technology (AIST), Japan
3. Toyohashi University of Technology, Japan
4. Kanagawa Academy of Science and Technology, Japan

Information on the mechanical properties of crystal particles is essential for understanding the mechanical function of Si_3N_4 ceramics. In this study, the mechanical properties of Si_3N_4 at the mesoscale were measured by bending microcantilever beam specimens fabricated in actual Si_3N_4 ceramics in a focused ion beam fabrication system. The load applied to the specimen increased linearly with increasing displacement, then the load-displacement relationship became nonlinear, and finally the specimen failed. Young's modulus measured from the linear region depended on crystallographic orientation. The flexural strength was extremely high and, like Young's modulus, was dependent on crystallographic orientation. In particular, the values were higher for microcantilever specimens whose longitudinal direction was closer to the c-axis. The yield stress, which is the nonlinear starting point, also showed crystallographic orientation dependence. TEM observation showed that high density of dislocations were generated in the region where high tensile stress was applied. This indicates that the plastic deformation of Si_3N_4 is caused by dislocations in the main slip system.

5th Pacific Rim Engineered Ceramics Summit

5th Pacific Rim Engineered Ceramics Summit I

Room: Coquina Salon B (North Tower)
Session Chair: Jan Seidel, UNSW Sydney

9:00 AM

(ICACC-PACRIM-001-2023) Characterizations of TiO_2-Silicate Glass Ceramic Composites Fabricated by Microwave Heating Technique (Invited)

N. Triamnak*[1]; S. Phupaichitkun[1]; S. Lapnookawow[1]; N. Wongdamnern[2]; T. Sareein[3]

1. Silpakorn University, Materials science and Engineering, Thailand
2. Rajamangala University of Technology Suvarnabhumi, Faculty of Science and Technology, Thailand
3. Rajamangala University of Technology Phra Nakhon, Thailand

Dielectric ceramics have been engineered and developed for decades to expand electronic applications. Enhancement of dielectric constant and dielectric breakdown strength are main investigations as well as the reduction of dielectric loss. Glass materials exhibit great dielectric breakdown strength because of zero grain boundary. Doped titanium dioxides (TiO_2) have been carried out as the giant dielectric ceramics, recently, due to their extrinsic defect pairs. Hence, in this study, the co-doped TiO_2, $Ti_{0.98}(Ni_{0.01}, Nb_{0.01})O_2$ was used as ceramics phase in the silicate glass ceramics i0n order to improve the dielectric properties. Microwave heating technique was used to fabricate samples in order to reduce active time of diffusion. X-ray diffraction results illustrated the crystalline phase existence as well as Raman spectroscopy information. Scanning electron microscope illustrated the existence of both crystalline and amorphous phases. UV-Vis spectroscopy revealed bandgap shift with the increase of $Ti_{0.98}(Ni_{0.01}, Nb_{0.01})O_2$ concentration. In addition, dielectric constant increased and dielectric loss decreased with the increase of $Ti_{0.98}(Ni_{0.01}, Nb_{0.01})O_2$ concentration.

9:30 AM

(ICACC-PACRIM-002-2023) Autonomous resonance tuning mechanism for environmental adaptive piezoelectric energy harvesting (Invited)

H. Song*[1]

1. Korea Institute of Science and Technology, Electronic Materials Research Center, Republic of Korea

Internet of Things (IoT) will play a variety of roles in a wide range of applications. The biggest obstacle to the expansion of IoTs is that they are not free from power sources. Energy harvesting technology can provide the most feasible solution for this problem. The piezoelectric method has been the most extensively investigated for energy harvesting. To maximize the power generated from vibrations, the energy harvester must operate at resonance. However, energy harvester typically exhibits narrowband natural frequencies and thus it is difficult to couple with broadband vibrations. For this reason, operating in resonance at actual environments with various frequencies is one of the biggest issues in energy harvesting research. In this talk, I will introduce novel strategies to achieve broadband operation through autonomous resonance tuning (ART) mechanisms that can automatically adjust their resonance frequencies by adapting to environments. First of all, I will introduce an idea for the ART mechanism employing the moving proof mass on a doubly clamped beam structure. I will discuss how the moving mass can provide the ART function both theoretically and experimentally. Second, the ART mechanism using the adaptive clamping system will be introduced and discussed its operation mechanism and experimental validation in detail.

5th Pacific Rim Engineered Ceramics Summit II

Room: Coquina Salon B (North Tower)

Session Chairs: Hyun-Cheol Song, Korea Institute of Science and Technology; Narit Triamnak, Silpakorn University

10:20 AM

(ICACC-PACRIM-003-2023) Dielectric breakdown strength for commercial and prepared silicon nitride substrate (Invited)

Y. Nakashima*[1]; Y. Zhou[1]; K. Hirao[1]; M. Fukushima[1]

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

Silicon nitride (Si_3N_4) ceramics have attracted increasing attention as insulated heat-dissipating substrates for power modules due to their high thermal conductivity and mechanical strength. However, there are very few reports on their dielectric breakdown strength (DBS), which was only evaluated for the substrates with thicknesses between 250 and 640μm, though thinner substrates are preferable for attaining better performances of the module. In this work, DBS of commercial Si_3N_4 substrates with thicknesses ranging from 285 μm to 15 μm was evaluated for the first time, and the value of prepared silicon nitride substrates with thicknesses ranging from 300 μm to 50 μm was also evaluated. Average DBS of commercial one increased from 36.38 kV/mm to 103.80 kV/mm with decreasing thickness from 285 to 15 μm. It should be noted that the silicon nitride specimen had very high DBS of 1.5 kV even with a thickness as small as 15 μm. When Si_3N_4 ceramics were prepared by longer holding times, the DBS sharply decreased at the thin substrate, because some large-elongated grains might span the substrate thickness-wise throughout. It was inferred that the interface between β-Si_3N_4 grains and grain boundary phase/intergranular glassy films might be a path of the dielectric breakdown.

10:40 AM

(ICACC-PACRIM-004-2023) Functional topological defects in ferroelectric and multiferroic materials (Invited)

J. Seidel*[1]

1. UNSW Sydney, School of Materials Science and Engineering, Australia

Topological structures in ferroic functional materials, such as domain walls and skyrmions, currently see increased attention due to their intriguing properties and application potential in nanoelectronics. I will discuss some of our recent results on ferroelastic, ferroelectric and multiferroic materials systems using scanning probe microscopy as the main investigative tool, which is combined with insight from electron microscopy and ab-initio theory, and discuss future prospects of this quickly evolving research field.

11:10 AM

(ICACC-PACRIM-005-2023) Creation of predictive models of 5d-4f emission energy of Ce^{3+} in garnet-type oxides based on first-principles calculations and machine learning (Invited)

K. Ogasawara*[1]

1. Kwansei Gakuin University, Department of Chemistry, Japan

The prediction of 5d-4f emission energies of Ce^{3+} in crystals is important for theoretical design of novel phosphors. However direct prediction based only on first-principles calculations is quite difficult due to the structural relaxation in the excited state corresponding to the initial state of the emission. In order to establish an efficient method, a combination of first-principles calculations and machine learning is useful. In this work, garnet type-oxides were considered since there are three types of cation sites and the local structure around the impurity Ce^{3+} ion can be modified widely by cation substitution. Considering the structural parameters and the electronic parameters obtained by first-principles molecular orbital (MO) calculations using simple CeO_8 clusters as attributes, a predictive model of the 5d-4f emission energy of Ce^{3+} in garnet-type oxides was obtained by machine learning. Moreover, 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 predictive models of the electronic parameters were created by machine learning. As a result, an efficient predictive model of the 5d-4f emission energy based only on structural parameters was also created.

FS2: Materials for Thermoelectric and Thermionic Energy Conversion

Thermionic Energy Conversion

Room: Flagler A (South Tower)

Session Chair: Mona Zebarjadi, University of Virginia

9:00 AM

(ICACC-FS2-005-2023) Thermionics: More than meets the eye (Invited)

A. Nojeh*[1]

1. University of British Columbia, Electrical and Computer Engineering, Canada

Thermionic energy converters, conceptually simple static heat engines, are excellent candidates for harvesting the vast amounts of high-temperature heat available in the world. They are suitable for both large-scale and small-scale/distributed electricity generation, and basic theory suggests that they can be highly efficient. However, a century after their inception, these converters have not yet become a practical reality, nor achieved their promised high performance even in the laboratory. This may appear as somewhat mysterious but, on closer look, one observes that the theory of these devices is much more extensive than it might appear. A whole host of physical phenomena are intricately inter-twined in these converters, ranging from thermal conduction, solid-state transport, and thermoelectric effects to thermal emission of electrons and far-field and near-field thermal radiation. Quantitative analysis of these devices requires the simultaneous inclusion of all this in order to obtain realistic estimates of converter performance and enable design and engineering of improved devices. We will discuss a comprehensive multiphysics approach to modeling thermionic converters and some of its unexpected results in the context of example devices. We will also review the effort needed on the experimental front to advance these converters, in particular on spectroscopic approaches to studying the thermal emission of electrons and photons.

9:30 AM

(ICACC-FS2-006-2023) Progress toward high power output in thermionic energy converters (Invited)

I. Bargatin*[1]

1. University of Pennsylvania, USA

Thermionic energy converters are solid-state heat engines that have the potential to produce electricity with efficiencies of over 30% and area-specific power densities of 100 W/cm^2. Despite this prospect, no prototypes reported in the literature have achieved true efficiencies close to this target, and many of the most recent investigations report power densities on the order of mW/cm^2 or less. These discrepancies stem in part from the low-temperature (<1300 K) test conditions used to evaluate these devices, the large vacuum gap distances (25–100 μm) employed by these devices, and material challenges related to these devices' electrodes. We argue that, for feasible electrode work functions available today, efficient performance requires generating output power densities of >1 W/cm^2 and employing emitter temperatures of 1300 K or higher. We provide an overview of historical and current design architectures and the recent progress in micron-gap converters, especially those using ceramic spacers between emitter and collectors.

Atomistic Control of Material Structures

Room: Flagler A (South Tower)
Session Chair: Igor Bargatin, University of Pennsylvania

10:30 AM

(ICACC-FS2-007-2023) Thermionic and Thermoelectric transport in 2D metal dichalcogenides (Invited)

M. Zebarjadi*[1]; G. Rosul[1]; S. Akhanda[1]

1. university of virginia, Electrical and Computer Eng., USA

Electron and phonon transport and their response to electric and magnetic fields as well as temperature gradient have been the subject of intense study in metal dichalcogenide systems. Due to their layered nature, these materials are anisotropic with low conductance in the cross-plane direction and large in-plane conductance. They also provide a wide range of electronic properties including some with semimetallic nature and some with layer (thickness)-dependent bandgap. These properties allow the design of various thermal to electrical power generators. In this talk, I will focus on our studies on two of the materials in this class. First, MoSe2, a semiconductor with an indirect bandgap of 0.8 eV in the bulk form and a direct bandgap of 1.55 eV in the monolayer form is studied for thermionic applications. We show the effect of various metallic contacts using first-principles calculations and we identify copper as the metal with the lowest energy barrier for this material. Then, we study MoTe2 which is a type II Weyl semimetal with temperature-induced phase transition from 1T' to Td phase. During the transition, we observe a large Thomson coefficient with values larger than the Seebeck coefficient. We also report that the Seebeck coefficient provides a great tool to study the thermal hysteresis of this material with more pronounced changes compared to that of the resistance.

11:00 AM

(ICACC-FS2-008-2023) The role of local symmetry breaking in $AgGaTe_2$: Ultralow thermal conductivity and high thermoelectric performance in diamondoid compounds

H. Xie*[1]; M. G. Kanatzidis[1]

1. Northwestern University, USA

The ternary AMQ_2 diamondoid materials (A = Cu, Ag; M = Al, Ga, In; Q = S, Se, Te) are a big family of wide band gap semiconductors, which possess various unique electronic and heat transport properties. They are studied as compelling candidates for thermoelectric applications. In this work, we studied the local structure and bonding geometry of $AgGaTe_2$. We discovered an unexpected local symmetry breaking in this compound, which, upon warming, evolves continuously from an undistorted ground state to a locally distorted state while retaining average crystallographic symmetry (the effect is known as emphanisis). This distorted state is caused by the weak sd^3 orbital hybridization of tetrahedral Ag atoms and results in their displacement off the tetrahedron center. This promotes a global distortion of the crystal structure resulting in strong acoustic–optical phonon scattering and an ultralow lattice thermal conductivity. Since the special electronic configuration of Ag causes this effect, the off-centering behavior is expected to be observed in other silver diamondoid compounds. We further demonstrated that introducing the Ag into the tetrahedral diamondoid we can take advantage of the local structure distortion and suppress the thermal conductivity for the materials. We achieved a ZT_{max}=1.5~1.6 in a series of Ag-alloyed diamondoid compounds.

11:20 AM

(ICACC-FS2-009-2023) Atomically-Controlled Nanomaterials for Thermoelectric Performance Enhancement (Invited)

Y. Nakamura*[1]; T. Ishibe[1]

1. Osaka University, Graduate School of Engineering Science, Japan

Atomically-Controlled Nanomaterials for Thermoelectric Performance Enhancement Thermoelectric material with high dimensionless figure of merit ZT described as $S^2\sigma T/\kappa$ has been demanded to realize thermoelectric conversion, where S is Seebeck coefficient, σ is electrical conductivity, κ is thermal conductivity and T is temperature. However, it has been difficult to obtain because of the correlation among the three thermoelectric properties. Nanostructuring is one of the promising approaches to enhance ZT. It needs the atomically-controlled nanostructured materials to develop the methodology for controlling three thermoelectric properties with solid proof. We have developed various kinds of unique well-controlled nanostructures. One example of our nanostructures is Si-based films including ultrasmall Ge nanodots with atomically-sharp interfaces and without strain. Therein, phonon was coherently scattered on nanoscale while keeping high electrical conductivity, resulting in the low κ and high σ. As another nanomaterial strategy for power factor enhancement, we formed Ca-intercalated multilayered silicene films. By deforming the silicene buckled structure, energy dispersion relationship was changed to enhance S while keeping high σ. In this talk, we will introduce various nanostructuring strategies for thermoelectric performance enhancement.

S1: Mechanical Behavior and Performance of Ceramics & Composites

Computational Approaches for Analyzing, and Predicting the Mechanical Behavior and Durability of Ceramic Matrix Composites (CMCs)

Room: Ballroom 5 (South Tower)
Session Chairs: Dong Liu, University of Bristol; Gregory Morscher, University of Akron; Craig Smith, NASA Glenn Research Center

8:30 AM

(ICACC-S1-010-2023) Multiphysics Multiscale Analysis Framework for CMCs Including Scale-dependent Characterization and Quantification of Variability (Invited)

A. Chattopadhyay*[1]

1. Arizona State University, School for Engineering of Matter, Transport, and Energy, USA

Computationally efficient and scalable simulation capabilities and material characterization techniques are critical to enhancing our knowledge of operational environment resistance, damage tolerance, and durability of current and next generation CMCs. The last decade has seen tremendous interest in developing high-fidelity multiscale analysis techniques that link material microstructural characteristics to macroscale response. A significant challenge is in bridging the temporospatial multi-physical behavior across the scales while accounting for inherent microstructural flaws stemming from manufacturing processes as well as from variations in the constituent thermal properties. It has been shown that the size, distribution, and morphology of voids significantly impact CMC constitutive response, thus highlighting the stochastic nature of defects. Therefore, systematically factoring in uncertainty into the analysis is just as important as implementing appropriate physics-based models. This talk will provide an overview of our multiscale modeling approach, which is steered by physics-based models, multiscale material characterization, and stochastic microstructures generated from deep learning algorithms to elucidate the deformation mechanisms at the fundamental scale. Representative results will be presented and current gaps will be identified.

9:00 AM

(ICACC-S1-011-2023) The behavior of self-healing ceramic-matrix composites under chemical and mechanical loads: Microscale characterization and modeling (Invited)

G. L. Vignoles*[1]; G. Bellezza[2]; A. Ebel[1]; R. Baggio[2]; G. Couégnat[1]; O. Caty[1]; F. Rebillat[1]; M. Ricchiuto[2]; M. Colin[2]; D. Bresch[3]; A. Mouret[4]

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Self-healing Ceramic-Matrix Composites (SH-CMCs) have long lifetimes under severe thermal, mechanical and chemical loads. They can resist corrosion by forming a sealing oxide that fills the matrix cracks, delaying the fibers degradation. We will present and discuss the results of a joint experimental/modeling research project on SH-CMCs. Static fatigue tests have been carried out at various temperatures under moist air, with AE and ER monitoring. The crack initiation, sealing/non-sealing by the oxide and posterior failure scenarios were revealed. X-ray CMT scans show the sealing oxide production/recession, in relation with the crack network. An image-based, mini-composite scale model allows predicting the lifetime as a function of applied stress, temperature and spatial distribution of the fiber strengths; the study shows a complex interplay between the fibers' initial strength and their spatial distribution within the tow which cannot be captured by simpler 1D models. At a lower scale, the physics of oxide wetting has been investigated, both by ad-hoc high-temperature experiments under a microscope and by developing a model based on the lubrication equation. Results show that the design of the multi-layer matrix greatly influences the self-healing behavior. The authors thank the French Research Agency for grant no. ANR-17-CE08-0030.

9:30 AM

(ICACC-S1-012-2023) Effect of microstructure on matrix cracking and fiber fracture in unidirectional SiC-SiC composites (Invited)

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Computed Tomography (CT) imaging has become a useful tool for characterizing the internal microstructure and the nature of damage in Ceramic Matrix Composites. CMC microstructures are complex and can include features such as non-uniformities in the orientations and spatial distribution of fibers as well as variations in fiber radii and coating thicknesses. These microstructural features may affect the formation of damage. An automated segmentation method based on deep learning is used to measure local microstructure and the extent of damage in CT images of SiC-SiC composites obtained during in-situ synchrotron experiments. This work aims to improve our understanding of CMC behavior by comparing the current understanding of the damage in ideally uniform microstructures to damage in actual microstructures.

10:20 AM

(ICACC-S1-013-2023) Analysis of the Effects of Local Mechanisms on the Tensile and Creep Response of Unidirectional Ceramic Matrix Composites

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Local material mechanisms such as fiber slipping and the initiation and development of matrix damage such as cracking can significantly affect the fast fracture and creep response of ceramic matrix minicomposites. Although the global stress level at which the creep response is analyzed is below the composite proportional limit, local stresses are assumed to be high enough that localized damage is present in the composite in the form of matrix microcracks. To analyze the effects of these local mechanisms on the material response, a micromechanics-based method based on a fiber shear lag analysis has been developed to analyze the fast fracture response of uncoated and coated ceramic matrix minicomposites under room temperature conditions as well as the creep response of uncoated minicomposites. Parameters such as the ratio of the fiber slip length to the crack spacing were found to have a significant effect on both the fast fracture and creep response of the material. A representative SiCf/SiC unidirectional minicomposite system is analyzed in this work. The computed tensile and creep response curves are compared to experimentally obtained values. This work will allow increased understanding of the key material mechanisms that take place during the fast fracture loading and can be expanded to provide improved analysis methods for full macro composites.

10:40 AM

(ICACC-S1-014-2023) Characterization of Heterogeneity and Mechanical Properties of SiC_f-SiC_m Composites

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Silicon carbide fiber-reinforced silicon carbide matrix composites (SiC_f-SiC_m) have been identified as a leading candidate material for high temperature applications such as gas turbines and nuclear fuel cladding for accident tolerant fuel systems because of their excellent properties, such as low chemical inertness, high-temperature strength retention, etc. However, there is an inherent heterogeneity associated with the composites, including variations in the fiber diameter, porosity distribution, and braid angle imperfections. This heterogeneity contributes to significant scatter in the strength of the composite and uncertainty in the mechanical properties. Digital image processing algorithms were developed to quantify and automatically measure the composites' heterogeneity. Lastly, the strength of the composites was determined under axial compression, hoop burst, and flexural loads to determine which architectural features influence the strength the most.

11:00 AM

(ICACC-S1-015-2023) An Analytical Model for Response and Residual Damage Prediction in Quasi-Statically Indented Alumina-Based Oxide/Oxide Ceramic Matrix Composites

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In this study, a mechanics based analytical model was developed to predict the quasi-static indentation response and state of residual damage in simply supported oxide/oxide alumina based ceramic matrix composite (CMC). The model utilized principles of classical laminate plate theory and fracture mechanics for the entire load-unload response and residual dent size prediction. Experimental results on damage evolution were used to demarcate the material response into different regimes based on key transition points, such as delamination onset and major through-thickness crack formation along the principle fiber directions. Modeling parameters, such as fracture based critical energy release rates, and other mechanical properties were determined in-house for use in model development. The modeling results upon validation with experimental data showed very good correlation. The proposed analytical model enables quick and reliable assessment of material response and residual damage state for quasi-static indentation and low energy impact events.

11:20 AM

(ICACC-S1-017-2023) Crack growth mechanism for an Orthogonal 3-D Woven Amorphous SiC Fiber/SiC/YSi2-Si Matrix Composites under fatigue testing at elevated temperature

S. Kanazawa*[1]; Y. Ikarashi[1]; T. Kishi[1]; K. Kubushiro[2]; T. Aoki[3]; T. Ogasawara[4]

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4. Tokyo University of Agriculture and Technology, Japan

This study aims at clarifying crack propagation mechanism of Orthogonal 3-D woven Amorphous SiC Fiber/SiC/YSi2-Si matrix composites (LMI-CMC) in high temperature fatigue tests. A sample specimen of LMI-CMC has a single notch to detect a microscopic crack clearly. The crack was observed to initiate and propagate from the bottom of the notch with X-ray CT under cyclic loading with 110MPa at the temperature of 1373K. The crack propagation rate was obviously high when it passed in either case of matrix layers and 90deg bundles. The crack in 0deg bundles propagated slowly compared to the other regions. These results indicate the crack in the 0 deg bundles decelerated the total crack propagation rate. Observations of the crack path revealed BN layers, which is interface coatings, around the fibers disappeared and oxidation of fiber surfaces developed during high temperature fatigue test. The SiC fibers and SiC matrix were fixed by the oxidization after the BN layer vanished near the region of the crack path in the 0deg bundles. In the other crack propagation areas, the SiC fibers and matrix layer were not fixed although they both were oxidized. This result indicates that not only fixation of the SiC fibers and SiC matrix but also abrasion or other factor effect on crack propagation behavior in high temperature fatigue tests.

11:40 AM

(ICACC-S1-016-2023) Fatigue lifetime prediction model of an orthogonal 3-D woven amorphous SiC fiber/SiC/YSi$_2$-Si based matrix composites at elevated temperature in air

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4. Tokyo University of Agriculture and Technology, Japan

The objective of this study is to propose a fatigue lifetime prediction model for an orthogonal 3-D woven amorphous SiC fiber/SiC/YSi$_2$-Si based matrix (SiC-f/SiC) composites at elevated temperature in air. Interrupted-fatigue testing was conducted at 1373 K using a single edge notched specimen. The crack growth behavior from the notch tip was evaluated using a digital microscope and an X-ray CT. Microscopic observation after fatigue testing was conducted to reveal the crack growth mechanisms. Experimental results show that the fatigue crack of the SiC-f/SiC composite under cyclic tensile loading consists of the fiber ruptured region and fiber bridging region. The microscopic observation results imply that fiber rupture in the bridging region was attributable to bonding of fiber-matrix interface caused by oxidation, interface wear caused by cyclic tensile loading, and the other mechanisms, resulting in fatigue crack growth with fiber rupture. In this study, that crack growth was modeled using a fracture mechanics model for monolithic ceramics. By introducing stress-dependent initial crack, which is the point at fatigue crack initiation, into that model, a fatigue lifetime prediction model for the SiC-f/SiC composite was proposed. The predicted lifetime agreed with the experimental result obtained using a dumbbell-shaped specimen.

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

Advanced Ceramic Coatings for Extreme Environments I

Room: Flagler C (South Tower)

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

8:40 AM

(ICACC-S2-012-2023) Structural study of long-term-resistant to oxidation at 600 °C electrically conductive Ti-Al-C and Ti-Cr-Al-C coatings

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The Ti-Al-C and Ti-Cr-Al-C 5-6 μm thick coatings were obtained by vacuum-arc evaporation and combine vacuum-arc evaporation with magnetron sputtering, respectively, using MAX phases Ti$_2$AlC and

Ti_3AlC_2 targets. In the case of Ti-Cr-Al-C coatings a Cr source was placed next to the target. Deposited Ti-Al-C coating by high energetic "Bulat–6" demonstrated crystalline structure and contained (wt.%): Ti_3AlC(16), Ti_2AlC(17), TiC (19) and Al_2Ti (37). And after 1000 h heating at 600 °C in air occurred ordering of Al solved in TiC phase and its composition transformed to (wt.%): Ti_3AlC(15), Ti_2AlC(37), TiC(2), AlTi(19), and $AlTi_3$(17); weight gain was $\Delta m/S=0.07$ mg/cm^2. In parallel, on the surface of the containing formed 0.5 µm film with small excess of O, N and C as compare to that in the coating. The observed structural transformations did not lead to the decrease of surface electrical conductivity ($\sigma=1.33\times10^6$ and 1.30×10^6 S/m before and after oxidation, respectively), but even resulted in 1.7 times higher nanohardness (H_B=9.5 GPa) and in 1.4 times higher Young modulus (E=190 GPa) due to nitriding. The Ti-Cr-Al-C coating deposited by magnetron using lower energy intensities had amorphous structure (with H_B=14 GPa, E=240 GPa) and after heating in air at 600 °C for 1000 h it demonstrated lover $\Delta m/S=0.048$ mg/cm^2 and increasing of σ from 4.35×10^5 to 1.02×10^6 S/m.

9:00 AM

(ICACC-S2-013-2023) Durability of hard protective coatings: Assessing the fracture and fatigue resistance of nanostructured thin films

L. Zauner[1]; R. Hahn*[1]; E. Aschauer[1]; T. Wojcik[1]; A. Davydok[2]; O. Hunold[3]; P. Polcik[4]; H. Riedl[5]

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2. Helmholtz-Zentrum Hereon, Institut für Werkstoffphysik, Germany
3. Oerlikon Balzers, Oerlikon Surface Solutions AG, Liechtenstein
4. Plansee Composite Materials GmbH, Germany
5. TU Wien, Institute of Materials Science and Technology, Austria

Tailoring the intrinsic fracture characteristics of hard protective coatings toward the fatigue properties of state-of-the-art bulk materials is paramount for applying innovative coating materials. Thus, an in-depth knowledge of the failure pathways of ceramic-based thin films – typically lacking in intrinsic ductility – is imperative to extend their lifetime. Here, we present a novel approach using quasi-static and cyclic bending of pre-notched, unstrained micro-cantilever beams in conjunction with in-situ synchrotron X-ray diffraction to reveal the intrinsic fracture toughness (K_{IC}) and critical failure aspects of Cr and Cr-based ceramic thin films (CrN, CrB_2, and Cr_2O_3) under various loading conditions. Up to the high-cycle fatigue regime (i.e., $N = 10^7$ cycles), the failure of monolithic coatings is shown to be dominated by the inherent fracture resistance, irrespective of the bonding character. The observed variation in fracture toughness is put into context with linear-elastic fracture theory and complementary micro-pillar compression, thereby elucidating the wide range of values from as low as 1.6±0.2 MPa m$^{0.5}$ for $Cr_{1.79}O_3$ up to 4.3±0.3 MPa m$^{0.5}$ for $Cr_{1.03}B_2$. Moreover, possible mechanisms governing the elastic-plastic deformation response of all coatings, both in quasi-static and cyclic-loading conditions, are discussed.

9:20 AM

(ICACC-S2-014-2023) $Ti_{1-x}Al_xN$ PVD Coatings in Hot-Corrosion Environments

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4. TU Wien, Institute of Materials Science and Technology, Austria

Hot corrosion is an accelerated oxidation process that is commonly observed in high temperature settings (650-950 °C), such as gas turbines, coal gasification plants and waste incinerators. It is a phenomenon where sulfur-rich atmospheres (exhaust gases) reacts with salts impurities to form high-melting sulfate-salts that then adhere to machining components. There, the salt deposits elicit an accelerated degradation of the operating parts through the formations of porous, non-protective oxide scales, drastically reducing the longevity of in service parts. Particularly Ni-, Co-, and Fe-based superalloys, which represent the backbone in the herein before mentioned applications, are especially prone to hot-corrosion attack. This contribution presents $Ti_{1-x}Al_xN$ as a potential candidate as protective PVD coating for hot-corrosion environments. $Ti_{1-x}Al_xN$ coatings with varying metal content ratios were arc-evaporated on a Ni-based superalloy and tested in an in-house built hot-corrosion testing rig. By applying a sulphate-salt mixture from the alkali and alkaline earth metal groups, coated and uncoated samples were tested in a SO_x-rich atmosphere at 700 and 850°C for a maximum duration of 30 h, and subsequently analysed using a set of high-resolution characterization techniques.

9:40 AM

(ICACC-S2-015-2023) Tribochemical Reactions of Si_3N_4 in Water Lubrication Revealed by Reactive Molecular Dynamics Simulations

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Water lubrication technology is expected to be used in water pumps to avoid oil contamination. Silicon nitride (Si_3N_4) is proposed as a sliding material for water lubrication because it shows super-low friction whose frictional coefficient is less than 0.01 under water lubrication. The super-low friction of Si_3N_4 is considered to be caused by tribochemical reactions during the running-in period. During the running-in period, a SiO_2 film is formed on the sliding interface by tribochemical reactions. Then, a tribolayer appears at the sliding interface and causes super-low friction. However, it is challenging to investigate the tribochemical reactions during the friction because of its complexity. A reactive molecular dynamics (RMD) simulation is a promising tool to clarify the atomic-scale mechanisms of tribochemical reactions. In this study, we analyzed the tribochemical reactions of the Si_3N_4 and water by the RMD simulation. The results show that the NH_3 appears due to the tribochemical reactions between the Si_3N_4 and water. This result is consistent with the previous experiments. When Si_3N_4 continues to slide in water, the termination of the Si_3N_4 surface changes from OH-termination to NH-termination. The change of the surface termination significantly affects the friction coefficient. These insights can lead to the development of low-friction water lubrication system.

10:20 AM

(ICACC-S2-016-2023) Key parameters in the formation of SiC coatings by Liquid Silicon Infiltration

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1. University of Augsburg, Institute of Materials Resource Management MRM, Materials Engineering, Germany

Environmental Barrier Coatings (EBCs) such as silicon carbide (SiC) are necessary for carbon matrix composites facing demanding environments. One of the most time and cost-efficient process routes for applying SiC coatings is a method based on the Liquid Silicon Infiltration, aiming only a siliconization of the component's surface. The success of such coatings depends on various material and process parameters, which are not fully understood yet. Failed siliconizations are usually indicated by a greenish color. The worst-case scenario is a detachment of the SiC coating after processing or early failure during application. For a deeper understanding graphite substrates, varying in density, open porosity, and impurities, as well as different silicon powders varying in particle size and impurities have been used for parameter studies. Molar ratios, dwell time, and heating rates were changed to investigate the influence of each parameter. The resulting samples have been analyzed by means of X-ray diffraction (XRD), scanning electron microscopy (SEM), and energy dispersive X-ray (EDX). Two different counteracting reaction mechanisms were identified, leading to alpha or beta silicon carbide formation. Different strategies have been developed to promote favorable reaction paths, leading to a successful SiC coating with the desired high-temperature resistance.

10:40 AM

(ICACC-S2-017-2023) Development of Ablation-Resistant, High Emittance Coatings for Carbon/Carbon Composites for Hypersonic Application

A. A. Saad*[1]; C. Martinez[1]; R. Trice[1]

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During a high-speed flight, the leading edges of hypersonic vehicles can experience enormous heat fluxes, with surface temperatures greater than 1600°C expected. While carbon/carbon (C/C) is a candidate material for leading edges and other hot structures, it is prone to oxidation and increasing ablation damage above 500°C. Ablation-resistant coatings can protect the C/C, while emittance can be engineered to lower the leading-edge surface temperature via radiative cooling. In this study, a novel multi-layer coating system based on individual layers consisting of borides, carbides, zirconium oxides, and rare-earth oxide dopants as emissivity modifiers is being applied to a C/C surface via pack cementation and plasma spray. Ablation testing is being performed to evaluate the effectiveness of the multi-layer coatings in simulated high-heat flux environments. An FTIR-based emissometer is being developed to measure the spectral emittance of the coating surface at high temperatures up to 1400°C. The results from these experiments will help design and optimize ablation-resistant, high emittance coating systems for C/C composite materials.

11:00 AM

(ICACC-S2-018-2023) Thermal properties and ablation resistance of a high-entropy metal diboride: $(Hf_{0.2}Zr_{0.2}Ti_{0.2}Ta_{0.2}Nb_{0.2})B_2$

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To meet the diverse demands of the aerospace, nuclear, and energy industries, it has become a necessity to expand the pool of ultra-high temperature ceramics (UHTCs). The successful fabrication of the high-entropy metal diborides and carbides can provide a solution to this issue. To properly assess the suitability high-entropy metal diborides (HEB) for extreme structural applications, we conduct detailed thermal and ablation characterizations of a HEB specimen: $(Hf_{0.2}Zr_{0.2}Ti_{0.2}Ta_{0.2}Nb_{0.2})B_2$ in this work. Our electrical conductivity measurements show that the thermal conductivity of HEB is electron-dominated. Due to this, the thermal conductivity increases as function of temperature and becomes comparable to that of other UHTCs at high temperatures. We conduct thermal conductivity mapping of HEB and a prototypical ZrB_2 system and find that the HEB possesses nearly isotropic thermal conductivity along different crystallographic orientations in contrast to that of ZrB_2. Additionally, ablation threshold measurements reveal that the HEB and ZrB_2 possess nearly identical ablation resistance. Our characterizations are vital for guiding the usage of HEB in extreme environment applications.

11:20 AM

(ICACC-S2-019-2023) The development of polymer-derived Si(Al)CN CMC for high temperature applications

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3. DSTL, United Kingdom
4. BAE Systems, United Kingdom

The work relates to the development of a new high temperature CMC by tailoring the ceramic microstructure using an inexpensive commercially available polysilazane doped with aluminium. The first set of results relate to the successful modification the polysilazane with aluminium; FT-IR and NMR analysis indicate that protonation occurs at the S-H and N-H bonds of the polysilazane. The characterisation of the subsequent polymer-to ceramic transformation with FT-IR shows the aluminium doping is retained during pyrolysis and that the resultant ceramic is amorphous in nature, as shown by XRD. EDX analysis of the Si(Al)CN ceramic also confirms that the aluminium is homogenously distributed on an atomic scale. In order to fabricate a CMC using the Si(Al)CN ceramic a powder processing strategy was developed using a cationic polymeric dispersant, combined with SiC powder to mitigate the shrinkage and porosity evolution that occurs during pyrolysis. The SiC powder / Al-modified polysilazane slurry was then used to manufacture a CMC with SiC fibre using a simple hand layup technique. Furthermore, initial oxidation tests of the metal-modified polysilazane at high temperature seem very promising, but further work in aggressive environments is necessary to ascertain the long-term performance.

S3: 20th International Symposium on Solid Oxide Cells (SOC): Materials, Science and Technology

Air Electrode

Room: Ponce de Leon (North Tower)
Session Chairs: Federico Smeacetto, Politecnico di Torino; Xingbo Liu, West Virginia University

8:30 AM

(ICACC-S3-010-2023) Enhancement of Low-Temperature Solid Oxide Fuel Cell Performance and Durability via Surface Chemistry Modification (Invited)

E. D. Wachsman*[1]; I. Robinson[1]; Y. Huang[1]; S. Horlick[1]; A. Hussain[1]; A. Pesaran[1]

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The development of active cathodes is one of the most critical challenges to lowering the operating temperature for solid oxide fuel cells (SOFCs). Here we demonstrated that by modifying the cathode surface chemistry at a relatively low temperature, the cathode activity and durability can be simultaneously enhanced. This low-temperature modification using multi-valent cations activates the highly defected surface and maintains the nanoscale electrocatalysts by bypassing the high-temperature sintering procedure for SOFC fabrication. The modified cathode at 600 °C shows an order of magnitude reduction in impedance to only 0.05 Ωcm^2 with a peak power density of 1.1 W/cm^2 and is stable over 2000 hours. The combination of in situ characterization, distribution of relaxation time analysis on impedance spectroscopy, and surface chemistry analysis reveals the importance of surface chemistry control on the gas-solid reaction activity and durability and provides the design principle for numerous future solid oxide cells.

9:00 AM

(ICACC-S3-011-2023) Enhancement of the SOE performance by fine-tuning the composition and microstructure of the air electrode based on state-of-the-art materials

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2. Institute of Power Engineering, Center for Hydrogen Technologies (CTH2), Poland
3. Institute of Power Engineering, Ceramic Department CEREL, Poland

State-of-the-art materials for air electrode of solid oxide electrochemical cells (SOC), namely LSC and LSCF, exhibit good catalytic activity and high mixed ionic-electronic conductivity, however, they are characterized by high thermal and chemical expansion which leads to a mechanical mismatch with an electrolyte, leading to accelerated degradation. In order to maximize the performance, and prevent degradation through delamination of the electrode, composite GDC-LSC/LSFC electrodes were proposed. Their composition gradually changes from electrolyte-electrode interphase to the electrode surface thanks to microstructural manipulations. The impact of modification of the microstructure by an increase of the porosity by addition of pore former (graphite or PMMA) and infiltration with catalytically active oxides (e.g. Pr_xO_y) was investigated. Cells with an active area of 16 cm^2 were evaluated in SOE mode in the temperature range from 650 to 750 °C. Tests included measurements of j-V dependences and the EIS spectra. The microstructure of the SOC was investigated using SEM/EDS analysis. The ability of the cell to operate at a higher current density was observed. The presented research was financially supported by the National Centre for Research and Development, Poland, within project no. LIDER/1/0003/L-12/20/NCBR/2021.

9:20 AM

(ICACC-S3-012-2023) Investigation of Cobalt-free air electrodes for solid oxide electrochemical cells

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Due to an extraordinary catalytic activity and high mixed ionic-electronic conductivity of cobalt-based perovskites they are considered as state-of-the-art air materials for electrodes of solid oxide electrochemical cells (SOC). However, the locations of cobalt mining in politically unstable regions, together with the continuously shrinking reserves, and rapidly growing demand stimulated seeking for alternative substitutes with substantially reduced environmental footprint. Among the various options discussed in the literature the Cu-based oxides seem to be a promising and economically viable candidate The work presents a study on fuel electrode supported SOCs with $La_{1-x}Ba_x(Cu,Ni)O_{3-\delta}$ used as the air electrodes. The proposed Co-free oxides were obtained by sol-gel method. The high pressure injection molding (HPIM) was used for fabricating the supports of SOC, while the preparation of thin functional layers was done using screen printing. Characterization of the cells included both the microstructural analyses (SEM, EDS) and the solid state electrochemistry methods (acquisition of IV curves and EIS spectra). Electrochemical performance of the fabricated cells was investigated in electrolysis (SOEC) and reversible (rSOC) modes in temperature range from 650 to 750°C. This project was financially supported by the National Science Centre, Poland, within project no. 2020/37/B/ST8/02097.

9:40 AM

(ICACC-S3-013-2023) Optimization of Solid Oxide Cells Air Electrodes for Infiltration

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Nano-catalyst infiltration is a proven technique for surface modification of solid oxide cell (SOC) air electrodes. Enhanced performance is achieved by an increase of both electrochemical activity and density of active reaction sites. Due to the complexity of the oxygen exchange reaction on mixed ionic-electronic conductive (MIEC) electrodes, improvement of electrode performance is not only determined by selection of catalyst, but also deeply impacted by the initial backbone microstructure. This study focuses on optimization of the SOC air electrode specifically for infiltration. Electrodes with various microstructure properties were prepared with and without catalyst infiltration. Changes of performance (e.g., ohmic resistance, concentration/activation polarization) under various operating conditions are also analyzed. Multiphysics simulations were carried out to gain insight into the nature of the relationship between the catalyst and backbone microstructure. This parametric study provides quantitative analysis of the resistance and other losses from different materials and microstructures. This study revealed that the electrode with the best initial performance may not be the suitable candidate for infiltration. The combination of material and microstructure parameters must be considered for optimizing electrode infiltration.

10:20 AM

(ICACC-S3-014-2023) Submicron- and Nano-materials for Solid Oxide Fuel Cells: Technical Challenges and Solutions

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1. Forschungszentrum Juelich, IEK-1, Germany
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In the last 20 years, the use of nano-scaled materials in solid oxide fuel cells (SOFCs) has increased steadily leading to a variety of advantages compared to conventional materials. Next to the lower operating temperatures of 500°C – 600°C (compared to 700°C – 850°C in conventional SOFCs), the enhanced surface vacancy concentration of nano-materials, show promising catalytic activities. Furthermore, increased ionic and electronic conductivity has been observed. These promising characteristics, encouraged us to focus on the development of SOFCs using submicron- and nano-scaled materials. Therefore, conventional production techniques, such as screen-printing, as well as unconventional ones, like infiltration experiments are targeted for the manufacturing of Ni/GDC fuel cells. In addition, the microstructural change of the produced SOFCs will be investigated by comparing the initial compositions of the microstructure with the final ones. We are in the process to develop manufacturing techniques to overcome emerging technical challenges caused by the use of submicron- and nano-scaled materials. Furthermore, the samples have been investigated upon its initial mechanical integrity as well as its initial electrochemical efficiency by measuring R_{pol} at 600°C. Eventually, the most promising samples will be part of a post-test analysis and the different manufacturing methods will be evaluated.

10:40 AM

(ICACC-S3-015-2023) High Entropy Perovskites as Solid Oxide Fuel Cell Cathodes

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1. West Virginia University, Mechanical & Aerospace Engineering, USA
2. West Virginia University, Chemical & Biomedical Engineering, USA

a new series of high-entropy perovskites (HEPs) are investigated as cathode materials for solid oxide fuel cells. Multiple rare-earth, alkaline-earth, and high-order transition metal elements are used for the A-site of this ABO3 structure. A pure phase is achieved through the designed combination of different elements in seven out of eight candidates. Three out of eight candidates show similar or better performance than the (La0.6Sr0.4)(Co0.2Fe0.8)O3−δ (LSCF) benchmark. It is found that A-site elements can cast a substantial influence on the overall performance even with a change as small as 10% of the total cations. It seems that each element has its individual “phenomenal activity” that can be transferred from one candidate to the other in the general setting of the perovskite structure, leading to the best candidate by using the three most active elements simultaneously at the A-site. Excellent Cr tolerance has been observed on the (La0.2Sr0.2Pr0.2Y0.2Ba0.2)Co0.2Fe0.8O3−δ sample, showing degradation of only 0.25%/kh during a 41 day operation in the presence of Cr, while LSCF increases by 100% within the first day in the same condition. X-ray photoelectron spectroscopy discovers no Sr segregation as LSCF is found in this HEP; rather, the active element Y takes more A-sites on the outermost layer after long-term operation.

11:00 AM

(ICACC-S3-016-2023) Structure-Property Relationships of $BaCo_{0.4}Fe_{0.4}Zr_{0.1}Y_{0.1}O_{3-\delta}$-type Triple-Conducting Materials

J. H. Duffy*[1]; N. Birkner[1]; E. M. Kelder[2]; K. Brinkman[1]

1. Clemson University, Materials Science and Engineering, USA
2. Delft University of Technology, Storage of Electrochemical Energy, Reactor Institute Delft, Netherlands

Protonic ceramic fuel cells (PCFCs) are an emerging technology for efficiently converting fuel into energy. Triple-ionic electronic conductors (TIECs) have recently been studied as effective cathodes for PCFCs due to their improved kinetics at intermediate temperatures. In this work, TIEC materials $BaCo_{0.4}Fe_{0.4}Zr_{0.2-X}Y_XO_{3-\delta}$ (BCFZY$_X$, X=0, 0.1, 0.2) and $BaCo_{0.8-X}Fe_XZr_{0.1}Y_{0.1}O_{3-\delta}$ (B(CF)$_X$ ZY, X=0, 0.4, 0.8) are characterized to reveal their complex structure-property relationships. All materials appear to exhibit disordered cubic perovskite structure in its bulk form through x-ray diffraction. However, upon investigation through neutron diffraction, it is revealed that changes in Zr to Y and Co to Fe concentrations cause significant structural changes in both material sets. The structural changes across materials are correlated with bulk electron, oxygen ion, and proton conductivities, as well as oxygen surface exchange kinetics, proton uptake, and thermodynamic stability. Selected materials are synthesized as porous electrodes to show the effect of the atomic structure and bulk properties on cathode performance. The presented results help to identify the bulk and atomic-level characteristics of high-performance cathode materials for PCFC applications.

11:20 AM

(ICACC-S3-017-2023) $BaCo_{0.4}Fe_{0.4}Zr_{0.1}Y_{0.1}O_{3-\sigma}$ Cathode Performance for Proton Conducting Solid Oxide Fuel Cells with $BaZr_{1-x}Ce_xY_{0.1}Yb_{0.1}O_{3-\delta}$ Electrolytes

W. Li*[1]; M. Sozal[1]; V. Drozd[1]; A. Durygin[1]; Z. Cheng[1]

1. Florida International University, Mechanical & Materials Engineering, USA

$BaCo_{0.4}Fe_{0.4}Zr_{0.1}Y_{0.1}O_{3-\sigma}$ (BCFZY) is a proton, oxygen-ion, and electron-hole conducting cathode material for solid oxide fuel cells. Its electrode performance as well as the rate-limiting step in air with moisture are not well understood. Therefore, we fabricated three types of symmetric cells with the same BCFZY cathode and $BaZr_{1-x}Ce_xY_{0.1}Yb_{0.1}O_{3-\delta}$ (x = 0.1, 0.4, and 0.7) proton conducting electrolytes. It is observed that the cathode interfacial resistance in dry air is not related to the ionic conductivity of the electrolyte, suggesting the cathode rate-limiting step is related to the oxygen absorption of the cathode. Upon introducing moisture to the air, the cathode interfacial resistance drops for the high-Ce electrolyte, while increases for the high-Zr electrolyte. It is hypothesized that during the cathode firing process, Ce has a stronger tendency than Zr to diffuse from the electrolyte to the cathode. The diffused Ce might increase the hydration capacity of the cathode and improves the proton conductivity and interfacial resistance. Such a hypothesis is supported by Rietveld refinement comparing the volume of BCFZY cathode before and after firing, as well as additional techniques such as energy-dispersive X-ray spectroscopy (EDS) illustrating the element concentration distribution at the cathode/electrolyte interface.

11:40 AM

(ICACC-S3-018-2023) Fabrication and Electrochemical Testing of Silver Pattern Cathodes for Proton Conducting IT-SOFC

M. Sozal[1]; W. Li[1]; S. Das[1]; B. Jafarizadeh[1]; A. Chowdhury[1]; A. Durygin[2]; V. Drozd[2]; C. Wang[1]; Z. Cheng*[1]

1. Florida International University, Mechanical & Materials Engineering, USA
2. Florida International University, Center for the Study of Matter at Extreme Conditions (CeSMEC), USA

Dense silver (Ag) cathodes with different triple phase boundary (TPB; the interface of gas, electrolyte and electrode) length (L_{TPB}) and electrode area (A_{ELT}) were fabricated by photolithography and E-beam evaporation over proton-conducting $BaZr_{0.4}Ce_{0.4}Y_{0.1}Yb_{0.1}O_{3-\delta}$ (BZCYYb4411) electrolyte. A bi-layer lift-off resist method appears more versatile than single layer for successful pattern cathode fabrication. The electrochemical behaviors of Ag pattern cathodes over the BZCYYb4411 electrolyte were tested with different counter electrodes and correlated against geometric features such as L_{TPB} and A_{ELT} to understand the oxygen reduction kinetics and mechanism for proton conducting solid oxide fuel cells.

12:00 PM

(ICACC-S3-019-2023) Mitigating Air Electrode Delamination and Improving Durability of Solid Oxide Electrolysis Cells by Infiltrating $SrFe_2O_{4-d}$ Nanoparticles

Y. Fan*[1]; Y. Chen[2]; H. W. Abernathy[3]; R. Pineault[3]; R. Addis[1]; X. Song[2]; G. Hackett[3]; T. Kalapos[1]

1. LRST, National Energy Technology Laboratory, USA
2. West Virginia University, USA
3. DOE National Energy Technology Laboratory, USA

Air electrode of LSM/YSZ cell exhibits delamination and accelerated performance degradation during short-term electrolysis operation. To mitigate such degradation, $SrFe_2O_{4-d}$ were introduced into the LSM/YSZ air electrode, and the impact of air electrode infiltration on the overall cell performance and long-term stability was evaluated. Uninfiltrated baseline cells exhibited significant increase in series resistance and became completely delaminated after 300h of operation under 800°C, 0.5A/cm^2 and 60% steam. Cells with air electrode $SrFe_2O_{4-d}$ infiltration exhibited immediate decrease in operation voltage and reduction of both series and polarization resistance while sustaining 1000h of continuous electrolysis operation without delamination. Nanostructure examination revealed active interaction of the $SrFe_2O_{4-d}$ infiltrate with cells after calcination. During electrolysis, there is continuous cation exchange between the nanoparticles and the backbone. The nanoparticles maintain intact morphology and distribution within the pores. The present study demonstrates a feasible and viable approach to mitigating electrode delamination while simultaneously increasing the performance and durability of solid oxide cells for electrolysis hydrogen production.

S5: Next-Generation Bioceramics and Biocomposites

Bioactive and Resorbable Ceramics

Room: Ballroom 1-2 (South Tower)
Session Chair: Cristina Balagna, Politecnico di Torino

8:30 AM

(ICACC-S5-009-2023) Bioresorbable devices enriched with multifunctional nano-biomaterials to stimulate compromised bone remodelling (Invited)

S. Fiorilli*[1]; G. Montalbano[1]; F. Banche Niclot[1]; C. Vitale-Brovarone[1]

1. Politecnico di Torino, Applied Science and Technology, Italy

Despite the excellent ability to heal, in 5-10% of bone defects, especially those caused by trauma, cancer, or osteoporosis, the healing process fails under conventional treatments. Great efforts have been dedicated to the design of nano-biomaterials able to boost bone repair, among which mesoporous bioactive glasses (MBGs), which provide excellent bioactivity joined to the release of therapeutic agents (ions, drugs, biomolecules) able to exert specific biological effects. Multifunctional devices to treat compromised bone healing in elderly and osteoporotic patients have been conceived by combining MBGs, as bioactive phases, with resorbable polymers (both natural and synthetic) or inorganic phases in order to provide advanced clinical solutions, including scaffolds, injectable cements and hydrogel for delivery of therapeutics. In addition, the employ of bio-fabrication technologies, mostly 3D printing and electrospinning, and the inclusion of specific biological cues allowed the development of personalized composite 3D scaffolds capable of supporting cell proliferation, cell guidance, and promoting the rebalance of physiological bone remodelling. Comprehensive physical-chemical characterisation and in vitro/in vivo biological assessment of the developed composite constructs are here reported and discussed, highlighting the related challenges and future perspectives.

9:00 AM

(ICACC-S5-010-2023) Preparation of Novel Guided Bone Regeneration Membrane by Lamination of Hydroxyapatite/ Collgen Bone-Like Nanocomposite and Poly-L-Lactide (Invited)

M. Kikuchi*[1]; T. Suraya[2]

1. National Institute for Materials Science (NIMS), Bioceramics Group, Japan
2. University of Tsukuba, Tsukuba Life Science Innovation, Graduate School of Science and Technology, Japan

Guided bone/tissue regenerations are widely used in dental field to repair small size defect of bone and periodontal tissues. These techniques are based on prevention of scar tissue invasion to the defect by using barrier membrane. The author had proposed biodegradable composite membrane 20 years ago; however, further osteoconductive property had seemed to be required in some situations. The author also prepares the hydroxyapatite/collagen bone-like nanocomposite (HAp/Col) with high bone regeneration properties and several shapes including membrane. In this study, novel guided bone regeneration membrane was prepared by lamination of the HAp/ Col and PLLA membranes, and its physicochemical properties and in vitro cell responses were examined. The HAp/Col were prepared from reagent or egg-shell (ES) derived calcium carbonate. The HAp in the ES-HAp/Col had low crystallinity than that in the HAp/Col prepared by the same conditions due to presence of small amount of Mg; however, its long and short fibers were successfully prepared. The HAp/Col or ES-HAp/Col membranes was prepared by filtration of suspension containing both the long and short fibers. After the dehydrothermal cross-link, the membrane was laminated with PLLA membrane. Results in physicochemical and biological properties will be presented at the podium.

9:30 AM

(ICACC-S5-011-2023) 3D printed PLA-TCP-CNPs scaffolds for bone tissue engineering

S. V. Harb*[1]; E. Kolanthai[2]; E. H. Backes[1]; C. A. Beatrice[1]; C. J. Neal[2]; S. Seal[2]; L. Pessan[1]

1. Federal University of Sao Carlos, Department of Materials Engineering, USA
2. University of Central Florida, Department of Materials Science and Engineering, USA

Bioactive and biodegradable scaffolds have been explored as temporary templates to guide the growth of new tissue in the human body. The poly(lactic acid) (PLA)-β tricalcium phosphate (TCP) (90 wt%-10 wt%) containing CeO_2 nanoparticles (CNPs; 1, 5, and 10 wt%) composites filament was prepared by a melted state extrusion method. Then, a 3D polymer composite scaffold was printed using the fused filament fabrication (FFF) technique. Physicochemical and biological studies were performed on these 3D printed scaffolds. The scaffolds presented precise geometry compared to the designed architecture and a homogenous distribution of TCP and CNPs, as confirmed by SEM-EDS. The scaffolds were analyzed by compression testing and presented elastic modulus between 80-100 MPa, suitable for bone tissue engineering. The presence of TCP and CNPs did not affect PLA's degradation ratio and molar mass, as evidenced by size-exclusion chromatography and weight measurements as a function of time, respectively. In addition, the presence of CNPs enhanced the proliferation and differentiation of mesenchymal stem cells; thereby, increasing alkaline phosphatase (ALP) activity and calcium deposition. Moreover, the presence of CNPs imparted bactericidal property against E. coli. Results from this study have shown that the PLA-TCP-CNPs composite scaffold could be used as an alternative polymeric implant for bone tissue engineering applications.

Nanostructured Bioceramics

Room: Ballroom 1-2 (South Tower)
Session Chair: Roger Narayan, North Carolina State University

10:20 AM

(ICACC-S5-012-2023) Light Triggered Theranostics (Invited)

F. Vetrone*[1]

1. INRS, Université du Québec, Centre Énergie, Matériaux et Télécommunications, Canada

Nanoparticles excited in the near-infrared (NIR), in particular rare earth doped nanoparticles, are quickly emerging as useful tools in diagnostic and therapeutic medicine. Given their versatile optical properties, they are at the forefront of light triggered theranostics (therapy + diagnostics). In particular, the usefulness of these nanoparticles stems primarily from the fact that NIR light is silent to tissues thus minimizing autofluorescence, possesses greater tissue penetration capabilities, reduced scattering, and does not cause photodamage to the specimen under investigation. Moreover, tailoring of the nanoparticles' absorption and emission wavelengths allow them to operate within the so-called "biological windows", regions of the spectrum in which tissues are partly transparent. Here, we present the synthesis and surface functionalization of various rare earth doped nanoparticles and show how they can be used as building blocks in the development of light triggered multifunctional theranostic nanoplatforms for a variety applications.

10:50 AM

(ICACC-S5-013-2023) Bioinspired and biobased composite materials: Promise for a greener future? (Invited)

T. Speck*[1]

1. University of Freiburg, Germany

During 3.8 billion years of biological evolution living beings developed fascinating often multifunctional materials systems for support and motion. Most of them are bioceramics or biocomposites.

Biocomposites in plants are usually fiber-reinforced and micro-laminated material systems with a hierarchical structuring. Plants with their light-weight (high) load-bearing fibre-reinforced stems have inspired lightweight constructions in architecture and many technical applications since decades. The same holds for impact damping and puncture resistance as found in seed shells and fruit peels consisting of fibre-reinforced or laminated biocomposites. More recently, plants have also been recognized as valuable concept generators for biomimetic soft machines, since their movement is typically based on elastic deformation and there are no mechanically highly loaded localized slip joints. The quality of these biocomposites and their potential as models for bioinspired and/or biobased composites is demonstrated by several current research projects of the livMatS cluster of excellence. Examples that can also contribute to more sustainable solutions are bioinspired fiber-reinforced lightweight load-bearing systems, damping and puncture-resistant material systems, (anti-) attachment structures, and bioinspired 4D-printed hygromorphic soft machines and self-adaptive building envelopes.

11:20 AM

(ICACC-S5-014-2023) Model Porous Minerals as Habitats for Microbial Consortia Through Suspension-based Freeze Casting

L. Quinn*[1]; D. Johnson[1]; K. Sharma[1]; Y. Jangir[1]; P. Samantaray[1]; V. Orphan[1]; J. Kornfield[1]; K. Faber[1]

1. California Institute of Technology, USA

In their native environments, microbial consortia of bacteria and archaea play a central role in methane, sulfur, and iron cycling, making them promising candidates to assist in such sustainability challenges as reducing methane levels or removing toxic metal species from contaminated water. To mimic the sediment environments in which these consortia are naturally found, we turn to porous ceramics as a model mineral that can withstand the pressures needed to house these organisms in laboratory settings. Freeze-casting is a tailorable method of producing porous ceramics in which solvent choice and freezing rate can be adjusted to alter the pore morphology. In this work, model porous minerals are produced using suspension-based freeze casting, where the permeability afforded by oriented pores is expected to facilitate inoculation of the porous scaffold and allow for nutrient and waste transport. By freeze casting with iron oxides, we produce porous ceramics that mimic iron-containing sediments in the native habitats of these microorganisms. Additionally, we freeze cast using materials with a refractive index close to that of water, aiming to produce transparent porous ceramics that allow for in-situ study of microbial processes within the ceramic. Through these studies, we link the pore morphology and the chemistry of the ceramic to the eventual function of the model mineral.

S6: Advanced Materials and Technologies for Rechargeable Energy Storage

All-solid-state Batteries III

Room: Coquina Salon E (North Tower)
Session Chairs: Rick Laine, University of Michigan;
Shusuke Kasamatsu, Yamagata University

8:30 AM

(ICACC-S6-009-2023) SoC-dependent Interfacial Stability of Electrolytes and Cathodes in Solid-state Lithium Batteries (Invited)

P. Tsai*[1]; Y. Wu[1]; J. Yang[1]

1. National Taiwan University of Science and Technology, Taiwan

Solid-state electrolytes (SSEs) with superior Li-ion conductivity are considered as the promising electrolytes for solid-state lithium batteries. However, many superior Li-ion conductivity SSEs have interfacial incomparability with conventional cathode materials as much capacity loss and impedance growth were observed in electrochemical cell testing. Here, we use ab-initio calculations to understand this interfacial incomparability between SSEs and cathodes as a function of state of charge (SoC). State-of-art oxide-based, sulfide-based, and halide-based SSEs and conventional layered-, spinel-, and olivine-structures cathodes are studied. Decomposition energy, chemical reaction energy, electrochemical stability window, and side-reaction products at the SSE/cathode interfaces are calculated. The quantification of the chemical and electrochemical stability between the SSEs and cathodes will be discussed in the presentation.

9:00 AM

(ICACC-S6-010-2023) Mechanistic Analysis of Interface Stability in Solid-State Batteries (Invited)

B. Vishnugopi*[1]; P. P. Mukherjee[1]

1. Purdue University, USA

Lithium metal anodes can potentially enable next-generation batteries with higher energy and power densities compared to conventional lithium-ion batteries. Pairing the lithium metal electrode with a solid electrolyte is considered a promising approach to address interfacial challenges such as dendrite growth and dead metal formation, which are encountered in liquid electrolytes. However, the morphological stability of such solid-solid interfaces in solid-state batteries persists to be a major concern due to the heterogeneous nature of electrochemical-mechanical-transport interactions. In this presentation, the role of microstructural/surface heterogeneities on the morphological evolution of the lithium-solid electrolyte interface including failure modes such as contact loss and filament evolution will be analyzed. The competing effect of reaction kinetics, vacancy diffusion and lithium mechanics on the onset and growth of interfacial voids is discussed.

9:30 AM

(ICACC-S6-011-2023) Towards ceramic-based room-temperature solid-state sodium batteries (Invited)

O. Guillon*[1]; A. Yang[1]; Q. Ma[1]; F. Tietz[1]; F. Dina[1]

1. Forschungszentrum Juelich, IEK-1, Germany

Compared to their lithium counterparts, solid-state sodium batteries (SSNBs) are regarded as less mature technology despite some clear advantages which are going to be detailed in this talk. Solid oxide electrolytes $Na_{3.4}Zr_2Si_{2.4}P_{0.6}O_{12}$ (NZSP) and $Na_5YSi_4O_{12}$ (NYS) have total conductivity of several mS cm^{-1} at 25°C, higher than reported values for polycrystalline Li-ion oxide conductors. In addition, their stability, convenience of handling and processability as layers are significantly better. For example, NYS can be suspended in an aqueous slurry, tape cast and freely sintered up to almost full density. Inhibition of dendrite-growth in SSNBs has long been a challenge to the field. In the present study, with simply sticking sodium metal to ceramic pellets and without extra pressure applied during operation, the critical current density (CCD) of Na/NZSP/Na symmetric SSNBs reaches 9 mA cm^{-2} at 25°C. An interesting Na dendrite-growth behavior along the surface rather than through the ceramic is also found for NZSP. Operando investigations and in situ SEM microelectrode experiments were conducted to reveal the Na plating mechanism.

All-solid-state Batteries IV

Room: Coquina Salon E (North Tower)
Session Chairs: Bairav Sabarish Vishnugopi, Purdue University; Palani Balaya, National University of Singapore

10:20 AM

(ICACC-S6-012-2023) Solid electrolyte (SE) coatings on high oxidation state cathode active materials (Invited)

R. M. Laine*[1]; M. Yu[2]; E. Temeche[1]; T. G. Brandt[1]

1. University of Michigan, Materials Science and Engineering, USA
2. University of Michigan, Macromolecular Science and Engineering, USA

High oxidation state cathode active materials often degrade with cycling due to leaching of active components to liquid electrolytes. In some instances, these leached components (Mn^{2+}) migrate and react with anode active materials and liquid electrolyte resulting in greatly reducing performance. Considerable efforts are ongoing, directed towards introducing coatings that allow efficient Li^+ diffusion during cycling coincident with electronic communication yet mitigate loss of active materials. In this presentation, we discuss the effects of coatings made using lithiated SE nanopowders to modify active material surfaces. Such coatings are easily applied using traditional ceramics processing approaches. We find that such coatings on NMC and LMNO for example greatly limit deleterious degradative processes. In some instances, the coatings provide long-term protection via processes that improve Li^+ transport, capacities, and cycling stability by eliminating active material dissolution. These are the subjects of this presentation. The work to be presented is supported by NSF DMR Grant 1926199 and Mercedes Benz Research and Development North America (Redford, MI).

10:50 AM

(ICACC-S6-013-2023) First-principles thermodynamics of ion order/disorder in many-component oxides and their interfaces (Invited)

S. Kasamatsu*[1]

1. Yamagata University, Japan

Microscopic understanding of ion ordering/disordering and interfacial space charge phenomena is essential for optimal design of materials for next generation solid-state electrochemical devices. First-principles calculations are highly expected as a method to theoretically visualize what is happening at the atomic scale. However, it is extremely challenging to perform modeling based on thermodynamic and electrochemical principles using only first-principles calculations. This is because of the high computational cost of first-principles calculations coupled with the huge number of configuration degrees of freedom coming from multiple ionic and defect species in materials used for electrochemical devices. To tackle this issue, we have developed the abICS (ab Initio Configuration Sampling) framework, which combines first-principles calculations, machine learning, and highly parallel statistical thermodynamics methods and applied it to ionic conductors and their interfaces. In the talk, I will introduce the methodology as well as its application to understanding water uptake in acceptor-doped $BaZrO_3$ and the interfacial space charge effect at electrode/solid electrolyte interfaces.

11:20 AM

(ICACC-S6-014-2023) Solid-state synthesized Argyrodite Li_6PS_5Cl Electrolytes with Si substitution for Enhanced Stability with Li-metal Anode

J. Song*[1]; J. Wang[2]; X. Dai[2]; D. Kim[2]; D. Kim[1]

1. Korea Advanced Institute of Science and Engineering (KAIST), Dept. of Mater Sci & Eng, Republic of Korea
2. University of New South Wales, School of Chemistry, Australia

Safety concerns of flammable organic liquid electrolytes present a significant challenge for the development of rechargeable Li-ion batteries. The use of non-flammable solid-state-electrolytes (SSE) plays an important role in the development of high-energy-density Li metal anodes. Argyrodite sulfide-based electrolytes are among the promising candidates of solid electrolytes due to high ion conductivity. However, the resistance at the interface caused by the reduction reaction between Li-metal and the electrolyte presents a long-term challenge. Indeed, the side reaction is a major factor in Li dendrite formations and the deterioration of battery performance. In this study, improved stability with Li metal anode was confirmed by partially substituting Si^{4+} for the P^{5+} site of Li_6PS_5Cl. The effect of Si^{4+} concentration on crystal structure and the reduction of interfacial resistance were characterized by X-ray diffraction and electrochemical impedance spectroscopy. Also, the cycling stability at the Li/SSE interface was demonstrated with the enhanced battery performance. The Si-based chemical species at the interface between $Li_{6+x}P_{1-x}Si_xS_5Cl$ and Li-metal was characterized by Raman spectroscopy and X-ray photoelectron spectroscopy.

11:40 AM

(ICACC-S6-015-2023) Garnet-based Solid-State Li Batteries by advanced sintering techniques

M. Ihrig*[1]; A. M. Laptev[1]; T. Mishra[1]; M. Finsterbusch[1]; D. Fattakhova-Rohlfing[1]; O. Guillon[1]

1. Forschungszentrum Juelich, IEK-1, Germany

Garnet-based $Li_7La_3Zr_2O_{12}$ (LLZ) solid-state Li batteries (ASBs) provide high intrinsic safety and energy density, and wide operational temperature range. Fabrication of such ASBs requires sintering, and the cathode material (CM) and electrolyte should be co-sintered. However, conventional sintering of CM and LLZ with long dwell time and high temperature leads to non-conductive phases and decomposes the CM. Advanced sintering techniques with short dwell time and low sintering temperatures are required to maintain phase purity. Ultrafast high-temperature sintering (UHS) can be used to sinter bulk, sintering additive-free LLZ pellets within seconds, kinetically inhibiting side reactions. Despite the short sintering, a mechanically stable, phase pure, and sufficiently dense LLZ with good ionic conductivity is obtained. UHS is predestined for co-sintering of LLZ and CMs, as it could help to overcome thermodynamic limitations and avoid the formation of a diffusion-based secondary phase. It could pave the way for a completely new type of ASBs. An alternative technique is Field Assisted Sintering Technique / Spark Plasma Sintering. The use of high mechanical pressure allows to lower the sintering temperature to 675 °C and reduce the dwell time to 10 min for the fabrication of different functional $LiCoCO_2$ (LCO)/LLZ pellets e.g. dense and thick LCO/LLZ cathodes providing excellent electrochemical properties.

S8: 17th International Symposium on Advanced Processing and Manufacturing Technologies for Structural and Multifunctional Materials and Systems (APMT17)

Advanced Composite Manufacturing Technologies, Hybrid Processes I

Room: Coquina Salon F (North Tower)
Session Chairs: Nobuhito Imanaka, Osaka University; Thi Mai Dung Do, Nagaoka University of Technology

8:30 AM

(ICACC-S8-007-2023) Pressureless sintering in hydrogen of hot isostatic pressed Al_2O_3 prepared from oxidized AlN powder (Invited)

C. Balazsi*[1]; M. Furko[1]; K. Balazsi[2]

1. ELKH Centre for Energy Research, Hungary
2. Centre for Energy Research HAS, Thin Film Physics, Hungary

The effect of hydrogen on the structural and mechanical properties of hot isostatic pressed Al_2O_3 prepared by oxidized AlN powder was studied. The micrometer size AlN powder was oxidized in ambient air at 900° C for different times (3, 6, 10 and 20 hours) and sintered by hot isostatic pressing at 1700°C for 5h. Pressureless sintering in H_2 gas at 1800° C for 10 hours was applied on all sintered samples. It was shown, that the oxidation caused the core-shell AlN/Al_2O_3 structure and the amount of Al_2O_3 increased with oxidation time of AlN power. All samples exhibited a main a-Al_2O_3 phase after hot isostatic pressing and pressureless sintering at H_2 atmosphere. No metastable "heat-treatment" oxide phases of Al_2O_3 were present in the samples owing to the high temperatures involved in the processing. Sintering in H_2 helped the full transformation of AlN to a-Al_2O_3 phase and better densification of samples. Therefore, the hardness of well sintered samples was 17 – 18 GPa with apparent densities between 3.11 and 3.39 g/cm^3.

9:00 AM

(ICACC-S8-008-2023) Manufacturing of SiC_f/SiC and Al_2O_{3f}/Al_2O_3 ceramic matrix composites by microwave assisted chemical vapour infiltration

Z. Li*[1]; J. Binner[2]

1. University of Birmingham, United Kingdom
2. University of Birmingham, Ceramic Science & Engineering, United Kingdom

A novel microwave assisted chemical vapour infiltration (MW-CVI) method is being used to manufacture both SiC_f/SiC and Al_2O_{3f}/Al_2O_3 ceramic matrix composites (CMCs). The characteristic volumetric heating of the fibre preform with an inverse temperature profile during MW-CVI should significantly reduce the cost of the manufacture of composites. SiC fibre shows good microwave heating behaviour and both plate and tubular shaped SiC_f/SiC composites were produced by MW-CVI with the use of methyltrichlorosilane (MTS) as the precursor. In contrast, alumina-based materials generally do not absorb microwave energy easily at low temperatures. Thus, heating of the alumina fibre preform remains a main challenge and is achieved by the use of microwave absorbing coating materials through either dip-coating or a continuous roll-to-roll process. The dielectric properties of the coated alumina fibre preforms were characterised as a function of the temperature and the microwave heating behaviour of the coated fibre preforms was investigated. Al_2O_{3f}/Al_2O_3 CMCs were produced by MW-CVI with aluminium tri-sec butoxide (ATSB) used as the precursor.

9:20 AM

(ICACC-S8-009-2023) Study of metalized silicon nitride substrate reliability under thermal cycling test using digital image correlation

N. M. Chu*[1]; H. Miyazaki[1]; K. Hirao[1]; M. Fukushima[1]

1. National Institute of Advanced Industrial Science and Technology (AIST), Multi-material Research Institute, Japan

The developed silicon nitride (Si_3N_4) with high thermal conductivity and superior mechanical properties has been considered as a new substrate in power modules such as in electric vehicles. Crack formation in Si_3N_4 active metal brazing (AMB) ceramic substrate and delamination of copper layers on the substrate through thermal cycling test from -40°C to 250°C has been studied in previous research by observing and analyzing sample before and after the test in real experiments and finite element method (FEM) simulation. In this research, by using a digital image correlation (DIC), the strain on metalized Si_3N_4 substrate induced during thermal cycling test from -40°C to 250°C was measured and investigated at 10, 50, 100, 500, 1000, and 1500 thermal cycles. Besides DIC, other evaluation methods of laser scanning microscopy (OLS4100) and scanning acoustic tomograph (SAT) were also conducted. From the experimental results, the thermal hysteresis with strain of metalized Si_3N_4 substrate due to nonlinear physical properties was observed, which was attributed to the plastic deformation of copper layer. The thermal hysteresis curve changed slightly with increasing number of thermal cycles; it was assumed that the work hardening had occurred on copper layer. This research result can be utilized to develop a reliable metalized Si_3N_4 substrate for power modules in the future.

Advanced Powder Synthesis and Processing

Room: Coquina Salon F (North Tower)
Session Chair: Ngo Chu, National Institute of Advanced Industrial Science and Technology (AIST)

10:20 AM

(ICACC-S8-011-2023) SPS densification and phase stability of Y_2O_3 - cBN composites

J. Li*[1]; Y. Wu[1]

1. Alfred University, Kazuo Inamori School of Engineering, New York State College of Ceramics, USA

Cubic boron nitride (cBN) has high hardness, thermal conductivity, large band gap, and secondary electron emission. Owing to its unique properties, cubic BN has attractive applications for optoelectronic devices and superhard materials. In this work, yttrium oxide -cubic boron nitride composites were prepared by the spark plasma sintering (SPS) method. The cubic boron nitride with particle sizes of 1-3μm and ~250 nm were used to mix with nano-sized yttrium oxide powders as the starting materials. The concentration of boron nitride varied from 30 to 50 vol%. The mixed powders were sintered at different temperatures for 5 min under a pressure of 104 MPa. We studied the effects of the particle sizes of the boron nitride and sintering parameters on the phase transformation of BN and the densification behaviors of the Y_2O_3-cBN composite ceramics.

10:40 AM

(ICACC-S8-012-2023) Influence of Powder Feedstock Characteristics and Process Parameters on Extrusion-based 3D Printing of Magnetocaloric Structures

V. Sharma*[1]; L. L. Balderson[2]; M. Dey[3]; S. Gupta[3]; R. Hadimani[1]; H. Zhao[1]; R. Barua[1]

1. Virginia Commonwealth University, Department of Mechanical & Nuclear Engineering, USA
2. University of Virginia, Materials Science and Engineering, USA
3. University of North Dakota, Department of Mechanical Engineering, USA

Magnetic refrigeration is a sustainable, energy-efficient alternative to the conventional vapor-compression cooling technology. A key challenge in manufacturing magnetocaloric devices is that lack of fabrication methods for shaping the brittle caloric alloys into thin-walled channeled regenerator structures with optimized heat transfer properties, and gradient transformation temperature, while preserving the functional response of the materials system. To this end, we have recently developed a novel extrusion-based additive manufacturing (AM) method to 3D print spatially designed porous magnetocaloric structures with channel dimensions in the range 150-800 μm. Building on this proof-of-concept demonstration, research efforts pertaining to AM process development using three precursor magnetocaloric powders will be presented - (1) $La_{0.6}Ca_{0.4}MnO_3$ nanoparticles (dia~10 nm) prepared by Pechini Sol-Gel synthesis guidelines for minimizing porosity and lack-of-fusion defects in the 3D printed magnetocaloric parts.

11:00 AM

(ICACC-S8-013-2023) AlON Powder via dynamic thermochemical method

H. Boussebha*[1]

1. Sakarya University, Materials Science and Engineering, Turkey

Being a major candidate for armor and infrared windows, Aluminum Oxynitride (AlON) is a ceramic with elite mechanical properties and excellent transparency. AlON has been for long, a subject of interest to determine an inexpensive method allowing the synthesis of its powder. The conventional process to synthesize AlON requires the use of highly pure alumina (Al_2O_3) and aluminum nitride (AlN) as well as excessive energy since AlON starts forming at 1640°C and is considered instable below 1750°C. In this study, the synthesis of aluminum oxynitride has been investigated via dynamic thermochemical method (DTM). DTM is a patented novel approach that has been deemed effective in the synthesis of technical ceramics. DTM is based on the carbothermal reduction nitridation method (CRN) with the difference of the synthesis taking place in a rotating kiln allowing the synthesis of homogenous spherical powders in a relatively shorter time. A conversion to AlON was obtained from aluminum hydroxide $Al(OH)_3$ after a continuous heating and two stages holding at 1100°C for 1.25h and 1500°C for 2h under flowing ammonia (NH_3) and propene (C_3H_8). The obtained powders were investigated via XRD and FESEM.

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

Material Technologies for Enhanced Accident Tolerance LWR Fuels and Core I

Room: Ballroom 4 (South Tower)
Session Chair: Ghatu Subhash, University of Florida

8:30 AM

(ICACC-S13-009-2023) Silicon carbide composite technology for accident-tolerant fuels – recent progress and updated R&D needs (Invited)

Y. Katoh*[1]; T. Koyanagi[1]; P. Xu[2]; C. Deck[3]; K. Shirvan[4]; L. Snead[5]

1. Oak Ridge National Laboratory, USA
2. Idaho National Lab, USA
3. General Atomics, USA
4. Massachusetts Institute of Technology, USA
5. Stony Brook University, USA

Silicon carbide composite-based materials remain as arguably the most attractive cladding option for the accident-tolerant light water reactor fuels (ATF) due to their desirable attributes including the exceptional high temperature capability and inertness in steam environment. Moreover, these composites are considered the enabling materials for advanced high temperature reactor concepts including those adopting helium, sodium, or salt cooling. This presentation will give an overview of the progress of this material technologies with the main emphasis on the remaining research and development opportunities toward the technology deployment in light water reactors and enabling the ceramic composite core design concepts for advanced high temperature reactors. This work was supported by Accident Tolerant Fuels Program, Office of Nuclear Energy, United States Department of Energy under contract DE-AC05-00OR22725 with Oak Ridge National Laboratory, managed by UT Battelle, LLC

9:00 AM

(ICACC-S13-010-2023) Advanced Ceramic Matrix Composites for Extreme Environments in Nuclear Applications (Invited)

C. Deck*[1]; L. Borowski[1]; S. Gonderman[1]; R. Haefelfinger[1]; A. Giles[1]; A. Moore[1]; C. Hill[1]; S. Oswald[1]; G. Jacobsen[1]; J. Gazza[1]

1. General Atomics, Nuclear Technologies and Materials, USA

General Atomics Electromagnetic Systems is developing SiGA® technology, an engineered SiC fiber reinforced, SiC matrix (SiC-SiC) composite for advanced nuclear applications. Efforts are focused on fabrication, scale-up, and demonstration of performance in extreme environments, and include the design of engineered structures to best leverage CMC properties. To demonstrate SiGA® fuel cladding for use in existing light water reactors, length scale-up is being coupled with increased fabrication yields and the application of integrated components to reduce handling steps to improve overall fabrication efficiency. These improvements, along with increasingly representative irradiations in the ATR ATF test loop and demonstration of engineered features that enable more economic fuel performance, are part of a comprehensive approach to position SiGA composites for commercial deployment. SiGA technology is also being leveraged to demonstrate new fabrication capabilities to deploy ceramic composites in space nuclear applications. These require even higher temperature limits than SiC, and new characterization methods were developed in order to quantify the performance in these extreme environments. Recently, GA-EMS has demonstrated a high temperature hydrogen exposure furnace to verify the resilience of ultra-high temperature carbide-based composites in conditions relevant to nuclear thermal propulsion.

9:30 AM

(ICACC-S13-011-2023) Ceramic coatings by chemical vapor deposition towards development of accident tolerant fuel claddings (Invited)

H. Katsui*[1]; R. Usukawa[1]; S. Kondo[2]; K. Shimoda[3]; K. Yabuuchi[5]; S. Ebara[4]

1. National Institute of Advanced Industrial Science and Technology (AIST), Multi-Material Research Institute, Japan
2. Tohoku University, Institute for Materials Research, Japan
3. National Institute for Materials Science (NIMS), Research Center for Structural Materials, Japan
4. Tohoku University, Department of Quantum Science and Energy Engineering, Japan
5. Kyoto University, Institute of Advanced Energy, Japan

As promising next-generation structural materials for nuclear fuel cladding, SiC and SiC/SiC composites are exposed to extremely corrosive environments with high-temperature and high-pressure water and steam, in which irradiation-accelerated corrosion is also an issue. Since SiC/SiC includes impurities and grain boundaries that are problematic for the corrosion resistance, anticorrosive ceramic coatings have gained much attention. However, the deposition of ceramic layers with excellent integrity is still challenging because the dissimilarity of thermophysical properties between the ceramic layers and SiC often causes surface cracking and delamination under thermal stress. We have been developing ceramic coatings on SiC and SiC/SiC by chemical vapor deposition (CVD) that is a versatile method that can readily form dense protective layers with multiscale thickness. In this presentation, multilayer coatings with an alumina top coat formed on SiC substrates by a laser-assisted CVD technique are demonstrated. Effects of deposition parameters on the microstructure, interfacial structure, and integrity of the multilayer coatings are discussed. We will also introduce our recent activities on the development of CVD-coatings on SiC components having complex shapes. This work was supported by Japanese MEXT Innovative Nuclear Research and Development Program (Grant Number JPMXD0222682488)

Material Technologies for Enhanced Accident Tolerance LWR Fuels and Core II

Room: Ballroom 4 (South Tower)
Session Chair: Kurt Terrani, Ultra Safe Nuclear

10:20 AM

(ICACC-S13-012-2023) Thermodynamic modelling of reactions between SiC and coating materials (Invited)

M. to Baben*[1]; P. J. Spencer[2]

1. GTT-Technologies, Germany
2. The Spencer Group, USA

SiC/SiC composites are a promising accident-tolerant fuel cladding material receiving significant attention in the last years. To limit the formation of SiO_2, its dissolution in water and the formation of volatile CO and SiO in nominal operating conditions and during an accident, coatings play an important role. However, it must be ensured that reactions between SiC and the coating materials are avoided both at operating conditions and during an accident. Thermodynamic modelling can be used to describe and improve thermal stability of base material-coating interfaces, as shown for Zr / Cr_2AlC by Mraz et al. [S. Mráz, M. Tyra, M. to Baben, M. Hans, X. Chen, F. Herrig, K. Lambrinou, J.M. Schneider, Journal of the European Ceramic Society 40 (2020) 1119]. Here, a materials informatics approach based on the same methodology is used to identify coating materials for SiC with promising properties. For this, high-throughput ChemApp calculations and FactSage calculations are performed based on the thermochemical database SpMCBN and aiMP/aiOQ. Stability of the interface, high melting temperatures, low tendency for evaporation in steam or air at high temperature and low solubility in supercritical water are the targeted material properties. Additionally, gaps in the knowledge of thermodynamic properties are pointed out.

10:50 AM

(ICACC-S13-013-2023) Characterization of Heterogeneity, Properties, and Uncertainty Quantification for SiC_f-SiC_m Braided Composite Tubes (Invited)

G. Subhash*[1]; J. Nance[2]; H. Thandaga Nagaraju[1]; B. Sankar[1]; N. Kim[1]

1. University of Florida, Mechanical and Aerospace Engineering, USA
2. University of Florida, Material Science Engineering, USA

Silicon carbide fiber-reinforced silicon carbide matrix composite (SiC_f-SiC_m) tubes are leading candidates for accident tolerant cladding in light water reactors because of their low neutron absorption cross-section, thermal resilience, resistance to irradiation, chemical inertness, etc. For successful implementation of these tubes in nuclear applications the relationship between material properties, manufacturing variables and the variability in the properties must be established. In this research, the microstructural heterogeneity (e.g., variations in fiber diameter, porosity distribution, braid angle imperfections, wall thickness variation, etc.), is quantified and their influence on mechanical strength and failure modes are evaluated under various stress states (compression, flexure, and hoop) for four different weave architectures. The heterogeneity was quantified in terms of probability distributions and Sobol sensitivity analysis was performed to identify the dominant variables influencing the properties. Concurrently, finite element methods coupled with Monte-Carlo simulations were developed to predict the distribution of elastic constants and strength of the composites for various loads. Finally, using the direct micromechanics method, we developed failure envelopes and quantified uncertainty in the strength properties.

11:20 AM

(ICACC-S13-014-2023) Development of CVI/CVD-SiC/SiC Composites for Accident-Tolerant Fuels in LWR Applications

S. Suyama*[1]; M. Ukai[1]; T. Nishimura[1]; T. Takada[1]

1. Toshiba Energy Systems & Solutions Corporation, Japan

CVI-SiC/SiC composites have been investigated for use in nuclear reactor cores because of their inherent low activation, low neutron absorption cross-section, and stability under irradiation due to their high purity and crystalline SiC matrix. After the nuclear power plant accident in Fukushima in March 2011, many research programs started to develop accident-tolerant fuels (ATFs). Toshiba Energy Systems Corporation has been developing material design and process technologies for thin-walled and elongated tubes and boxes made of CVI/CVD-SiC/SiC composites since 2012. However, in Japan, as of 2022, over 10 years after the Fukushima nuclear accident, only 10 nuclear reactors have been restarted. Therefore, we would like to promote the application of ATFs for commercial LWRs to further improve their safety. This study describes progress in the development of process technology for thin-walled and elongated CVI/CVD-SiC/SiC composite-based tubes and boxes. The components are required to have hydrothermal corrosion resistance and good hermetic seal in normal operation environments. They are also required to have a period of durability in a water vapor environment above 1200°C during an accident. In particular, the hydrothermal corrosion resistance properties of CVI/CVD-SiC/SiC composites under BWR conditions, and surface modification and coatings to improve these properties will be presented.

11:40 AM

(ICACC-S13-015-2023) Irradiation testing and technique development for evaluating radiation-induced bowing in SiC/SiC components

C. Petrie*[1]; J. W. Geringer[1]; A. James[1]; C. Deck[2]; T. Koyanagi[1]

1. Oak Ridge National Laboratory, USA
2. General Atomics, USA

Silicon carbide (SiC) fiber–reinforced, SiC ceramic matrix composites (SiC/SiC composites) are a potential accident tolerant fuel cladding material because of their high-temperature strength, dimensional stability under irradiation, minimal neutron absorption, and relatively slow oxidation kinetics in steam and air environments. SiC/SiC materials could also be a near term replacement for current zirconium alloy channel boxes in boiling water reactors if concerns related to lateral bowing due to differential radiation-induced swelling can be resolved. This work summarizes a neutron irradiation experiment that was conducted in the High Flux Isotope Reactor to evaluate the bowing of a miniature SiC/SiC channel box and pressurized water reactor cladding tubes exposed to a fast neutron flux gradient. Surface profilometry was performed using a custom measurement system that utilizes an optical micrometer attached to a precision stage. In addition, a digital microscope was attached to the same stage and used to record the precise positions of fine engraving marks that were etched along all faces of each sample. These measurements will be repeated after irradiation to evaluate local deformations and overall lateral bowing compared with values predicted using high-fidelity modeling and simulation.

S15: 7th International Symposium on Additive Manufacturing and 3-D Printing Technologies

Materials and Process Characterization Tools

Room: Coquina Salon H (North Tower)
Session Chair: Fiona Spirrett, Osaka University

8:30 AM

(ICACC-S15-001-2023) In-situ sensing system of suspension-based additive manufacturing (Invited)

J. Lee[1]; S. Jang[1]; H. Son[1]; S. Park[1]; C. Bae*[1]

1. Korea Institute of Materials Science, Department of 3D printing materials, Republic of Korea

Ceramic additive manufacturing is an innovative technology for directly developing complex ceramic structures, while the challenges remain how to instantly realize defects in ceramic green body before a final object is completely fabricated. Here, we demonstrate a novel in-situ sensing system on the sequential layers developed using layer-by-layer process, quantitatively analyzing reproducible signals on the rheological, curing and mechanical behaviors in a real-time. Given the signals recognized at the steps of ink leveling (I) and curing (II), the rheology and curing shrinkage of 50 vol% silica suspension were U-shaped pattern and small shrinkage resulted from resistances and anchoring of silica powders compared to the M-shaped pattern and large shrinkage of pure resin, respectively. Furthermore, cured ceramic green layers delaminated from Teflon film at the step of detachment (III) continually undergo higher detachment stress owing to higher elastic modulus of solid particles than that of pure resin system.

9:00 AM

(ICACC-S15-002-2023) Ceramic additive manufacturing direct-ink-write and densification measurement needs

A. J. Allen*[1]; R. Maier[2]; F. Zhang[1]; I. Levin[2]

1. NIST, Materials Measurement Science Division, USA
2. National Institute of Standards and Technology, USA

Ceramics additive manufacturing (AM) or 3D printing has the potential to transform the ceramics industry by allowing complex and customized ceramic components to be fabricated efficiently with minimal waste and environmental impacts, compared to conventional ceramic manufacture. However, to realize this potential, the multiple complex materials phenomena involved need to be better understood and controlled. Taking just one ceramics AM route as an example: direct-ink-write jet printing, issues that need to be considered include feedstock morphology, ceramic particle suspension slurry rheology as a function of pressure and deposition rate, changing physical properties with time of the deposited material due to, e.g., evaporation effects, the physics and chemistry of de-binding and sintering, and overall efficiency in materials, energy, time and cost. In this paper, we will describe progress in evaluating these issues using a combination of in-situ materials characterization methods, both in regard to the direct-ink-write ceramics AM route and also to possible novel densification routes such as cold sintering. Relevant standards development needs will also be discussed.

9:20 AM

(ICACC-S15-003-2023) Tailoring the mechanical properties of alumina ceramics through 3D-printing

J. Schlacher*[1]; A. Hofer[1]; S. Geier[2]; I. Kraleva[1]; S. Nohut[2]; M. Schwentenwein[2]; R. Bermejo[1]

1. Montanuniversitaet Leoben, Department of Materials Science, Austria
2. Lithoz GmbH, Austria

In this work, the potential of the Lithography-based Ceramic Manufacturing (LCM) technology to design novel alumina-based ceramic systems is discussed. The first approach demonstrates the use of the LCM technology to print alumina-based ceramic parts with superior strength. The combination of alumina (outer regions) with alumina-zirconia (ZTA) layers introduces compressive residual stresses in the surface layers. A characteristic biaxial strength as high as 1 GPa is measured on the alumina-based multilayers, as compared to 650 MPa in bulk alumina, the difference corresponding to the magnitude of in-plane residual stresses in the external alumina layers. The second design approach deals with the possibility of fabricating highly-textured alumina ceramics (~80% texture degree) through the combination of the stereolithographic vat polymerization and the Templated Grain Growth technique. A sound characteristic strength of ~650 MPa has been measured for 3D-printed textured alumina, which may open new ways to fabricate damage-tolerant (more reliable) 3D-printed ceramics by mimicking the nature (i.e. mussels). In addition, recent research activities on the mechanical properties of 3D-printed alumina ceramics with tailored porosities will be shown.

9:40 AM

(ICACC-S15-004-2023) The process-structure-property relationship of additive manufacturing mullite material

J. Tsai[1]; D. Singh*[1]

1. Argonne National Laboratory, Applied Materials Division, USA

A mullite material was successfully additive manufactured from digital light processing (DLP) with the combination of polymer-derived ceramic (PDC). The printed geometry involves a one-step process of pyrolysis and sintering under a plain atmosphere to achieve an average of 76% ceramic yield and linear shrinkage of 12%. The thermal profile was carefully controlled to obtain consistent phases in which the XRD shows a mullite phase with the chemical composition of either $Al_{2.25}O_{4.87}Si_{0.75}$ or $Al_{4.8}O_{9.6}Si_{1.2}$. In addition, the Weibull analysis was conducted on the printed flexural specimens, showing a Weibull modulus (m) of 13 with characteristic strength (σ_0) of 95 MPa. Lastly, large-scale prototypes are printed concurrently with fine-resolution (25 μm) without design/structure compromise. The work has been supported by the ANL's LDRD program, project number 2022 – 0078.

Vat Photopolymerization / Substrate Stereolithography I

Room: Coquina Salon H (North Tower)
Session Chair: Martin Schwentenwein, Lithoz GmbH

10:20 AM

(ICACC-S15-005-2023) Micro-printing of metallized ceramics for microelectronics (Invited)

K. Porter*[1]; A. Yu[1]; E. Wernick[1]; E. Stonkevitch[1]; M. O'Masta[1]; P. Bui[1]; T. Schaedler[1]

1. HRL Laboratories, USA

This talk explores the development of an additive micro-manufacturing technology for ceramics. We demonstrate 3D printed parts on the cm scale with 10 μm sized features, using Micro Projection Stereolithography. Millions of channels (i.e., vias) – printed directly into the ceramic – can be subsequently metallized to provide electric pathways between part faces. These combined features engender a rethinking of the 3D integration of microelectronic subsystems from the unprecedented via routing and packaging options realized. We discuss the challenges overcome in micron-scale printing of preceramic resins that have allowed us to print arrays of straight, angled and curved vias with a pitch of less than 20μm. Additions of varying amounts of different nanoparticles enable tailoring the coefficient of thermal expansion to match silicon or III-V semiconductors. Lastly, strategies to achieve the extremely tight tolerances required for integration with semiconductor devices are presented. This material is based upon work supported by the DARPA under Contract No. N66001-20-C-4012. Any opinions, findings and conclusions or recommendations expressed in this material are those of the author(s) and do not necessarily reflect the views of the DARPA.

10:50 AM

(ICACC-S15-006-2023) How to eliminate delamination cracks in ceramics manufactured via vat-photopolymerization: A practitioner's perspective

W. Yared*[1]; R. Gadow[1]

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

Vat-photopolymerization of ceramics allows the creation of ceramic parts with unprecedented resolution for various applications, from medical tooling to archeological restoration. However, the manufacturing of a crack-free sintered ceramic part using vat-photopolymerization has been an arduous challenge in both industrial and academic research. Thus, as a result of numerous experimental and analytical studies, this contribution provides a guide for the elimination of delamination cracks in sintered ceramic parts manufactured via vat-photopolymerization. Photo-rheology, UV/Vis spectrophotometry and multiple microscopy techniques were used to critically analyze the formation of delamination cracks, their culprits and the possible methods to eliminate such cracks. It was found that proper particle dispersion, sufficient air extraction, optimized resin formulation, optimized printing parameters and sound design principles jointly contribute to the manufacturing of a delamination cracks-free ceramic part using vat-photopolymerization. In this oral contribution, the author will address each one of the aforementioned factors from a practitioner's perspective, offering practical knowledge for a more successful additive manufacturing of ceramics using vat-photopolymerization.

11:10 AM

(ICACC-S15-007-2023) Stereolithography-based additive manufacturing of polymer-derived structures exhibiting catalytic activity

J. Eßmeister*[1]; L. Schachtner[1]; A. Fuchsberger[1]; E. Szoldatits[3]; K. Föttinger[3]; A. A. Altun[2]; A. Lale[2]; M. Schwentenwein[2]; T. Konegger[1]

1. TU Wien, Institute of Chemical Technologies and Analytics, Austria
2. Lithoz GmbH, Austria
3. TU Wien, Institute of Materials Chemistry, Austria

Polymer-derived ceramics exhibit outstanding properties and allow for a direct introduction of metal centers into the ceramic matrix through chemical modification, rendering them ideal candidate materials for catalysis applications. To overcome processing-related limitations of conventional shaping techniques, additive manufacturing such as stereolithography is highly suitable to obtain macroporous structures for high product flow. The fabrication of complex polysiloxane-derived SiOC lattice structures is demonstrated. Two distinct approaches to modifying the polysiloxane-based printing system were developed. First, the addition of SiC particles to the printing resin significantly increased the stability of the specimens and the freedom of design during the process. This combination allowed for the production of crack-free, high-density SiOC/SiC composites. Second, a chemical modification of the preceramic polymer was carried out using metalorganic compounds, facilitating the in-situ generation of nano-scaled metal particles during polymer-to-ceramic conversion. Subsequently, the effect of printing resin composition, printing parameters, and pyrolytic conversion conditions on phase development and microstructural evolution were elucidated for both material systems. Finally, the influence of these parameters on the catalytic activity of the produced materials was determined.

11:30 AM

(ICACC-S15-008-2023) Novel routes for the fabrication of carbide components via sol-gel-based additive manufacturing

A. Zanini[1]; M. Y. Moshkovitz[2]; S. M. Carturan[3]; S. Corradetti[3]; P. Colombo[1]; S. Magdassi[2]; G. Franchin*[1]

1. University of Padova, Industrial Engineering, Italy
2. Institute of Chemistry and Center for Nanoscience and Nanotechnology, The Hebrew University of Jerusalem, Israel
3. Istituto Nazionale di Fisica Nucleare, Laboratori Nazionali di Legnaro, Italy

Additive manufacturing combined with sol-gel processing has gained worldwide attention as a technique for the fabrication of a large panel of materials. Sol-gel-based feedstocks rely on the assembly of the building blocks in the network, providing a versatile approach for the control on the arrangement of the structural units and offering new possibilities to synthesize materials with tuneable properties. Moreover, chemical and physical properties of the sols can be tailored to suit different additive manufacturing techniques with advantages over feedstock based on particle suspensions. This work has been devoted to carbide materials, generally difficult to shape through AM approaches; particularly, structured titanium carbide has attracted an increasing interest as a target material for the production of radioisotopes in the ISOL facilities. However, the release of radioisotopes relies on the presence of a network of open, interconnected pores enhancing the diffusion from the target. To fulfil this requirement, the sol-gel route allows for the tuning of the textural properties; here, a protocol for the fabrication of hierarchical porous titanium carbide components was developed. Moreover, by playing with the choice of the structural motifs, this approach allows for a large variety of modifications and can be extended to the fabrication of different carbide-based materials.

11:50 AM

(ICACC-S15-009-2023) Ti-6Al-4V/hydroxyapatite co-printing using digital light processing

C. Gal*[1]; J. Kim[2]; Y. Choi[1]; H. Park[1]; A. Sung[1]; H. Yun[1]

1. Korea Institute of Materials Science, Republic of Korea
2. Pusan National University, Republic of Korea

Multi-material components are economic and they allow us to combine the properties of two or more materials. Therefore, the need to fabricate a component with dissimilar materials and structures is increasing in various fields, such as biomedical engineering, electronics, and aerospace, among others. However, fabricating a multi-material component with high resolution and precision using general additive manufacturing technologies is challenging. Herein, we investigated the new applicability of digital light processing 3D printing technology for multi-printing with Ti-6Al-4V and hydroxyapatite. By observing the rheological, sedimentation, and UV-curing behaviors, the conditions of both UV-curable slurry preparation and the 3D printing process were optimized for each material. We printed 3D structures with good resource efficiency and high structural accuracy. The cross-contamination between the two materials was also controlled by applying a novel washing process. As a result, multi-structure of Ti-6Al-4V/hydroxyapatite was printed using digital light processing. We believe that the proposed system will potentially provide stereolithographic co-printing with dissimilar materials.

S16: Geopolymers, Inorganic Polymers and Sustainable Construction Materials

Geopolymers made from or with Waste Materials I

Room: Coquina Salon C (North Tower)
Session Chair: Waltraud Kriven, University of Illinois at Urbana-Champaign

8:30 AM

(ICACC-S16-001-2023) Chemical resistance to the acidic environment of metakaolin-based binders containing spent corundum grit

G. Dal Poggetto*[1]; C. Leonelli[2]

1. University of Modena and Reggio Emilia, Engineering, Italy
2. University of Modena and Reggio Emilia, Department of Engineering Enzo Ferrari, Italy

The resistance to acid environment of metakaolin-based geopolymers with spent corundum grit is proposed. With the aim to decrease the amount of waste to be disposed of in landfills, novel geopolymer formulations containing spent Al_2O_3 grit were designed taking advantage to the well-known acid resistance of corundum. Geopolymeric mix with different percentages and with different grain size of recycled corundum powders up to 50 wt% on dry basis and the pure metakaolin were prepared. Fourier transform infrared spectroscopy (FTIR) has allowed us to verify the bonds formation between the geopolymeric matrix and the recycled corundum powders. The consolidation of the materials has been confirmed by pH and conductivity after 28 days of curing at room temperature. After 28 days, the geopolymers were tested in an acid environment. Sulfuric, nitric and hydrochloric acids were used all to the same normality. The tests were conducted by immersing the geopolymers in acids respecting a ratio between surface area and volume of 8. The samples remain immersed in acid for 7 days observing the physical changes. The tests were performed on a minimum of 5 samples for each formulation. After 7 days, the extracted samples were weighed and subjected to compression stress tests. Finally, changes in the network were assessed through SEM / EDS and XRD.

8:50 AM

(ICACC-S16-003-2023) Characterization and synthesis of phosphogypsum reinforced geopolymer composites fabricated from industrial wastes.

H. Majdoubi*[1]; S. Sbi[1]; M. Nadi[2]; M. Bouchaib[1]; A. H. Jones[1]; H. Hannache[1]; Y. Tamraoui[1]

1. University Mohammed 6 Poytechnics, MSN, Morocco
2. University Hassan II Casablanca, Chimie, Morocco

Phosphogypsum (PG) constitute the principal by-product of the phosphate industry which largely restricts its wide development. The incorporation of PG as a reinforcement agent into geopolymer cement, can play however a significant role to enhance their mechanical and microstructural properties and to develop a sustainable geopolymer– PG based circular economy. In this study, geopolymer reinforced PG composites were prepared at different PG content according to PG/MK ratio in the range of 0-1. The mechanical properties of the composites were tested under compressive and flexural strength. The results show a significant enhancement of compressive and flexural strength from 40.14 MPa and 5.13 MPa to MPa 48.43 and MPa MPa 9.09 respectively with addition of MPa 20.6% MPa and 70.16% compared standalone geopolymer. This improvement was linked systematically with crack self-healing mechanism due to PG rod-like morphology and its high adhesion to the geopolymer matrix. The heavy metal leaching test shows that most of the developed acid-PG geopolymer composites have a leaching value below international recommendations. Thus, geopolymer-PG composites can be an efficient and sustainable alternative to conventional building materials and can be extended to various industrial applications.

Geopolymers made from or with Waste Materials II

Room: Coquina Salon C (North Tower)
Session Chair: Khadija Qureshi, Mehran University of engineering and Technology Jamshoro

10:20 AM

(ICACC-S16-005-2023) Properties of Potassium Activated Metakaolin and Fly Ash Based Geopolymer Mortars and Composites

R. Abufarsakh*[1]; H. Noorvand[1]; G. Arce[2]; M. Hassan[1]; S. Subedi[1]; O. D. Huang[3]; M. Radovic[3]

1. Louisiana State University, USA
2. Virginia Department of Transportation, USA
3. Texas A&M University, Materials Science & Engineering, USA

Geopolymers (GP) are inorganic aluminosilicate polymers manufactured by activating an aluminosilicate source (i.e., metakaolin, fly ash, etc) with an alkali silicate activator solution. To date, researchers have studied a combination of metakaolin and fly ash as GP precursors activated with sodium silicate and sodium hydroxide solutions. To this end, potassium hydroxide activated metakaolin and fly Ash (MKFA) Based GPs were investigated. Aspects of the material composition studied include the effect of metakaolin and fly ash content on the workability and compressive strength of GP mortars. Furthermore, silica fume and a combination of polyvinyl alcohol (PVA) and/or polypropylene (PP) fibers totaling 1.75 vol.% were added to the best-performing MKFA mortar to produce geopolymer composites. Based on the workability and 28-day compressive strength results, the mortar with 50% metakaolin and 50% fly ash (i.e., $MK_{50}FA_{50}$ K321) presented the highest compressive strength of 10.8 MPa with a flow spread diameter of 180.4 mm. Furthermore, 1.25%PVA-0.5%PP presented the highest compressive strength of 21 MPa. However, uniaxial tensile test results revealed that 1%PVA-0.75%PP presented the highest tensile strain capacity of 0.36% while having a tensile strength of 2.1 MPa. An improvement was seen when the PVA fibers were partially replaced with PP fibers.

10:40 AM

(ICACC-S16-006-2023) Alkali Activation of Discarded Pharmaceutical Glass for Novel 'Unfired' Structural and Functional Materials (Invited)

G. Tameni[1]; F. Cammelli[1]; M. Mahmoud[2]; J. Kraxner[2]; H. Elsayed[1]; E. Bernardo*[1]

1. University of Padova, Department of Industrial Engineering, Italy
2. Alexander Dubcek University of Trencin, FunGlass, Slovakia

Covid-19 has dramatically increased the demand for pharmaceutical containers and the amounts of related waste. This paper deals with the upcycling of discarded pharmaceutical glass into various porous ceramics, starting from the activation of fine powders suspended in relatively weak alkaline solutions (2.5 M NaOH/KOH). The alkaline attack determines the gelation, according to hydration of glass surfaces, followed by condensation at 40-70 °C ('cold consolidation'). Alkali are mostly expelled from the gel, according to the formation of soluble hydrated carbonates. The mutual binding of activated powders may be exploited for the encapsulation of other inorganic waste, such waste foundry sand and residues from the cutting and polishing of clay bricks. The waste mixtures led to products similar to facing bricks directly after cold consolidation or after application of low temperature (800 °C) firing. As an alternative, activated suspensions of glass powders, at the early stages of gelation, may undergo direct foaming (supported by the use of sodium perborate additive), for the determination of highly porous foams, or even direct ink writing, for the determination of reticulated scaffolds. Such scaffolds were successfully used as sorbents for dye removal from contaminated waters; the inclusion of TiO2 particles in the starting slurries enabled photocatalytic die degradation.

11:10 AM

(ICACC-S16-007-2023) Raw earth stabilization by alkali-activated slag: Mechanical study (Invited)

É. Prud'homme*[1]; U. De Filippis[1]; S. Meille[2]

1. MATEIS - INSA Lyon, Civil Engineering and Urban planning, France
2. MATEIS - INSA Lyon, Materials Science, France

The main benefits of using raw earth-based materials in construction is their low environmental footprint. However, durability and mechanical properties of raw earth-based materials are usually too low for them to be commonly used in the construction field. Then, a stabilization with a hydraulic binder, such as cement, is needed to trigger the construction standards. Stabilization of earth by alkali-activated binders could be an alternative, improving mechanical strength, durability and maintaining a low environmental footprint of raw earth concretes. In this study, blast furnace slag and sodium hydroxide are used as raw materials for alkali-activated binder and added to three raw earths for stabilization. The parameters of formulation are: mineralogy of the earth, slag and sodium content. The efficiency of stabilization is evaluated through hardness measurements by instrumented indentation with a sphere. The results showed different mechanical behaviors, highlighting the influence of the clay content and mineralogy on the stabilization process. A characterization of the hydrates formed by the binder and the impact of alkali-activation on the integrity of clay particles is made through microstructural analyses. Calcium silicate hydrates were detectable only with the highest slag content (15%) and sodium concentration (12M). No alteration of the initial clayey structures was detected in these conditions.

S17: Advanced Ceramic Materials and Processing for Photonics and Energy

Advanced and Nanostructured Materials for Photonics, Electronics and Sensing II

Room: Coquina Salon G (North Tower)
Session Chair: Alessandro Martucci, University of Padova

8:30 AM

(ICACC-S17-009-2023) Lanthanide-based nanoparticles with tuneable emission wavelengths from visible to mid-infrared (Invited)

E. Hemmer*[1]

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

Based on their outstanding optical properties, lanthanide(Ln)-based compounds have been suggested for a wide range of applications including the fields of biomedicine, optoelectronics, and solar energy conversion. For instance, Ln-based materials are known to emit visible and near-infrared (NIR) light under NIR excitation, which is sought after when aiming for enhanced penetration depth into media opaque to visible light. Recently, Ln-based compounds were suggested as mid-infrared (MIR) emitters, extending their spectral features into the 2000nm region. Lanthanide fluorides ($MLnF_4$, M = alkali metal) are an excellent choice when seeking bright emitters. In this vein, we developed rapid microwave-assisted strategies towards Ln^{3+}-doped $NaGdF_4$ nanoparticles (NPs). Controlling process parameters enables phase-selective synthesis of NPs with sizes tailored at the sub-20nm regime. The use of various Ln^{3+} dopants allows to tune the NPs' emission profile, including the underexplored MIR region, which potentially offers new opportunities for various applications.

9:00 AM

(ICACC-S17-010-2023) Lanthanide doped ZnO; From micrometer to Ångström scale (Invited)

G. Westin*[1]

1. Uppsala University, Sweden

Herein, a fast salt-based synthesis route to nano-crystalline ZnO:Eu (0-15% $EuO_{1.5}$) sponges built of ~10 nm sized ZnO:Eu crystallites is presented. A detailed description of the phase-development using TG, DSC, XRD, IR spectroscopy, SEM, TEM, XPS, and EXAFS is also presented. The ZnO:Eu sponges obtained on heating for 3 min at 200-600 °C showed peaks due to hex-ZnO with unit cell-dimensions very close to the un-doped ZnO. Upon heating at 700-1000 °C, depending on Eu content, phase separation took place yielding c-Eu_2O_3 nano-crystals on the ZnO:Eu sponge surface. The ZnO:Eu sponges obtained at 600 °C were elementally homogenous while containing high concentrations of the large, alio-valent Eu^{3+}-ions. The nature and even existence of such high doping levels has been debated and was thus of particular interest. The dopant structure was studied in detail using EXAFS and DFT. It was shown that the ZnO structure could be close to unchanged while containing 5% $EuO_{1.5}$ for structures containing Eu-oxide clusters of 4 or 8 Eu^{3+}-ions. These structures showed excellent agreement with all other experimental data. Structures containing smaller Eu-oxide clusters with 2 Eu^{3+}-ions were less stable and deviated from the experimental data, but were more stable than a random 2 Eu^{3+} + 1 Zn^{2+} vacancy based ZnO structure of the same Eu content. These structures were highly unexpected, and may help in the understanding of semiconductor oxides.

9:30 AM

(ICACC-S17-011-2023) Functionalized light emitting silicon nanoparticles (Invited)

N. Daldosso*[1]

1. University of Verona, Italy

Silicon micro and nano particles are an ideal traceable carrier for biomedical purposes thanks to its interesting properties such as the visible photoluminescence, the size-dependent properties, the large surface-to-volume ratio; it is biodegradable, and biocompatible. The material properties can be tailored and controlled via surface and structural modifications. Synthesis and structural, physical and chemical characterization of functionalized Si nanostructures will be presented and the results about the different approaches and strategies to enhance and stabilize the optical properties and to add further functionalities to the system will be discussed. In particular: - results about the overcoming of the problem of optical and structural degradation in aqueous media by developing methods for organic (PEG and chitosan) and inorganic (TiO_2) coatings - the success to add magnetic properties by decorating the internal pore surface by superparamagnetic iron oxides nanoparticles (SPIONs) - the results about loading and release of molecules/drugs and rare-earths (e.g. Eu, Tb, Yb) oxides for optical sensing and bio medical applications. The combination of these results paves the way to the system to become a multimodal platform that can find application in theranostics and optical sensing.

Multi-functional Materials I

Room: Coquina Salon G (North Tower)
Session Chair: Federico Polo, Ca' Foscari University of Venice

10:10 AM

(ICACC-S17-012-2023) Non-hydrolytic sol-gel chemistry to functional hybrid materials (Invited)

N. Pinna*[1]

1. Humboldt-Universität zu Berlin, Department of Chemistry, Germany

The current trend in various energy applications, ranging from lighting to batteries and electrolizers, lays in the control of structural, physicochemical and morphological properties of materials and their interfaces. During this presentation, recent strategies for nanostructured materials synthesis, targeting energy and environmental applications will be discussed. Especially, we will focus on one-pot strategies for the fabrication of hybrid and complex nanomaterials focusing on the importance of the organic-inorganic and inorganic-inorganic interfaces. Among the examples presented, we will discuss the synthesis of complex nanostructures and the stabilization of metastable phases for applications in energy storage and conversion. We will see that nowadays the available strategies allow a control in terms of composition, crystalline structure, morphology and nanostructuration that would have been unimaginable just few years ago. Finally, the open challenges the field is currently facing and possible further developments which are needed to meet the always growing demand for high performing materials will be also discussed.

10:40 AM

(ICACC-S17-013-2023) Optical Emitters for Thermophotovoltaics: from Photonics to Thermochemical Stability (Invited)

M. Leite*[1]

1. UC Davis, Materials Science and Engineering, USA

Thermophotovoltaics (TPV) represent a promising route for converting heat generated as a byproduct into usable electricity through a clean energy paradigm. However, the material options used to date for the optical emitters substantially constrain the power conversion efficiency of this process. Specifically, we focused our search to dual-layer, thin film emitters, which are scalable. Thus, we screened the optical response (i.e. permittivity) of >2,800 material combinations with melting point >2,000 oC, comprising refractory silicides, borides, carbides, nitrides, oxides, and metals. Overall, the mismatch in permittivity allowed for emission control, key for the development of high performing TPV. We found a handful of emitters for TPV with theoretical efficiency >60% at 1800 °C, for GaSb solar cells. To complement our analysis, we also identified optimal material combinations for InGaAsSb, InGaAs, Ge, GaSb, and Si solar cells. The final down selection of materials is also based on their thermal expansion and thermochemical stability, which are frequently overlooked. The discussion of the best material options will be accompanied by the optical characterization of selected materials up to 1500C, using in situ reflection and transmission measurements.

11:10 AM

(ICACC-S17-014-2023) Designing metal oxides and sulphides incorporated carbon nanocomposites for clean energy applications (Invited)

D. Chua*[1]

1. National University of Singapore, Materials Science & Engineering, Singapore

Electrocatalyst and its supports are critical in having good reaction rates and controlled selectivity in clean energy generation applications. Carbon materials has always been at the fore-front due to their unique properties, ranging from low dimensional effects, good structural integrity, high electrical and thermal conductivity, and chemical stability, making it suitable as supports. In this aspect, one area of interest lies in how well metal-oxides and sulfides can integrate with these carbon as a fully functioning electrocatalyst. In this talk, we will show how metal-oxide/sulfides can easily integrate with carbon supports increasing it performance. For example, MoO integrated wtih carbon-support exhibits good water-splitting electrocatalytical effects. We will further show how various ternary noble metal on metal oxides on carbon support also exhibit extremely good electrocatalytical performance in hydrogen fuel cells. A series of in-situ tests are also performed which includes accelerated degradation test and electrochemical impedance spectroscopy to validate the effectiveness and robustness of these materials. We will briefly mention about some other materials that we have tested.

S18: Ultra-High Temperature Ceramics

Compositionally Complex UHTCs II

Room: Coquina Salon A (North Tower)
Session Chair: Stefano Curtarolo, Duke University

8:30 AM

(ICACC-S18-009-2023) Plasmonic high-entropy carbides (Invited)

S. Curtarolo*[1]; A. Calzolari[3]; C. Oses[1]; C. Toher[1]; M. Esters[1]; X. Campilongo[1]; S. Stepanoff[2]; D. E. Wolfe[2]

1. Duke University, Materials Science, Electrical Engineering and Physics, USA
2. Pennsylvania State University, USA
3. Istituto Nanoscienze CNR-NANO-S3, Italy

Discovering multifunctional materials with tunable plasmonic properties, capable of surviving harsh environments is critical for advanced optical and telecommunication applications. We chose high-entropy transition-metal carbides because of their exceptional thermal, chemical stability, and mechanical properties. By integrating computational thermodynamic disorder modeling and time-dependent density functional theory characterization, we discovered a crossover energy in the infrared and visible range, corresponding to a metal-to-dielectric transition, exploitable for plasmonics. It was also found that the optical response of high-entropy carbides can be largely tuned from the near-IR to visible when changing the transition metal components and their concentration. By monitoring the electronic structures, we suggest rules for optimizing optical properties and designing tailored high-entropy ceramics. Experiments performed on the archetype carbide $HfTa_4C_5$ yielded plasmonic properties from room temperature to 1500K. Here we propose plasmonic transition-metal high-entropy carbides as a class of multifunctional materials. Their combination of plasmonic activity, high-hardness, and extraordinary thermal stability will result in yet unexplored applications. Research sponsored by DOD-ONR. Reference: Calzolari et al, Nat. Commun. (2022) 10.1038/s41467-022-33497-1

9:00 AM

(ICACC-S18-010-2023) The Role of Entropy and Enthalpy in Mixed Transition Metal Carbides

C. R. Weinberger*[1]; G. Thompson[2]; X. Tang[1]; K. Ma[1]

1. Colorado State University, Department of Mechanical Engineering, USA
2. Thompson, Metallurgical & Materials Engineering, USA

One avenue of developing new ultrahigh temperature ceramics is through the use of multiple principal component ceramics. These so-called high entropy ceramics are similar to high entropy alloys and have the potential to exhibit superior properties as compared materials with fewer elements, e.g. $TaHfZrTiC_4$ compared to TiC. However, the cause of mixing, be it enthalpy or entropy, and the thermodynamic stability of these compounds is not well known. Here, we investigate the thermodynamics of mixing multiple principal elements in the transition metal carbides from binary solutions to 7 element transition metal solutions using a combination of density functional theory and computational thermodynamics. We find that while increasing the number of elements increases solubility of the solution in a general sense, this is not a universal trend and depends strongly on the types of transition metals in the solution. Furthermore, we demonstrate that many multiple principal element transition metal carbides are not stable below 1000°C. These results provide direct insight into the stability of thye solutions, and which transiton elements will increase solubility and therefore stability.

9:20 AM

(ICACC-S18-011-2023) Processing and properties of high entropy carbide ceramics synthesized by carbothermal reduction

P. Brune*[1]; L. Feng[3]; G. Hilmas[2]; W. Fahrenholtz[3]; J. Watts[3]

1. Missouri University of Science & Technology, Ceramic Engineering, USA
2. Missouri University of Science & Technology, USA
3. Missouri University of Science & Technology, Dept. of Materials Science and Engineering, USA

High entropy carbide (HEC) research is expanding rapidly due to their exceptionally high hardness over the baseline of their constituents. While most of the current body of work covers the production of these ceramics using commercial carbide powders, the present research focuses on the use of reaction synthesis to produce pure and dense HEC ceramics. In this work five single phase HECs $(Hf_{0.2},Nb_{0.2},Ta_{0.2},Ti_{0.2},Zr_{0.2})$ C, $(Hf_{0.2},Cr_{0.2},Ta_{0.2},Ti_{0.2},Zr_{0.2})$ C, $(Hf_{0.2},Mo_{0.2},Ta_{0.2},Ti_{0.2},Zr_{0.2})$ C, $(Hf_{0.2},W_{0.2},Ta_{0.2},Ti_{0.2},Zr_{0.2})$ C, and $(Hf_{0.2},Mo_{0.2},W_{0.2},Ti_{0.2},Zr_{0.2})$ C were synthesized by carbothermal reduction and densified by direct current sintering. The single phase HECs were examined with scanning electron microscopy and X-ray spectroscopy to ensure that a fully solutionized single phase had formed. The Vickers hardness of each HEC was measured over a range of indentation loads with a maximum measured hardness of 43.5 ± 2.9 GPa at an indentation load of 0.05 kgf for $(Hf_{0.2},Mo_{0.2},W_{0.2},Ti_{0.2},Zr_{0.2})$ C. The thermal diffusivity, heat capacity, and thermal conductivity of each HEC were also measured using the laser flash method and compared to the constituent monocarbide phases.

9:40 AM

(ICACC-S18-012-2023) Direct selective laser sintering of high-entropy carbide ceramics

X. Zhang[1]; N. Li[1]; X. Chen[1]; M. Stroup[1]; Y. Lu[1]; B. Cui*[1]

1. University of Nebraska–Lincoln, USA

The direct selective laser sintering (SLS) process was successfully demonstrated for additive manufacturing of high-entropy carbide ceramics (HECC) for the first time, in which a Yb fiber laser was employed for ultrafast (in seconds) reactive sintering of HECC specimens from a powder mixture of constitute monocarbides. A single-phase non-equiatomic HECC was successfully formed in the 4-HECC specimen with a uniform distribution of Zr, Nb, Hf, Ta, and C. In contrast, a three-layer microstructure was formed in the 5-HECC specimen with five metal elements (Zr, Nb, Hf, Ta and Ti), consisting of a TiC-rich top layer, a Zr-Hf-C enriched intermediate layer, and a non-equiatomic Zr-Ta-Nb-Hf-C HECC layer. Vickers hardness of 4- and 5-HECC specimens were 22.2and 21.8 GPa, respectively, on the surface. These findings have important implications on the fundamental mechanisms governing interactions between lasers and monocarbide powders to form a solid solution of HECCs during SLS.

Novel Processing Methods II

Room: Coquina Salon A (North Tower)
Session Chairs: Tamás Csanádi, Institute of Materials Research, Slovak Academy of Sciences; Sea-Hoon Lee, Korea Institute of Materials Science

10:20 AM

(ICACC-S18-013-2023) Recent research activity about UHTC and brief introduction of a Extreme Materials Demonstration Research Center project in KIMS (Invited)

S. Lee*[1]; M. Park[1]; Y. Zou[1]

1. Korea Institute of Materials Science, Republic of Korea

Non-oxide anti-ablative ceramics material and tiles were fabricated for high temperature application above 2,000°C. For this purpose, ultra-fine UHTC (ultra-high temperature ceramics) powders were synthesized and ultra-high concentration slurries were prepared using Zr- and Hf-based UHTC powders. Ablation resistant materials were fabricated by the densification of the UHTC materials by spark plasma sintering (SPS) or precursor impregnation & pyrolysis (PIP) technique and the ablation behavior was evaluated using an oxy-acetylene torch, arc-jet plasma wind tunnel and HVOF (High velocity oxygen fuel) system. Subsequently, ceramic tiles with 100×100×25mm in size were fabricated by forming protective layers on carbon/carbon composites. The fabrication process and ablation property analysis will be discussed. Recently, Korea Institute of Materials Science (KIMS) developed a big project tentatively entitled "Extreme Materials Demonstration Research Center". The project will start from 2023 for 6 years and the total budget is $ 0.23 billion. The target and plan of the project will be briefly introduce.

10:50 AM

(ICACC-S18-014-2023) Reaction Flash Sintering of Ternary Metal Nitrides

S. Das*[1]; A. Durygin[1]; V. Drozd[1]; A. Eskandariyun[1]; J. S. Smith[2]; Z. Cheng[1]

1. Florida International University, Mechanical and Materials Engineering, USA
2. Argonne National Laboratory, Advanced Photon Source, USA

This study aims to demonstrate reaction flash sintering (RFS) for two systems: TiN-ZrN (both conducting) and AlN-TiN (TiN conducting but AlN insulating). Commercial nitride powders (1:1 molar ratio) were mixed by Spex milling and consolidated into dense bulk ceramic/composite with a homemade flash sintering setup. A constant DC electrical field of ~50 V/cm and pressure of

~15 MPa at room temperature triggers RFS without pre-heating, and the entire process finishes in ~350 s. In situ synchrotron XRD is also performed at the Advanced Photon Source to track the phase evolution in both systems during RFS and subsequent cooling. It shows although both systems have a wide miscibility gap extending to high temperature, uniform (Ti, Zr)N solution formed in RFS persists upon cooling, while (Ti, Al)N solid solution formed in RFS at high temperature goes through a quick phase separation in the cooling process. The final products' microstructure, as well as mechanical and oxidation resistance properties, are characterized by SEM/EDS, Vickers indentation, and TGA, respectively.

11:10 AM

(ICACC-S18-015-2023) Effects of Ball-Milled Ti on Densification and Strength of Titanium Diboride

Y. Jimba*[1]; S. Kondo[2]; H. Yu[2]; Y. Okuno[3]; S. Nogami[1]; R. Kasada[2]

1. Tohoku University, Department of Quantum Science and Energy Engineering, Graduate School of Engineering, Japan
2. IMR Tohoku University, Japan
3. Institute for Integrated Radiation and Nuclear Science, Kyoto University, Japan

Group IV transition metal borides are potential candidates for various applications, including hypersonic vehicles and nuclear systems, due to their unique properties such as high melting point (T_m), hardness, and thermal conductivity. However, the sintering requires a high temperature of ≥1800 °C and pressure of ≥30 MPa. Therefore, lowering the process temperature is essential for further applications, especially for complex shapes. Although little addition of metallic aid, which constructs boride itself, is known to provide high density without strength degradation, the temperature is limited by the T_m of the aid element. This work aims to lower the temperature with ball-milled (BM) metallic aid, using titanium and its diboride as a case study, by evaluating the effects on density and strength in spark plasma sintering at 1300 °C. The BM aid densified 10% more than the case with as-received (AR) Ti owing to its decreased particle size and possibly T_m by ball-milling. The highest density was ~99% theoretical density of TiB_2 for the sample with 6 wt% BM, and the flexural strengths at RT were ~2.5 times higher than the one with AR. Fracture surface analyses implied the bond strengthening of matrix particles by reactively producing titanium monoboride. The promising results open a new perspective of ball-milling, which may lower the sintering temperatures of the ceramics with metallic aids.

11:30 AM

(ICACC-S18-016-2023) Field Assisted Sintering of Ternary Hafnium-Tantalum Carbides for Reusable Hypersonic Thermal Protection Systems

D. E. Wolfe*[1]; C. DeSalle[1]; P. Albert[1]; J. Reiss[1]; C. Ryan[1]; S. Stepanoff[1]; Z. Boring[1]; P. Kolonin[1]

1. Pennsylvania State University, USA

Refractory carbides are promising candidates for hypersonic applications due to their superior thermomechanical and thermochemical properties at ultra-high temperature (UHT). However, densification often requires sintering aids, which may compromise performance by forming liquid/glassy secondary phases at grain boundaries. Field Assisted Sintering Technology (FAST) achieves near theoretical density without sintering aids by applied pressure with pulsed DC resistance heating of powders and surrounding graphite dies. Optimized processing parameters for a range of HfC-TaC compositions produce high relative density compacts (up to 99.5%) with ultra-fine microstructures. Rapid processing limits grain growth, resulting in strengthening via the Hall-Petch effect. This work demonstrated record-breaking nanohardness (41.45 ± 1.37 GPa), Vickers microhardness (30.2 ± 3.1 GPa), and elastic modulus (590.12 ± 10.64 GPa) values. Flexural strength, Weibull analysis, and Young's modulus tests show higher strength and toughness values to minimize thermal shock-induced failure. Oxidation kinetics testing allowed for investigating surface oxide growth/microstructure, while mechanical property testing provided confirmation of retained strength. The UHT-stable $Hf_6Ta_2O_{17}$ layer is expected to provide ablation resistance to create a superior thermal protection system (TPS).

S19: Molecular-level Processing and Chemical Engineering of Functional Materials

Polymer-Derived Ceramics I

Room: Ballroom 3 (South Tower)
Session Chair: Peter Kroll, University of Texas, Arlington

8:30 AM

(ICACC-S19-009-2023) Solution chemistry engineering of catalyst interface for solar water splitting (Invited)

F. L. de Souza*[1]

1. Federal University of ABC, Center of Natural Science and Humanity, Brazil

Nanostructure design faster becomes a key factor in the race to enhance the photoelectrochemical device performance, because it may lead to a reduction of the charge recombination rate. This talk will describe a simple chemistry manipulation of the polymeric precursor solution preparation that succeeded in creating a back-contact rich interface between the commercial fluorine-doped tin oxide coated glass and the nanoceramic photocatalyst layer. Nanoscale analysis revealed that the stress between FTO-nanoceramic photocatalyst at the back contact interface during the fabrication process can be alleviated by combining Zr^{4+} addition and manipulating the polymeric precursor solution. This result will have an important impact on the effective charge injection through the back-contact interface by partially mitigating the shunting recombination regularly observed in mesoporous materials. Chemical mapping analysis was conducted during the nanoscale investigation by scanning transmission electron microscopy showing that the Zr^{4+} are distributed through the samples and preferentially segregating at hematite grains. Photoelectrochemical performance recorded at front-side illumination increases with Zr^{4+} concentration, which can be associated with a reduction in the energy barrier between the grains provoked by Zr^{4+} presence and owned by a change in the surface chemistry.

9:00 AM

(ICACC-S19-010-2023) Polymer Derived Ceramic particles capped with hyaluronic acid nanogates for the smart release of antiviral drugs (Invited)

M. Garces[2]; A. Martin-Illana[2]; R. Cazorla-Luna[2]; F. Notario[2]; A. Tamayo*[1]

1. Institute of Ceramics and Glass, CSIC, Spain
2. Complutense University of Madrid, Spain

Calcium-containing polymer derived ceramic particles were prepared and subsequently loaded with the antiviral drug tenofovir. The loaded particles were then covered with strands of hyaluronic acid to prevent the drug to be released. The drug releasing kinetics was studied in three different simulant media: vaginal, seminal and a mixture of both in a 1/10 ratio to simulate ejaculation. The different solubility of the drug at the pH of the releasing media leads to an increase in the drug release in the presence of the seminal fluid whereas if a mixture of the two simulant fluids is used, the formation of several complexes difficult the diffusion of the drug out of the particle environment. The efficacy of the hyaluronic acid nanogates has been tested by incorporating catalytic amounts of the hyaluronidase enzyme to the seminal fluid. The enzyme degrades the hyaluronic acid strand thus provoking the release of the enzyme. Due to the increase in the viscosity of the seminal fluid, the parameter n of the kinetic constant (fitted to the Korsmeyer-Peppas model) increases in the presence of the enzyme thus leading to a fast release of the entrapped drug. This local viscosity increase has been confirmed in vaginal films prepared with the polymer derived ceramic particles by means of electrochemical impedance measurements.

9:30 AM

(ICACC-S19-011-2023) Strategies for macro- and microporosification of polymer-derived ceramics (Invited)

T. Konegger*[1]; C. Drechsel[1]; H. Peterlik[2]; K. Rauchenwald[1]

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

The polymer precursor technique is an innovative processing approach for the fabrication of porous ceramic materials using preceramic organosilicon polymers as starting materials. After adequate structuring and shaping, the precursors can be converted to polymer-derived ceramic (PDC) materials by a controlled pyrolytic decomposition treatment. This talk gives an overview of a variety of strategies developed over the past years which have been successfully implemented for the fabrication, modification, and characterization of novel porous PDC materials. The main focus is set on the generation of macroporosity through distinct porosification strategies, including templating of PDCs using sacrificial porogens as well as photopolymerization-assisted solidification templating of PDCs. Furthermore, concepts for the generation of microporous PDC structures are shown, including a special focus on elucidating and controlling the micropore evolution, supported by the presentation of a characterization routine to monitor the state of microporosity in-situ during pyrolytic conversion. By identifying the role of relevant parameters specific to each processing strategy, the overall aim of tailoring pore morphologies to obtain material structures ideally suited for a range of prospective applications can be achieved, examples including membrane-based gas separation or catalytic conversion processes.

Polymer-Derived Ceramics II

Room: Ballroom 3 (South Tower)
Session Chair: Yoshiyuki Sugahara, Waseda Universitry

10:20 AM

(ICACC-S19-012-2023) Ceramic Nonwovens with Exceptional Properties from Silazane/Polyacrylonitrile Hybrid Polymers (Invited)

G. Motz*[1]; J. Denk[1]; S. Schafföner[1]; X. Liao[2]; S. Agarwal[2]

1. University of Bayreuth, Ceramic Materials Engineering, Germany
2. University of Bayreuth, Macromolecular Chemistry 2, Germany

The specific synthesis of materials that combine very contradictory properties is very complicated. However, the combination of silazanes with polyacrylonitrile leads to a hybrid polymer suitable for electrospinning. Curing of the resulting nonwovens in air and subsequent pyrolysis in nitrogen atmosphere yielded ceramic nonwovens with more than 50 wt.% of free carbon. Despite this, the material has significantly improved oxidation and fire resistance compared to carbon, which results from the very homogeneously distributed ceramic nano-phase within the carbon matrix. In addition, the material is characterized by very high electrical conductivity because of the carbon phase but very low thermal conductivity due to excessive phonon scattering at the boundaries of carbon to the ceramic nano-particles. The developed ceramic nonwovens are also stand out by their excellent flexibility and mechanical durability. Such an easy-to-manufacture material is very interesting for many functional applications.

10:50 AM

(ICACC-S19-013-2023) Preceramic Polymer Pre-processing of Polycarbosilanes (Invited)

S. Bullock*[1]; C. L. Cramer[2]; T. Aguirre[2]; D. Mitchell[1]

1. Oak Ridge National Lab, MSTD, USA
2. Oak Ridge National Lab, Manufacturing Science Division, USA

Preceramic polymer for additive manufacturing has unique rheology requirements for deposition of higher aspect ratio extruded preforms. Increasing the yield stress of the preceramic polymer provides greater dimensional stability during printing. Incorporating fillers can provide rheological properties to reduce slumping of the printed parts. Also, slumping can occur during the cure cycle for polycarbosilanes, higher yield stress is needed to reduce slumping. This work focuses on methods to increase yield stress of preceramic polymers to provide stable printed structures for larger prints (> 1″ x1″). The use of crosslinking agents with Starfire Systems SMP-10 resin and catalyst, allows room temperature B-staging of these preforms. Shore A of 70 indicates these are relative flexible B-staged polymers with increased yield stress. TGA, DSC, FTIR and NMR of these crosslinked system show network formation at temperatures <50 C and allow printing of flexible AM preceramic polymers.

11:20 AM

(ICACC-S19-014-2023) Hydrocarbon Precursor Effects on the Deposition Structure of Carbon Fibers

C. A. Cook*[1]; G. Thompson[2]

1. The University of Alabama, Metallurgical and Materials Engineering, USA
2. Thompson, Metallurgical & Materials Engineering, USA

Laser Chemical Vapor Deposition (LCVD) is an additive processing technique in which freestanding fibers are deposited by the dissociation of a precursor gas under a translating laser focal point. For carbon fiber deposition, this precursor is a hydrocarbon gas. In this study the relationship between the precursor chemistry of different hydrocarbon gases and the deposited carbon fiber structure and mechanical properties are compared. Here, different precursors were selected based on hydrocarbon concentrations and bond types (single or double). Depending on processing conditions (pressure, growth rate and temperature), the fibers exhibited drastic changes in morphology and strength. For example, Methane (CH_4) resulted in substantially lower growth speeds and had half the fracture strength when compared to Ethylene (C_2H_4) formed fibers. In addition, the activation energy of CH_4 was found to be significantly higher than that of C_2H_4. These differences enabled C_2H_4 to have fewer or more homogeneously distributed flaws in the grown fiber, which was confirmed through a Weibull statical analysis as well as Raman spectroscopy.

11:40 AM

(ICACC-S19-015-2023) Preceramic Polymer / Nanoparticle Hybrids Assembled via Ionic Grafting

N. D. Posey*[1]; M. B. Dickerson[2]

1. Air Force Research Lab/UES Inc., Materials and Manufacturing Directorate, USA
2. Air Force Research Laboratory, Materials and Manufacturing Directorate, USA

Ceramics are leading candidates for a variety of emerging defense needs, including high-temperature structural applications. Ceramic matrix composites (CMCs) are attractive for such applications due to their robust thermal and mechanical properties. CMC constituent production may involve the use of preceramic polymers (e.g. fiber spinning and matrix generation). Though preceramic polymers (PCPs) offer manufacturing advantages, current commercial PCPs lack chemical diversity and often shrink during pyrolysis. To mitigate these issues, our team has recently developed PCP-grafted nanoparticles, where PCPs are covalently grafted from solid

nanoparticle cores. Here, we report an alternative to covalently grafted nanoparticles, whereby PCPs were grafted to ceramic nanocores via non-covalent interactions. Polycarbosilanes with cationic amine end groups were synthesized and complexed with sulfonated nanoparticles resulting in preceramic nanoscale ionic materials. The rheological and thermal properties of these new materials along with chemistry/processing/structure/property relationships of the ionically grafted preceramic nanoparticle hybrids and their resulting ceramics will be discussed. Our alternative grafting-to approach creates the potential to design a variety of ceramic materials through the mixing-and-matching of ceramic nanoparticles with oppositely charged PCPs.

Emerging Materials and Sustainable Manufacturing Technologies in a Global Landscape: Symposium in Honor of Dr. Tatsuki Ohji

Tatsuki Ohji Honorary Symposium 2

Room: Coquina Salon D (North Tower)
Session Chairs: Michael Halbig, NASA Glenn Research Center; Csaba Balazsi, ELKH Centre for Energy Research

1:30 PM

(ICACC-HS-008-2023) Opening new perspectives for Ceramic-Matrix Composites in seldom explored application sectors (Invited)

G. L. Vignoles*[1]

1. University Bordeaux, LCTS - Lab for ThermStructural Composites, France

Long fiber-reinforced Ceramic-Matrix Composites have been historically developed for space and military applications because of their outstanding high-temperature thermal and structural performances; their application range has broadened to friction components (brakes, clutches, etc.) and to industry (casings, heat exchangers, etc). They also are developed in the frame of nuclear fission and fusion technologies. Nonetheless, they are still often expensive materials because their production volume is not large. Finding new markets for CMCs is therefore an important objective for their development. This presentation will discuss three cases of potential uses of CMC in economic areas in which they were scarcely present. These incoming opportunities for CMCs are fed by evolutions in the energy market and by its shift towards the use of more hydrogen and "green fuels". Examples of ongoing prospective works in steelmaking and in nuclear waste storage engineering will be discussed in this talk. The author thanks the European Commission for grants to the projects « CEM-WAVE » n° 958170 and « RecHycle » n° 101058692 and the French agency ANDRA for grant n° 20081163 to the project "C3N".

2:00 PM

(ICACC-HS-009-2023) Short Fiber Reinforced Ceramic Matrix Composites (Invited)

W. Krenkel*[1]; G. Puchas[2]; J. Winkelbauer[1]; S. Flauder[1]

1. University of Bayreuth, Germany
2. University of Bayreuth, Ceramic Materials Engineering, Germany

Short fibers or chopped fiber bundles are typically produced by cutting long fibers or rovings to chopped fibers with lengths of several millimeters to few centimeters. In contrast to polymeric or metallic matrices, the minimum fiber length for ceramics is typically one order of magnitude higher and mostly defined rather by technological than by theoretical reasons. Short fiber reinforced ceramic matrix composites (SF-CMCs) have been developed both as non-oxide composites with ceramic or carbon fiber reinforcements and as oxide-based composites in which both, fibers and matrix, are comprised of oxide materials. They represent an attractive alternative to reinforcements with continuous fibers although their mechanical properties are somewhat lower. Lightweight components in the combustion environment (e.g. nozzles, burners), in aerospace (e.g. telescopes), heat treatment (e.g. charging racks) or in frictional applications (e.g. brake disks, pads, clutch linings) represent applications of this type of ceramic matrix composites.

2:30 PM

(ICACC-HS-010-2023) Joining and Integration of Silicon Carbide-Based Ceramic Materials for Aerospace Applications: An Overview (Invited)

M. C. Halbig*[1]; M. Singh[2]

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

In order to fully utilize silicon carbide-based ceramics and ceramic matrix composites (CMCs) for aerospace and ground based applications, advanced joining and integration technologies are critically needed for achieving large, complex shaped, and integrated components. Careful material selection of joining inter-layers needs to consider chemical and thermal compatibilities with the parent materials that are being joined. Also, optimization needs to be conducted to select the proper processing conditions for achieving robust joints that are well adhered, strong, uniform, crack free, and have minimal residual stresses. NASA GRC developed pressureless and pressure aided approaches for joining ceramics and CMCs include brazing, diffusion bonding, refractory eutectic phase joining, and Single-step Elevated Temperature (SET) Joining. For joining ceramics and CMCs to metals, several brazes have been utilized. Brazing approaches for joining ceramics and metals have also included tailored brazes with multiple interlayers and particle additions. An overview of the joining approaches will be provided. In addition, processing-microstructure-property relationships will be discussed.

3:20 PM

(ICACC-HS-011-2023) Bandgap engineering of epitaxial β-$(Al_xGa_{1-x})_2O_3$ films and its optical properties (Invited)

I. Milisavljevic[1]; Y. Wu*[1]

1. Alfred University, Kazuo Inamori School of Engineering, New York State College of Ceramics, USA

Wide bandgap materials such as β-Ga_2O_3 have been receiving significant research attention in the recent period due to their high number of potential applications in electronic and optoelectronic devices. By alloying β-Ga_2O_3 with elements such as Al_2O_3, the bandgap can be further increased, enabling new applications of β-$(Al_xGa_{1-x})_2O_3$ compounds in ultra-high-power electronics, ultra-short wavelength optoelectronics, and other. In addition, doping with rare-earth ions coupled with existing perks of the engineered bandgap of the host allows the materials with luminescence emission, which add new functionalities to the materials. This work provides an overview of the findings on undoped and Tb-doped β-$(Al_xGa_{1-x})_2O_3$ epitaxial films with different Al concentrations on sapphire substrates prepared using the metal-alkoxide sol-gel spin-coating approach. Through the comprehensive analysis of the structural, chemical, and optical properties, the effect of Al concentration, rare-earth doping, and substrate orientation (including c-, a-, and r-sapphire substrates) on the bandgap engineering of the films was discussed. The obtained results demonstrate the great potential of the sol-gel spin-coating technique for fabricating good-quality epitaxial β-$(Al_xGa_{1-x})_2O_3$ film with engineered bandgap and luminescence characteristics.

3:50 PM

(ICACC-HS-012-2023) Dielectric Properties of Lanthanum Doped $BaTiO_3$ Ceramics Fabricated with Defect-controlled Nanoparticles (Invited)

S. Lee*[2]; J. Song[2]; J. Oh[1]; D. Kim[2]

1. Samsung Electro-Mechanics, Republic of Korea
2. Korea Advanced Institute of Science and Engineering (KAIST), Dept. of Mater Sci & Eng, Republic of Korea

Colossal permittivity (CP) materials are promising candidates for the next generation ceramic capacitors. Among them, highly reduced $BaTiO_3$ is widely known candidate which presents giant permittivity ($\varepsilon_r \sim 10^6$). However, dielectric loss ($0.1<\tan\delta$) remains a major problem for electrical applications. Although several studies proposed the origin of CP characteristic, few reported on the relations between CP behavior of ceramics and the defects of raw powder. Here, we synthesized 100nm of Lanthanum doped $BaTiO_3$ nanoparticles (NPs) by conventional hydrothermal method. Highly defective cation vacancies (i.e. V_{Ba} and V_{Ti}) are introduced by controlling the synthesis parameter, which are confirmed by Raman spectroscopy. Moreover, ceramics fabricated with defective NPs clearly showed different dielectric characteristics ($\varepsilon_r \sim 10^3$) compared to the ones with high crystalline. Thus, high concentrated defects distributed in synthesized particles hindered the formation of oxygen vacancies during sintering and exhibited low capacitance. Further increase in $[La^{3+}]$ reduced the concentration within the particles and showed CP behavior. This clearly proves the importance of the controlling defects of raw NPs for fabricating CP dielectric ceramics.

4:20 PM

(ICACC-HS-014-2023) Photopolymerization-Based 3D Printing of Inorganic-Organic Hybrid Materials (Invited)

R. Narayan*[1]

1. North Carolina State University, USA

Two-photon polymerization is a 3d printing technology that uses pulses from a titanium:sapphire laser to excite photoinitiator molecules within a photosensitive material for selective polymerization. Polymerization takes place at positions in the photosensitive material when the excitation threshold of the photoinitiator is exceeded. Polymerization of biomedical devices with sub-microscale features is possible since the two-photon absorption process exhibits a nonlinear relationship with the incident light intensity. Two-photon polymerization has been used to prepare biomedical devices with sub-microscale attributes from photosensitive organically-modified ceramic materials (e.g., zirconium oxide hybrid materials). Several kinds of medical devices (e.g., drug delivery devices, tissue engineering scaffolds, and sensors) have been manufactured using two-photon polymerization. Methods to optimize the materials, processing parameters, and postprocessing steps for medical device applications will be described. Materials characterization and biological evaluation of two-photon polymerization-created medical devices will be considered. Next steps in the development of photopolymerization-based 3d printing technologies for commercially scalable manufacturing of medical devices will be described.

5th Pacific Rim Engineered Ceramics Summit

5th Pacific Rim Engineered Ceramics Summit III

Room: Coquina Salon B (North Tower)
Session Chairs: Mati Horprathum, National Electronics and Computer Technology Center (NECTEC); Jae Chul Kim, Stevens Institute of Technology

1:30 PM

(ICACC-PACRIM-006-2023) Glass Scintillator: Review and Their Recent Development (Invited)

J. Kaewkhao*[1]

1. Nakhon Pathom Rajabhat University, Center of Excellence in Glass Technology and Materials Science, Thailand

Glasses doped with Lanthanide ions (Ln^{3+}) can be developed as scintillation material because of high emission efficiencies, corresponding to 4f–4f and 4f–5d electronic transitions in the Ln^{3+}. In case of 4f–4f transition, sharp emission spectra from the ultraviolet to the infrared region are obtained, because of their shielding effects of the outer 5s and 5p orbitals on the 4f electrons, while 4f–5d transition of Ce^{3+} shows very fast timing respond behavior. Ce^{3+}/Ce^{4+} concentration and ratio are also key for enhancement of scintillation intensity. In this presentation, Lanthanide ions doped scintillating glasses have been reviewed. Scintillation and luminescence and properties of the glasses doped with several lanthanide ions have been explained. Moreover, the current status of their potential applications for scintillation material are also given.

2:00 PM

(ICACC-PACRIM-007-2023) Transparent and fluorescent rare-earth doped α-SiAlON ceramics (Invited)

J. Tatami*[1]; K. Aminaka[1]; M. Iijima[1]; T. Takahashi[2]; T. Yahagi[2]; M. Yokouchi[2]

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

Recently, novel transparent and fluorescent materials are in demand for various optical applications such as lasers, scintillators, and solid-state lighting. α-SiAlON, which has excellent thermal and mechanical properties, also exhibits photoluminescence depending on the stabilized doped rare-earth ions. In this study, we fabricated α-SiAlON ceramics by adding various rare-earth oxides to elucidate their effects on the transparency and fluorescence of these ceramics. High-transparency α-SiAlON ceramics were fabricated by adding rare-earth oxides whose rare-earth ions have small ionic radii: Y_2O_3, Ho_2O_3, Er_2O_3, Tm_2O_3, Yb_2O_3, and Lu_2O_3. Because the fraction of α-SiAlON was high, the relative density was high, and the microstructure was composed of fine grains. In particular, α-SiAlON ceramics prepared by adding Ho_2O_3 showed lower light scattering than the other fabricated α-SiAlON ceramics because of the smaller α-SiAlON grains, resulting in higher in-line transmittance (48% at 600 nm). Furthermore, these transparent α-SiAlON ceramics exhibited fluorescence corresponding to the activated rare-earth ions: Ho^{3+}, Er^{3+}, Tm^{3+}, and Yb^{3+} or Yb^{2+}. In addition, the effect of rare earth co-doping on the transparency and fluorescence of α-SiAlON ceramics is also reported.

2:20 PM

(ICACC-PACRIM-008-2023) Utilization of nanosecond pulsed electric field in water purification process by electrocoagulation method (Invited)

T. Nakayama*[1]; N. Que[2]; A. Munkhbadrakh[1]; H. Suematsu[1]; T. Goto[1]; K. Niihara[1]

1. Nagaoka University of Technology, Japan
2. Kyungpook National University, Republic of Korea

This paper describes a series of research results that enabled improvement of COD (Chemical Oxygen Demand) removal efficiency and energy saving by a new electrocoagulation method using a nanosecond pulsed electric field. The electric flocculation sewage treatment system has the advantages of small size and high efficiency, and is a promising wastewater treatment method. This method can also treat relatively high-concentration wastewater, and has the advantage of not requiring pretreatment, and has been put to practical use in paper mills and the like. However, it has been pointed out that this technology consumes more energy than other wastewater treatment methods. In order to overcome these problems, this paper proposes and demonstrates an improved electrocoagulation method using a nanosecond pulsed electric field.

2:40 PM

(ICACC-PACRIM-009-2023) Catalytic Liquid-phase Oxidation of Phenol Utilizing Novel Catalysts Based on Lanthanum Oxyfluoride (Invited)

N. Imanaka*[1]

1. Osaka University, Applied Chesistry, Japan

For the purpose of developing novel catalysts for the effective phenol oxidation under the moderate conditions (below 100°C with the atmospheric pressure), we focused on the compounds, oxyfluorides. Here, we selected lanthanum oxyfluoride (LaOF), which is the most chemically stable among the oxyfluoride series. In addition, the reducible Bi^{3+} ion was introduced into LaOF to provide redox properties. Here, we synthesized novel Pt/$La_{1-x}Bi_xOF$/SBA-16 catalysts, composed of platinum having high phenol oxidation ability, the $La_{1-x}Bi_xOF$ promoters, and mesoporous silica SBA-16 (Santa Barbara Amorphous No.16), and the catalytic activities for the phenol decomposition were studied. By introducing Bi^{3+} into the LaOF lattice, the removal percentage was greatly enhanced, and the highest activity was obtained for Pt/$La_{0.99}Bi_{0.01}OF$/SBA (97%). This high activity might be caused by the facilitation of the phenol adsorption and the redox properties of the Bi^{3+} ion. In addition, the phenol removal percentage of Pt/$La_{0.99}Bi_{0.01}OF$/SBA (97%) was higher than previously reported Pt/CeO_2-ZrO_2 (92%) catalyst, conducted at more severe condition of 160°C and 2 MPa.

5th Pacific Rim Engineered Ceramics Summit IV

Room: Coquina Salon B (North Tower)
Session Chairs: Tadachika Nakayama, Nagaoka University of Technology; Jakrapong Kaewkhao, Nakhon Pathom Rajabhat University

3:20 PM

(ICACC-PACRIM-010-2023) Design and engineering metal oxide ceramic nanostructures based on optical and electrical sensors for identification of VOCs (Invited)

M. Horprathum*[1]

1. National Electronics and Computer Technology Center (NECTEC), Thailand

Developing high-performance volatile organic compounds (VOCs) sensors was important in environmental monitoring. The metal oxide based advanced ceramic nanostructure has attracted attention for VOCs sensors due to its large surface area and high chemical stability. This work presents a fabrication technique based on physical vapor deposition for prepared ceramic nanostructure as sensing materials. Firstly, the glancing angle deposition technique was utilized to architecture metal oxide nanostructure-based chemiresistive gas sensors. The effects of deposition parameters on morphologies, physical structures, composition, and VOCs sensing properties were investigated. For optical sensing devices, by controlling kinetic energy during the sputtering process, high porosity metal oxide waveguide film can be achieved with high chemical gas sensitivity, which allows more molecular diffusion and adsorption on nanostructure sensors. Combined with data processing to extract meaningful information from electrical and optical sensor devices, data analysis as machine learning has been proposed and discussed to identification VOCs.

3:50 PM

(ICACC-PACRIM-011-2023) Growth of β-MoO_3 whiskers for medical radiopharmacy applications (Invited)

H. Suematsu*[1]; N. M. Chu[2]; Y. Yang[1]; T. Do[3]; T. Nakayama[1]; K. Niihara[1]

1. Nagaoka University of Technology, Extreme Energy-Density Research Institute, Japan
2. AIST, Japan
3. Nagaoka University of Technology, Nuclear System Safety Engineering, Japan

^{99}Mo is a mother nuclide of ^{99m}Tc, which is a γ-ray emitter and has been used in radiopharmacies. ^{99}Mo has been prepared from highly enriched uranium spent fuel in nuclear reactors, which have nuclear security risks and are not newly built. Instead, a nuclear reaction method to irradiate neutrons onto α-$^{98}MoO_3$ targets has been investigated. Comparing to conventional α-MoO_3, in our preliminary neutron irradiation experiments, β-MoO_3 shows superior ^{99}Mo extraction characteristics to water because of the larger spacing between Mo-O layers. In order to separate water to the irradiated β-MoO_3 targets by a filter, growth of β-MoO_3 whiskers by pulsed wire discharge and evaporation methods for the first time. In the latter method, α-MoO_3 powder was heated to 750-1000°C in a tube furnace with a long cooling zone. With an O_2 gas flow, the MoO_3 vapor was transferred to the cooling zone to grow β-MoO_3 whiskers below 450°C. The average width was 10nm and the length was approximately 300nm. The β-MoO_3 whiskers are suitable irradiation targets to prepare ^{99}Mo.

4:10 PM

(ICACC-PACRIM-012-2023) Phase transformations driven by complex atomic interactions in layered sodium transition metal oxides (Invited)

J. Kim*[1]

1. Stevens Institute of Technology, Chemical Engineering & Materials Science, USA

As an alternative to Li-based technology for large-scale applications, Na-ion batteries composed of earth-abundant elements have attracted substantial interest. Designing Na storage cathode materials that operate reversibly at high voltage is key to developing high-performance Na-ion batteries. Layered oxide cathodes have ideal Na storage frameworks that are known to undergo reversible phase transformations upon Na intercalation. However, large desodiation often leads to an unclarified, irreversible phase transformation at high voltage due to complex interatomic interactions, hindering the full use of transition metal (TM) redox. In this talk, how TM chemistry affects the stability of desodiated sodium TM oxides will be discussed, and recent findings on a quaternary TM system including Ti and Fe will be underlined. A highly-desodiated phase that exhibits peculiar oxygen stacking to afford alternating octahedral and prismatic Na layers, namely OP2 stacking, will be demonstrated. The formation of OP2 is rationalized by distortion-tolerant Ti and Jahn-Teller-active Fe. This new phase participates in redox reaction reversibly, fundamentally distinct from inactive high-voltage phases in many Na-ion cathodes.

4:40 PM

(ICACC-PACRIM-013-2023) Design and Analysis of Additively Manufactured Battery Thermal Management Systems

W. Nelson*[1]; A. S. Almansour[2]; M. Singh[3]; M. C. Halbig[4]; E. McNichols[4]

1. St. Cloud State University, USA
2. NASA Glenn Research Center, Mechanical Engineering, USA
3. Ohio Aerospace Institute, USA
4. NASA Glenn Research Center, USA

Thermal management of battery cell packs is a critically needed technology. The purpose of this work is to design new and improved Battery Thermal Management Systems (BTMS) for use in electric airplanes. The BTMS should be 3D printable and hold twelve to sixteen 18650 batteries. To minimize aircraft's weight, an actively air-cooled battery pack was chosen to avoid the excess weight of water-cooled and phase change material (PCM) cooled packs. As a result, significant changes were made to remove all metal in the pack and replace it with lower density polymer matrix composites (PMCs) which can be additively manufactured. Two air cooled battery pack configurations (traditional propeller fans & bladeless fans) were designed, modeled, and compared. These packs were first modeled in SolidWorks 2021 3D CAD, then imported into COMSOL MultiPhysics to be studied using the "Heat Transfer in Solids and Fluids" module. The design with the bladeless fans eliminated the need to use high conductivity heavy metal to remove unwanted thermal energy. These bladeless fans were designed entirely out of PMCs. This thermal pack design weighs 0.04 kg less than that of the traditional propeller fans design and has increased the battery pack energy density by 8.25 Wh/kg.

FS2: Materials for Thermoelectric and Thermionic Energy Conversion

Thermoelectric Generators I

Room: Flagler A (South Tower)
Session Chair: Mofasser Mallick, Karlsruhe Institute of Technology South Campus

1:30 PM

(ICACC-FS2-010-2023) Thermoelectric Materials for Low Temperature Power Generation (Invited)

Z. Ren*[1]

1. University of Houston, Physics & TcSUH, USA

Mg_3Sb_2-based materials have been extensively studied in the recent years because of their high thermoelectric figure-of-merit (ZT) at 500 °C. However, it is widely reported that they are thermally instable at above 300 °C, which is a serious problem for power generation above this temperature. We have carried out an extensive research and found the mechanism of the instability. Furthermore, we have developed effective methods to overcome the thermal instability to make the materials stable at up to 400 °C, which makes power generation at this temperature possible. The modules working at this temperature exhibit a power conversion efficiency of about 12%.

2:00 PM

(ICACC-FS2-011-2023) Robust thermoelectric modules based on MgAgSb and $Mg_3(Sb,Bi)_2$ with conversion efficiency of 8.5% and maximum cooling of 72 K (Invited)

K. Nielsch*[1]

1. Leibniz Institute for Solid State and Materials Research, Institute of Metallic Materials, Germany

The applications of thermoelectric (TE) technology around room temperature are dominated by bismuth telluride (Bi_2Te_3). Due to the scarcity of tellurium (Te), it is vital to develop a next-generation technology to mitigate the potential bottleneck in raw material supply for a sustainable future. We developed a Te-free n-type compound $Mg_3Sb_{0.6}Bi_{1.4}$ for near-room-temperature applications. The optimizations of processing and doping yield a high average zT of 1.1 in between 300 K and 573 K. Together with the p-type MgAgSb, we demonstrate module-level conversion efficiencies of 3% and 8.5% under temperature differences of 75 K and 260 K, respectively, and concomitantly a maximum cooling of 72 K when the module is used as a cooler. TheTE-free module exhibits exceptional thermal robustness with a < 10% loss of the output power after thermal cycling for 32000 times between 323 K and 500 K. These proof-of-principle demonstrations will pave the way for robust, high-performance, and sustainable solid-state power generation and cooling to substitute Bi_2Te_3.

2:30 PM

(ICACC-FS2-012-2023) Thermoelectric oxides for Transverse Multilayer Thermoelectric Generators (Invited)

J. Topfer*[1]; R. Loehnert[1]; A. Bochmann[1]; A. Ibrahim[1]; B. Capraro[2]

1. Ernst-Abbe-Hochschule Jena, Germany
2. Fraunhofer IKTS, Germany

Oxide thermoelectric generators (TEG) are typically fabricated using the standard dual-leg design including manufacturing, arranging and contacting many individual sintered p- and n-type ceramic blocks. Alternatively, application of the ceramic multilayer technology represents a promising option enabling miniaturization and easy fabrication of multilayer TEGs. We present the concept of transverse multilayer thermoelectric generators (TMLTEG) with charge transport perpendicular to the heat flow direction. Such generators consist of layers of tape-cast p- or n-type thermoelectric oxides in combination with metal layers printed at a certain angle with respect to the heat flow direction to create anisotropic thermoelectric properties. We report on sintering behavior and thermoelectric properties of the individual thermoelectric oxides, as well as on co-firing behavior of metal and oxides. Based on analytical calculations, we have developed the concept of Babin-plots, describing the power output and conversion efficiency for a given material combination as function of the internal and external device geometry. We demonstrate this approach in combination with device simulations for pellet-based and multilayer transverse generators. We will report on the fabrication of TMLTEGs using various thermoelectric oxides, e.g., $Ca_3Co_4O_9$, La_2CuO_4 and $CaMnO_3$ for low-power applications, e.g. autonomous sensor networks.

Thermoelectric Generators II

Room: Flagler A (South Tower)
Session Chair: Kornelius Nielsch, Leibniz Institute for Solid State and Materials Research

3:20 PM

(ICACC-FS2-013-2023) High ZT telluride-based flexible thermoelectric films through interfacial modification via millisecond photonic-curing for fully printed TEGs (Invited)

M. Mallick*[1]; L. Franke[1]; A. Rösch[1]; U. Lemmer[1]

1. Karlsruhe Institute of Technology South Campus, Light Technology Institute, Germany

Due to complex manufacturing processes, the low-cost production of bulk thermoelectric (TE) devices is a challenge. In addition, the conventional TE devices are neither flexible nor shape-conformable; hence, it is difficult to integrate on non-flat surfaces. Printed technologies could reduce TE devices' manufacturing costs and offer shape conformity. The conductive polymers have been targeted for printed TE applications; however, low efficiency than their inorganic-based counterparts limits their applications. Here, we report flexible $(SbBi)_2(TeSe)_3$-based screen-printed TE films exhibiting record-high performances. We have developed a strategy to fabricate the high-efficiency printed TE films through interfacial modification using a novel Cu-Se-based inorganic binder minimizing the detrimental effects of binder and solvent at grain boundaries. As a result, a high power factor of 24 μWcm^{-1}K^{-2} (ZT$_{max}$=1.45) for a p-type film and a power factor of 10.5 μWcm^{-1}K^{-2} (ZT$_{max}$=0.75) for an n-type film are achieved. Using printable materials, different printed TE devices have been fabricated. A half-millimeter thick f-TEG exhibits an open-circuit voltage (V$_{OC}$) of 203 mV with a maximum power density (p$_{max}$) of 5.1 Wm^{-2} at ∇T= 68 K. This result signifies a few millimeters thick f-TEG could power IoT devices converting low-grade heat to electricity.

3:50 PM

(ICACC-FS2-014-2023) Thermoelectrics for direct conversion of industrial waste heat into useful electrical power (Invited)

J. Cho*[1]

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

Thermoelectric power generation technology has been considered as a promising green technology due to its static and simple application to resolve a growing concern of environment/climate change and the limitation of energy resources. However, to date high cost and limited research progress on thermoelectric power generation technology allows it to be only utilized in niches markets or in lower temperature applications. Many issues still remain unresolved to bring this sustainable and green technology to access an untouched and huge potential of industrial mid to high temperature waste heat recovery. In this presentation, I will start by discussing the challenges and opportunities associated with thermoelectric power generation technology comparing to other renewable energy technologies. Then, thermoelectric materials and module work at Korea Institute of Ceramic Engineering & Technology (KICET) for mid to high temperature range targeting industrial waste heat will be discussed. Finally, I will introduce the ongoing several thermoelectric power generation projects for industrial waste heat recovery at KICET.

4:20 PM

(ICACC-FS2-015-2023) Colossal Nernst power factor in topological semimetals for Ettingshausen refrigeration (Invited)

Q. Li*[1]

1. Stony Brook University and Brookhaven National Laboratory, USA

Today solid-state cooling technologies below liquid nitrogen boiling temperature (77 K), crucial to quantum information technology and probing quantum state of matter, are greatly limited due to the lack of good thermoelectric and/or thermomagnetic materials. In this talk, I will discuss a number of recent results showing colossal Nernst power factor at low temeatures and strong magnetic fields in topological semimetals, which is orders of magnitude higher than other conventional thermoelectric and thermomagnetic materials at a similar temperature range. The observed high Nernst figure-of-merit is attributed to large Nernst thermopower and longitudinal electrical conductivity, and relatively low transverse thermal conductivity. Theser results open a new avenue for exploring material option for the solid-state heat pumping below liquid nitrogen temperature.

S1: Mechanical Behavior and Performance of Ceramics & Composites

Processing, Testing, and Characterization of Ceramic Fibers and Ceramic Matrix Composites (CMCs)

Room: Ballroom 5 (South Tower)
Session Chairs: Jason Baker, Rolls-Royce Corporation; Monica Ferraris, Politecnico di Torino

1:30 PM

(ICACC-S1-018-2023) Diametral C-ring Compression Strength of SiC Composite (Invited)

J. Park*[1]; W. Kim[2]

1. Korea Atomic Energy Research Institute, Nuclear Materials Development Division, Republic of Korea
2. Korea Atomic Energy Research Institute, Republic of Korea

SiC tubular geometry is applied for various high temperature structural components and required evaluation of the mechanical properties. Flexural tests for uniaxial load testing, which are widely used, are difficult or impractical to apply to tubes and other cylindrical specimens. There are several test methods for evaluating the strength of tubular specimens, such as a hoop test, an O-ring test, and a C-ring test. Among them, the diameteral C-ring compression method is effective for evaluation of tubular specimens due to its advantages such as simple shape, easy processing, high temperature testing and statistical data collection in a relatively small amount of the samples. In this paper, CVD-SiC monolith and different SiC$_f$/SiC composites were prepared to evaluate the diametral C-ring compression strength of SiC tube. SiC$_f$/SiC composite tubes were fabricated through CVI using Tyranno-SA^{TM} fibers with methyltrichlorosilane(CH_3SiCl_3). C-ring geometry specimens with various b/t (width/thickness of specimen) values were prepared by slicing each tube specimen and machining the slot. Diametral compression tests of the prepared C-ring specimens were conducted according to the ASTM C1323-16. The effect of the b/t ratio on the C-ring compression strength was evaluated.

2:00 PM

(ICACC-S1-019-2023) Non-Destructive Evaluation of processing defects in fiber reinforced, MI-SiC/SiC using electrical resistance

A. Gupta*[1]; G. N. Morscher[1]

1. University of Akron, Dept. of Mechanical Engineering, USA

Electrical resistance has been shown to be effective at detecting matrix cracks and delamination cracks in Si melt-infiltrated composites. Since the resistivity of the composite is dependent on the most conductive constituent, processing defects such as porosity should be detectable by differences in local electrical resistance. Several 2D woven melt-infiltrated SiC/SiC composites with different types and degrees of porosity were examined to assess this hypothesis. A number of local four-point probe measurements were made on the specimens along the axial length of a dogbone tensile specimen in the gauge section. Different variations of the axial-type measurement are introduced by varying Id, Vd, and both Id & Vd, where Id represents the distance between the positive and negative of the current lead pair and Vd represents the distance between the positive and negative of the voltage lead pair. The measurements were sensitive to two different types of porosity cases: (1) poor infiltration of Si into the SiC slurry, referred to as “dry slurry”, and (2) more rounded porosity due to internal gas formation. The results and sensitivity will be presented and discussed.

2:20 PM

(ICACC-S1-020-2023) Strategic production method of cost-competitive SiC fibers via innovative precursor synthesis

R. Naito*[1]; R. Iuchi[2]; J. Saito[1]; T. Goto[2]; K. Yamakawa[3]

1. Kureha Corporation, Organic Synthesis Research Department, Japan
2. Kureha Corporation, Process Innovation Department, Japan
3. Kureha Corporation, New Business Creation Project, Japan

SiC fibers are promising components with superior mechanical strength and thermal stability for aerospace application as ceramic matrix composites. Conventional SiC fibers production requires high energy and long time in electron beam based polycarbosilane(PCS) curing process, which results in high material cost and limits its suppliability. To address these critical challenges, new strategy for manufacturing SiC fiber was established. This new method can eliminate the electron beam irradiation by using novel polycarbosilane, while still providing the same high mechanical and temperature properties as the conventional fibers such as Hi-Nicalon Type-S. A newly discovered parameter relating to the branched PCS allows quantitative discussion of the relationship between the PCS properties and the physical properties of produced SiC fibers. Utilizing this parameter, the optimal combination of raw materials and synthesis method was found to obtain SiC fibers with excellent physical properties as targeted.

2:40 PM

(ICACC-S1-021-2023) Characterization of SiC fiber obtained by a cost-competitive manufacturing method using our proprietary polycarbosilane as the key material

R. Iuchi*[1]; R. Naito[2]; J. Saito[2]; T. Goto[1]; K. Yamakawa[3]

1. Kureha Corporation, Process Innovation Department, Japan
2. Kureha Corporation, Organic Synthesis Research Department, Japan
3. Kureha Corporation, New Business Creation Project, Japan

SiC fiber utilized in ceramics matrix composite as aircraft engine components, combines optimal crystallite size, appropriate C/Si stoichiometry ratio, and low oxygen content to exhibit excellent mechanical strength under extreme high temperature environment. In the recent study, new parameters were discovered in polycarbosilane (PCS) as precursor of SiC fibers for excellent mechanical properties of produced SiC fibers. This discovery leads to an innovative manufacturing method without electron beam irradiation curing which results in high SiC fiber manufacturing cost. PCS with these parameters optimized was also developed. In this work, characterization of SiC fibers made from this developed PCS were conducted. It remains excellent mechanical strength even after exposure to 1500 deg C in an argon atmosphere. The factors contributed to such performance were also discussed through structural and chemical analyses.

3:20 PM

(ICACC-S1-022-2023) Processing and mechanical evaluation of oxide-oxide ceramic matrix composites manufactured using automated fibre placement

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2. University of Birmingham, Ceramic Science & Engineering, United Kingdom
3. Rolls-Royce Derby, United Kingdom

Oxide-oxide ceramic matrix composite (CMC) manufacture can involve manual slurry infiltration into pre-cut fabric sheets, followed by hand lay-up onto a mould of required shape. Automated fibre placement (AFP), previously used for production of polymer matrix composites, promises a capability for highly reproducible laminate production of complex geometries using towpreg feedstock. The process of AFP involves using a computer controlled loaded roller to place layers of towpreg material, containing a dry matrix, onto a mould. However, the optimal parameters for further processing of laminates from the as-laid state to final sintered component are currently unknown. This work presents a processing study of AFP laminates undertaken using both heat press curing, which tends to result in delaminated samples after sintering, and the use of autoclave-based curing cycles. The eventual aim of this project is to compare the mechanical performance of optimised, commercially available AFP laminates to those manufactured in-house, from spools of towpreg material. Other considerations will also be explored, such as the towpreg tack and behaviour of green bodies after AFP. Imaging of the material using non-destructive techniques, such as micro-CT, may give an insight into defect distribution to allow greater understanding of the mechanisms occurring during processing.

3:40 PM

(ICACC-S1-023-2023) Automated spraying process for the manufacturing of short fiber oxide fiber composites

G. Puchas*[1]; J. Winkelbauer[1]; S. Schafföner[1]; W. Krenkel[1]

1. University of Bayreuth, Ceramic Materials Engineering, Germany

Oxide fiber composites (OFC) combine lightweight construction potential with excellent thermomechanical behavior in oxidizing and corrosive atmospheres. Currently, OFC are predominantly manufactured with continuous fiber reinforcements, mostly fabrics. For more complex structures with spherical geometries and narrow radii, fabrics quickly reach their limits due to their poor drapeability. For such applications, drapeable short-fiber reinforcements are more suitable and allow a considerable reduction in material costs compared to fabric reinforcements. Hence, a novel automated fiber spraying process for the production of short fiber bundle reinforced OFC was developed, in which continuous oxide fiber bundles are chopped to a predefined length and immediately entrained by a slurry jet. The fiber bundles are infiltrated by slurry in flight and layerwise applied to the mold together with excess slurry. The resulting preimpregnated fiber preforms (prepregs) are conditioned, laminated or formed if necessary, consolidated and finally sintered. The significance of various process parameters on the specimen strength was analyzed and the novel material was characterized with regard to fiber length and orientation. The sprayed short fiber OFC showed in-plane isotropic material properties and achieved a mean bending strength of up to 163 MPa, strains of 0.64 % and a quasi-ductile fracture behavior.

4:00 PM

(ICACC-S1-024-2023) Improving the mechanical performance of phosphate-based ceramic matrix composites

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1. University of Birmingham, Ceramic Science & Engineering, United Kingdom
2. BAE Systems, United Kingdom

Oxide ceramic matrix composites based on alumina with a continuous fibre reinforcement can achieve tensile strengths of 133 MPa using conventional sintering methods. These systems are well defined and are used in a wide variety of applications, however other systems are being sought for structural applications requiring greater strength at elevated temperatures. In recent years' phosphate systems, such as dental cement and chemically bonded phosphates, have been investigated to fulfil this role. However, they are not as strong as traditional oxide CMCs. This work aims to improve the mechanical performance of a phosphate-based system to match that of a conventional alumina system whilst using Hiltex DC3 as the reinforcing media. A number of different routes are being investigated to achieve this goal, these include directly modifying the matrix with silica prior to infiltration and modifying grain boundaries using sintering aids during the heat treatment process. After

processing, samples are heat treated before undergoing tensile testing using a 4-point-bend facility, after which the fracture surface is observed using an SEM to further evaluate their performance.

4:20 PM

(ICACC-S1-025-2023) Strength Degradation Due to Solid Particle Erosion at Elevated Temperatures of Oxide/Oxide Ceramic Matrix Composites

F. Mirza*[1]; R. Panakarajupally[1]; J. El Rassi[1]; G. N. Morscher[1]; F. Abdi[2]; S. R. Choi[3]

1. University of Akron, Mechanical Engineering, USA
2. AlphaSTAR Corporation, USA
3. Naval Air Station Patuxent River, USA

An oxide/oxide ceramic matrix composites erosion behavior was characterized at 800 °C and 1200 °C environment. The effect of erodent particle velocity and temperature were investigated. Microscopic analysis was done to understand intensity of damage through depth profiling before room temperature mechanical testing. The room temperature tensile strengths with digital image correlation were performed to evaluate retained mechanical properties of oxide composites. The results indicate that erosion rate increases with increase in particle velocity and temperature, the former being more consequential. Residual strength tests indicate that specimens impacted at higher velocity and temperature showed lower strengths due to increasing crater depth. The erosion behavior was similar to previous results on oxide/oxide composites; however greater strength was degradation was observed in this study

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

Advanced Ceramic Coatings for Extreme Environments II

Room: Flagler C (South Tower)
Session Chair: Peter Mechnich, DLR - German Aerospace Center

1:30 PM

(ICACC-S2-020-2023) Aerosol Deposition – novel fields of applications beyond semiconductor business

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The Aerosol Deposition is a new coating method based on the mechanisms of Room Temperature Impact Consolidation. A special feature and a clear advantage of this new technology is that the coating of all kinds of materials on all substrate types is done entirely at room temperature at high deposition rates. While this coating technology is well established in semiconductor equipment fabrication, e.g. yttria based coatings in plasma-etch chambers, few applications are disclosed beyond this industry. In the last two years Heraeus continuously developed the aerosol deposition to optimize processes and machines for production on industrial scale and subsequently broadened the application range of the Aerosol Deposition Method. In this contribution two potential applications are presented on the field of sensing and high-temperature applications. The first case features the production of High-Temperature Sensors. These are usually coated with a protective PVD layer to secure performance. By using aerosol deposited α-Al_2O_3, long term stability is improved, and measurement accuracy increased over the life span of the sensor. The second case study deals with protective coatings for high temperature nickel alloys that are commonly used in kilns, e.g. for battery material synthesis. By using aerosol deposited Al_2O_3 coatings the service life of the system parts can be extended while reducing flaking significantly.

1:50 PM

(ICACC-S2-021-2023) Advanced ceramic coatings on aluminum by laser treatment of filled organosilazane-based composites

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1. University of Bayreuth, Ceramic Materials Engineering, Germany
2. Bayerisches Laserzentrum GmbH, Germany

Aluminum alloys are particularly suitable for structural components due to their high specific strength and stiffness. However, the low hardness and wear resistance limits the use of aluminum. To increase the surface hardness, ceramic-based coatings are frequently the most suitable solutions. A suitable method for the preparation of ceramic coating is the precursor technology. Nevertheless, the required high temperature for the ceramization of the precursor-based coatings in a furnace limits this approach to high temperature-resistant substrates. Laser radiation as a heat source for the pyrolysis of the preceramic polymer is an approach to overcome this restriction. Therefore, a coating system for aluminum substrates was developed, consisting of a polysilazane (Durazane 2250) bond-coat and a hard top-coat composed of an organosilazane (Durazane 1800) with tetragonal ZrO_2 and aluminum fillers pyrolyzed using Nd:YVO4 laser. The laser irradiation led to pyrolysis of the silazane and to a dendritic microstructure, indicating complete melting of the fillers. Despite the high temperatures within the coating, thermally-activated processes in the substrates as a result of laser irradiation could be excluded. The laser-treated coatings exhibited good adhesion to the substrate, even under bending load. The hardness of the laser treated coating was determined by nanoindentation measurements.

2:10 PM

(ICACC-S2-022-2023) A Novel Method of Surface Fluorination of Y_2O_3 Ceramics via Gas Phase Reaction

S. Lee*[1]; K. Bae[2]; Y. Oh[1]

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

Yttrium-based ceramics have been widely used in the semiconductor industry as plasma facing materials. In their application, a fluorin to oxygen ratio is the crticital parameter for reducing the generation of contamination particles. In this study, we are proposing a novel method to modifiy the surface of Y_2O_3 ceramics into the fluorine containing YO_xF_y phase via gas phase reaction. When sintered bulk or thin films of Y_2O_3 were exposed to the fluorine-containing vapor, which was evaporated from NH_4F source in the crucible, their surface was modified to YO_xF_y. The main benefit of this method is that the vapor from NH4F source is neutral, not acidic nor basic, causing no harmful erosion of the equipments and their treatment can be done at relative low temperatures. The surface modificaiton and their thickness depended on the exposed time and temperature. In this study, the mechanism of the surface modification will be proposed.

2:30 PM

(ICACC-S2-023-2023) Thermally and mechanically stable superhydrophobic glass coatings containing nanoparticles

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

Superhydrophobic coatings continue to be of great interest in science and industry due to their wide range of applications. Functional surface coatings that protects a surface from environmental factors such as heat, moisture and rain result in longer surface durability. As an alternative to oxides such SiO_2, TiO_2 and hexagonal Boron nitride (hBN) particles can also be used for coating due to its high temperature stability, inert properties and antimicrobial effect. In a first step hBN particles were functionalized with 1H,1H,2H,2H-perfluorooctyl-triethoxysilane (PES). Glass coatings containing functionalized nanoparticles were prepared on soda lime substrates leading to a thermally stable (400 °C) superhydrophobic surface (≈ 180°). In addition, the commercially available polymer based resins Silikoftal® HTT and Silikophen® P80/X was used as an adhesive system. After several scrubbing cycles of the coating on a sponge, the contact angle never decreased below 130°, showing high mechanical stability. The particles were investigated by x-ray diffraction (XRD), fourier transform infrared (FTIR) spectroscopy and the coatings were examined by scanning electron microscopy (SEM), atomic force microscopy (AFM), scratch test and contact angle measurements.

CMAS-related Degradation and Mitigation Strategies I

Room: Flagler C (South Tower)
Session Chair: Peter Mechnich, DLR - German Aerospace Center

3:20 PM

(ICACC-S2-024-2023) Thermochemistry of Reactions between Ceramic Coating Materials and Silicate Melts (Invited)

G. Costa*[1]

1. NASA Glenn Research Center, USA

The thermochemistry involved in the corrosion of ceramic coating materials at high temperature is crucial for thermodynamic modeling of phase equilibria to improve their durability in aerospace applications. One such emerging area is the corrosion of gas turbine engine coatings by molten silicate debris. Here, we summarize and discuss our previous and current studies of using high temperature reaction calorimetry to evaluate the energetics of reactions of ceramic coating materials and their binary oxide components in silicate melts with varying network former SiO_2 mol percentage in the $CaO-MgO-Al_2O_3-SiO_2$ (CMAS) system. The solution and mixing enthalpies indicate that the energetic stability of coating materials and their binary oxide components with the CMAS melts depends on the difference between their acid-base character. Essentially, a decreasing difference in acid-base character between the materials studied and the CMAS melts correlates to an increasing energetic stability between those selfsame materials and melts. Moreover, the stability of the coating materials against silicate melt corrosion is expected to increase as their formation enthalpies become less exothermic (more positive). Implications of these results on predicting the energetic contribution of each binary oxide component on the silicate-induced corrosion of the coating materials and melt structure are discussed.

3:50 PM

(ICACC-S2-025-2023) Interactions between EBCs and CMAS: A thermochemical approach

J. Bonnal*[1]; C. Petitjean[1]; P. Panteix[1]; D. Bonina[1]; C. Gendarme[1]; S. Arnal[2]; M. Vilasi[1]

1. Université de Lorraine, CNRS, IJL, France
2. Safran Ceramics, France

Environmental Barrier Coatings (EBCs) are required for the protection of CMC used in turbine gas engines against degradation by combustion products. In this application, rare-earth (RE) silicates are considered as good candidates. However, the development of EBCs is hindered by the high-temperature corrosion due to interactions with liquid calcium-magnesium-aluminosilicates (CMAS). Indeed, the infiltration of these silicate melts into the coating causes thermochemical degradations by acido-basic dissolution of the RE-silicate in the melt and the precipitation of new phases. Many parameters, as the temperature, the composition (i.e. basicity, viscosity) of the melt, or the nature of the EBC, can modify the equilibrium between RE-silicate and CMAS. The aim of this work is to study the mechanism of interaction between yttrium disilicate (YDS) and a simplified molten silicate at 1300°C by determining: (i) the solubility limits of the species, (ii) the nature of the precipitated phases, and (iii) the influence of the melt composition, which can be locally modified during the dissolution/precipitation process. An original device has been used to provide optimal reproducibility and allow fast attainment of the thermodynamic equilibrium. A complementary thermodynamic study brings further information about the equilibria and the evolution of the extent of the liquid domain.

4:10 PM

(ICACC-S2-026-2023) Modeling and design of CMAS-resistant multiphase T-EBC with CALPHAD tools

N. Arai*[1]; O. Kontsevoi[1]; Z. Liang[1]; D. L. Poerschke[2]; E. P. Godbole[3]; Y. Yu[2]; J. Gong[1]

1. QuesTek innovations, USA
2. University of Minnesota, Chemical Engineering and Materials Science, USA
3. University of Minnesota, Twin Cities, Chemical Engineering and Materials Science, USA

The temperature capability of ceramic matrix composites for jet engines and gas turbines is still limited by the lack of environmental durability of coatings. One of the main degradation mechanisms at high temperatures is due to CMAS deposit. CMAS deposits exist in a wide composition range which changes the melting temperature, the fraction of melt at a given temperature, viscosities of that melt, resulting in changes in the reaction products as well as different attack mechanisms. These considerations make the design of CMAS-resistant thermal and environmental barrier coatings (T-EBCs) challenging, especially through the conventional "trial-and-error" approach, which is time-consuming and impractical. Hence, there is a critical need for a high-throughput computational approach to model T-EBC and its interaction with CMAS based on composition and service temperature. In this study, a computational thermodynamic approach (CALPHAD) to model the microstructure and properties T-EBC and CMAS systems will be presented. The microstructure of selected rare-earth zirconate-aluminate systems before CMAS exposure and reaction products after CMAS exposure was modeled and compared to results from the validation experiments. Modeling of key properties such as viscosity will also be presented. These modeling approaches aim to establish a computational framework to accelerate the design of CMAS-resistant T-EBCs.

4:30 PM

(ICACC-S2-027-2023) Thermochemical/Thermomechanical Synergies in High Temperature Particle Erosion of CMAS Exposed EBCs

J. L. Stokes*[1]; M. J. Presby[1]; R. Webster[1]; J. Setlock[2]; B. J. Harder[1]

1. NASA Glenn Research Center, Environmental Effects and Coatings Branch, USA
2. University of Toledo, USA

Environmental barrier coatings (EBCs) are an enabling technology for the use of SiC-based ceramic matrix composites in next generation gas turbine engines. In the extreme environment within the hot section of the engine, EBCs must be able to withstand a variety of damage mechanisms and the synergies of degradation between them. Ingested particulate/debris can cause both thermochemical and thermomechanical degradation of EBCs. Siliceous debris primarily based on calcium magnesium aluminosilicates (CMAS) can become molten, infiltrate and react with EBCs at high temperatures. Similarly, ingestion of any debris in the engine can lead to mechanical damage and recession of coatings due to particulate erosion. Because both modes of degradation can occur simultaneously during engine operation, it is crucial to understand the mechanisms of coating failure due to high temperature particulate interactions. This study assesses the erosion durability of $Yb_2Si_2O_7$-based EBCs reacted with CMAS at various loadings in NASA Glenn's Erosion Burner Rig Facility. CMAS exposures and erosion testing were carried out at 1316°C. Using 60 μm Al_2O_3 particles as the erodent material, the effects of CMAS loading and time of exposure on erosion durability were evaluated.

4:50 PM

(ICACC-S2-028-2023) Rare-earth Monosilicate Interactions with Calcuim-magnesium Aluminosilicate

C. Miller*[1]; E. Opila[1]

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

Rare-earth silicates are excellent candidate materials for environmental barrier coatings in next generation aero turbine engines. Siliceous debris, such as ash and dust, can be ingested into engines and deposits as molten calcium-magnesium aluminosilicate (CMAS) glass on hot section components, degrading them. Single-cation rare-earth monosilicates were exposed to CMAS for 1-24h at 1300°C in a stagnant air box furnace, and their reactions characterized by SEM, EDS and XRD. Monosilicates containing the largest and the smallest rare-earth cations formed the thinnest, most protective reaction product layers, while the middle of the rare-earth series produced thick, low density reaction layers. Their reaction kinetics were investigated, and rates of reaction determined. These results are important for the selection of constituents used in multicomponent silicate coatings, which are designed for enhanced thermal properties in addition to thermochemical stability.

S3: 20th International Symposium on Solid Oxide Cells (SOC): Materials, Science and Technology

Proton Conducting Ceramic Cells

Room: Ponce de Leon (North Tower)
Session Chairs: Eric Wachsman, University of Maryland; Jakub Kupecki, Institute of Power Engineering

1:30 PM

(ICACC-S3-020-2023) Proton ceramic electrolysers: An overview of pressurized tubular stack technology (Invited)

M. Fontaine*[1]; E. Vøllestad[1]

1. SINTEF AS, Sustainable Energy Technologies, Norway

Tubular Proton Ceramic Electrolyser (PCE) stack technology is developed to produce pure dry pressurized hydrogen using high volume production of tubular PCEs containing BZCY-based electrolyte, BZCY-Ni tubular hydrogen electrode and BGLC-BZCY composite steam + O_2 electrode. The cells can be operated in both fuel cell and electrolysis modes with durable performance. For upscale of the technology with pressurized operation, the cells are integrated in tubular steel shells forming "single engineering units" (SEU). SEUs have ca. 50-60 cm^2 active surface area and are operated in pressurized steam electrolysis operation at intermediate temperature (600°C) and up to 10 bar pressure with high faradaic efficiency and reasonable area specific resistance. SEUs assembly consisting of 16 SEUs mounted in series has been produced and tested in a container plant equipped with necessary balance of plant components. This presentation gives an overview of the technology, spanning from cell manufacturing and SEU production, SEU testing up to 10 bar, the rack design and assembly development together with system design, building and testing.

2:00 PM

(ICACC-S3-021-2023) Tubular Solid Oxide Cell Testing (Invited)

Y. Du*[1]; D. Panthi[2]

1. Kent State University, USA
2. Kent State University, Engineering Technology, USA

Solid oxide fuel cells (SOFCs) and solid oxide electrolysis cells (SOECs) are of interest in the transition from our current fossil fuel era to a renewable energy future. Solid oxide cells (SOCs), including SOFCs and SOECs, could be made in a planar or tubular geometry. Both configurations possess pros and cons. Strategies and practical operations are different in cell preparations and setups for testing. Easy preparation and setup for acquiring reliable and repeatable data are the goal for many, especially those who newly enter the SOFC and/or SOEC field. This paper reviews the common cell designs, discusses the challenges facing the SOC testing setups, suggests potentially available solutions, and shares the authors' own research in this field.

2:30 PM

(ICACC-S3-022-2023) Current status of protonic ceramic fuel cell development at KIST (Invited)

J. Lee*[1]; H. Ji[1]; J. Kim[1]

1. Korea Institute of Science and Technology, Republic of Korea

Proton conducting oxides have been widely adopted to electrochemical devices for efficient energy conversion and storage due to their low cost and high ionic conductivity. In spite of these advantages, protonic ceramic fuel cells (PCFC) did not prove its worth in the actual field. Especially, distinct skepticism still remains about the applicability of PCFCs to the level of commercialization owing to their lower-than-predicted performance and difficulty with scale-up in cell fabrication. These challenges mainly stem from the refractory nature of proton-conducting ceramic electrolytes and the low chemical stability of PCFC materials during the sintering process. Recently, we have demonstrated a strong feasibility of high performance and scalability of PCFCs through facile anode-assisted densification of the protonic ceramic electrolyte on an anode support. According to our results, an internal supply of the transient phase from the anode to the electrolyte is the key factor to promote the densification of the electrolyte at a much lower temperature than normal. However, although we succeeded in obtaining thin and dense membrane, a exact explanation on underlying mechanism for enhanced densification is not established yet. Here we report the first proof-of-mechnism in formation and supplying process of vapor-phase sintering activator for refractory proton-conducting oxide.

3:20 PM

(ICACC-S3-023-2023) Intermediate Temperatures Solid State Energy Conversions by Protonic Ceramics: A Key for Cost-Effective Decarbonized Economy (Invited)

D. Ding*[1]

1. Idaho National Lab, Hydrogen and Electrochemistry, USA

Highly efficient energy conversion technologies hold the promise for the future of the decarbonized society. However, the cost is always the obstacle for their penetration to the market. How to make them cost-competitive is precisely the objective of Dr. Ding's team at INL. Their vision is to use their technical experience, capability, and scientific approach to develop new electrochemical processes via intermediate temperature. These technologies can reach the sweet zone by significantly reducing the PGM and relevant material cost associated with the low-temperature electrochemical technologies while bringing in cost benefits of the stack component and BOP compared with the conventional high temperature electrochemical ones. Dr. Ding will give several special cases on intermediate temperatures solid state energy conversions using protonic ceramic electrochemical cells (PCECs) at Idaho National Laboratory and offer his perspective on PCECs.

3:50 PM

(ICACC-S3-025-2023) Rejuvenating the Electrolyte Surface of Protonic Ceramic Electrochemical Cells by Acid Etch

W. Bian*[1]

1. Idaho National Lab, USA

The coupled electrochemomechanics at the electrode–electrolyte interface affects the thermomechanical integrity, microstructural stability, electrochemical performance, and durability of ceramic electrochemical cells, which become critically paramount for high performance protonic ceramic electrochemical cells (PCECs). In this presentation, we present our recent result that demonstrates a simple acid treatment can effectively rejuvenate the high-temperature annealed electrolyte surface, resulting in reactive bonding between the oxygen electrode and the electrolyte and improved electrochemical performance and stability of PCECs for hydrogen production and power generation. This scalable approach has implications to be utilized for large cell manufacturing and accelerate the technology penetration. Additionally, it can expand PCEC's application for chemical and ammonia production. Through this talk, we will also cover detailed protocols for a good performing PCEC as well as many lessons and tricks we learned for different electrolyte compositions.

S5: Next-Generation Bioceramics and Biocomposites

Bioceramic Scaffolds

Room: Ballroom 1-2 (South Tower)
Session Chair: Dušan Galusek, IIC SAS

1:30 PM

(ICACC-S5-015-2023) Composite 3D Printing for Bone and Osteochondral Tissue Engineering (Invited)

M. Wang*[1]

1. The University of Hong Kong, Department of Mechanical Engineering, Hong Kong

Bone is a nanocomposite. Many non-porous, polymer-based composites modeled on bone have been investigated for bone tissue repair (M.Wang, Biomaterials, Vol.24(2003), p2133). With the emergence of tissue engineering, the composite approach is adopted for developing porous scaffolds for regenerating bone. For producing composite scaffolds for bone tissue engineering, we have investigated various technologies such as electrospinning (Q.Zhao, et al., Acta Biomaterialia, Vol.123(2021), p312). However, 3D printing is a powerful platform for creating novel tissue engineering scaffolds for regenerating body tissues (J.Lai, et al., Applied Physics Review, Vol.8(2021), 021322). For over 15 years, we have been conducting R&D on composite 3D printing for bone tissue engineering, investigating technologies such as selective laser sintering (B.Duan, et al., Acta Biomaterialia, Vol.6(2010), p4495), extrusion-based 3D printing (C.Wang, et al. Biofabrication, Vol.9(2017), 025031) and digital light processing (Y.Wang, et al. Ceramics International, Vol.48(2022), p27681). Furthermore, 3D printing can construct complex scaffolds for osteochondral tissue engineering (C.Wang, et al., Biofabrication, Vol.12(2020), 025030). This talk will give an overview of our work on composite 3D printing and provide design guidelines and practical approaches in composite 3D printing for bone and osteochondral tissue engineering.

2:00 PM

(ICACC-S5-016-2023) Additive manufacturing of 3D ceramic structures in hydrogel bath using self-setting bioceramic inks for bone tissue engineering

N. Raja*[1]; Y. Choi[1]; H. Park[1]; H. Yun[1]

1. Korea Institute of Materials Science, Republic of Korea

Conventional ceramic scaffolds have to be sintered at high temperature to remove polymeric components and achieve dense structure. However, sintered structures lack sufficient porosity for vascularization. Alternatively, 3D printed scaffolds can have customized pore size and shape with fine control over porosity and other physical properties as per the application. One of the prominently used technique is material extrusion (ME) where the ceramic pastes or inks are laid over in layer by layer arrangement of cylindrical struts. Complex ceramic structures usually contain parts which require balancing, which is provided by non-functional support layer that is usually printed with same material. In addition to wasting material and time, the contact between support and 3D structure can result in defects like cracks and contamination. To avoid this, we extruded the self-setting ceramic inks in solid hydrogel poloxamer bath. The alpha tricalcium phosphate (α-TCP) ceramic ink transforms to calcium deficient hydroxy apatite (CDHA) in the hydrogel bath by hydrolysis-based cement reaction, thereby achieving improved compressive strength and natural bone-like apatite composition. Scaffolds showed good cell attachment on the surface and had porosity (upto 60%) that is required for vascularization with compressive strength suitable for cancellous bone regeneration.

2:20 PM

(ICACC-S5-017-2023) Evaluation of Different Types of PEEK-based Composites for Biomedical Response

S. Javaid[1]; C. Matzke*[1]; A. Thorn[1]; S. Gupta[1]

1. University of North Dakota, Mechanical Engineering, USA

In this study, we will explore Polyetheretherketone (PEEK), a semicrystalline thermoplastic polymer for biomedical application. We will design different types of composites by incoporating additives like MAX/MAB phases, biochar and ceramic additives like Xonotlite. The microstructure, mechanical and thermal properties of these composites will be characterized. An important criteria of the design criteria is to manufacture composites with controlled wettablity. We will also explore the triboactive behavior of the composites. Detailed characterization of the wear tracks will be presented.

2:40 PM

(ICACC-S5-018-2023) Development of osteogenesis enhanced bioink by cell-laden α-TCP/GelMA hydrogel with cement reaction

J. Kim*[1]; H. Park[2]; H. Yun[2]

1. University of Science and Technology (UST), Republic of Korea
2. Korea Institute of Materials Science, Republic of Korea

Bioinks are for three-dimensional bioprinting which designed to mimic tissue characteristics and provide functionality for tissue regeneration. Generally, for the bioinks, hydrogels have been intensively investigated in various tissue engineering field due to their favorable cell anchoring and metabolic activities. However, they are mostly considered for soft tissue engineering because of their poor mechanical strength. For that reason, in this study, we attempted to fabricate a 3D construct suitable for hard tissue regeneration through enhancement of mechanical strength by adding alpha-tricalcium phosphate (α-TCP) to a photo-crosslinkable gelatin methacrylate (GelMA). According to results, α-TCP cement reaction significantly improved the mechanical property of the hydrogels. Furthermore, compare to the hydrogel without α-TCP, the encapsulated cells in the α-TCP/GelMA hydrogel showed identical cell proliferation and enhanced osteogenesis differentiation. After in vitro osteogenic cellular activities of the hydrogel were investigated, the possibility of printing of cell-laden α-TCP/GelMA were studied and as a result it was verified that cell-laden α-TCP/GelMA showed high cell viability after printing and high fidelity of printing. Therefore, we anticipated this composite of hydrogel developed in this study, can potentially be used for bone tissue regeneration.

Bioceramic Coatings, Implants

Room: Ballroom 1-2 (South Tower)
Session Chair: Antonia Ressler, Faculty of Chemical Engineering and Technology

3:20 PM

(ICACC-S5-019-2023) Antipathogen composite coatings via co-sputtering technique for different applications

C. Balagna*[1]; A. Luceri[1]; S. Perero[1]; M. Ferraris[1]

1. Politecnico di Torino, Dept. Applied Science and Technology, Italy

Emerging and re-emerging bug/virus-related diseases represent a serious risk for human wellbeing. Despite all potential restrictive measures and precautions, diseases can develop and spread on a large scale, as happened with the recent COVID-19 pandemic. Antipathogen composite coatings, via co-sputtering technique, were developed and patented. The composite coatings, composed of silver nanoclusters inside glass or ceramic matrix, are deposited on different surfaces. The nanoclusters show a gradual and controlled release of metallic ions without being dispersed into the surrounding environment or in human skin thanks to the matrix. Antipathogen silver effect prevented adhesion and proliferation of several bacterial strains and fungi and developed a significant virucidal activity against several airborne viruses including as respiratory syncytial virus, the influenza virus type A, human coronavirus (OC43) and SARS-COV-2. The technique of co-sputtering is an industrially scalable, versatile and green deposition method which needs only gases and bulk starting materials without requiring or producing dangerous liquid materials.

3:40 PM

(ICACC-S5-020-2023) Antimicrobial glass and ceramic-based coatings for air and water filtration

A. Luceri*[1]; S. Perero[1]; M. Ferraris[1]; C. Balagna[1]

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

Our surrounding environment is characterized by the presence of viruses, bacteria, fungi. Furthermore, the discovery of microorganisms resistant to antibiotics and the widespread viral diseases, such as Corona Virus Disease, focused attention on the importance of reducing microbial colonization and proliferation, to both prevent risks to human wellness and to the integrity of structures and materials. The development of new technologies for satisfying these requirements has a crucial role in the scientific world. Silver nanoparticles have attracted research attention, due to their antimicrobial and antiviral properties. Innovative coatings constituted by silver nanoclusters well embedded in glass or ceramic matrices, deposited by the co-sputtering technique, were developed for air and water filtration systems. Process parameters are optimized to obtain coatings on different substrates, to guarantee excellent performances and antimicrobial effect, without changing mechanical properties. The coatings were tested towards bacteria strains, Staphylococcus Epidermidis and Escherichia Coli, and a fungus, Candida Albicans, whose adhesion and proliferation were slowed down or stopped. The antiviral effect was evaluated towards Rhinovirus, Respiratory Syncytial Virus, and Influenza Virus. In addition, silver ions release was evaluated to demonstrate that the amount is under the threshold considered toxic to humans.

4:00 PM

(ICACC-S5-021-2023) Biomineralization and in vitro biocompatibility of marine-resource-based hydroxyapatite bioceramics with interconnected porous architecture

K. Hadagalli*[1]; A. Panda[2]; S. Mandal[3]; B. Basu[2]; R. Bordia[1]

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2. Indian Institute of Science (IISc), Materials Research Centre, India
3. National Institute of Technology Karnataka, Metallurgical and Materials Engineering, India

Bioceramics should be manufactured using methods capable of tailoring their porosity, with large and perfect interconnections. In this study, we have investigated biomimetic HA from marine sources with tunable porosity. A wet-chemical synthesis approach was used to obtain submicron HA powders from cuttlefish bone. The cuttlefish-bone-derived HA can be sintered conventionally with different pore formers to produce scaffolds with porous architecture that are physiologically relevant. Cuttlefish-bone-derived HA with wheat flour as the pore former, yielded moderate compressive strength (12-15 MPa), an elastic modulus of 1.6 GPa, and open porosity of 35-41% with ~98% interconnected porosity. HA scaffolds with porosity exhibit a functional relationship between their compressive strength and elastic modulus. The specific HA scaffold supports fast nucleation and growth of the biomineralized apatite layer with complete coverage within three days of incubation in simulated body fluid. According to MTT assay and cell morphological analysis, marine resource-based HA porous scaffolds are compatible with osteoblast cells based on their cellular adhesion, proliferation, and phenotypic characteristics. Furthermore, this biogenic HA significantly enhanced the adhesion and proliferation of murine osteoblasts compared with nonbiogenic HA powders.

4:20 PM

(ICACC-S5-022-2023) Novel polyhydroxyalkanoate blends as coatings of β tricalcium phosphate scaffolds

S. Skibinski*[1]; J. Czechowska[1]; E. Cichon[1]; P. Pantak[1]; M. Guzik[2]; P. Szymczak[1]; A. Zima[1]

1. Faculty of Materials Science and Ceramics, AGH University of Science and Technology, Poland
2. Jerzy Haber Institute of Catalysis and Surface Chemistry, Polish Academy of Sciences, Poland

Polymeric coatings can improve the durability of porous β tricalcium phosphate (βTCP) and may serve as platforms for delivering nourishing agents to surrounding tissues. In our study, βTCP scaffolds were covered with poly(3-hydroxybutyrate) and medium chain length PHA blends. The influence of the coating type on the physicochemical properties of scaffolds was investigated using various techniques. XRD, ATR-FTIR, DSC/TG and SEM studies confirmed the presence of βTCP and PHA blends in the composites. PHA coatings significantly changed the character of βTCP surface from hydrophilic to hydrophobic and influenced its roughness, as evidenced by AFM. Degradation studies of the composites revealed that after 6 months of incubation in water, hydroxyacids, and also their oligomers, were detected by UHPLC-MS analysis. Those compounds are known to successfully cure osteoporosis in both *in vitro* and *in vivo* studies, which additionally broadens the range of applications of the obtained bone substitutes. Acknowledgement: Research funded by the National Centre for Research and Development, Poland, grant Techmatstrateg no. TECHMATSTRATEG2/407507/1/NCBR/2019 and partly supported by program, “Excellence initiative—research university” for the AGH University of Science and Technology. SS has been partly supported by the EU Project POWR.03.03.00-IP.08-00-P13/18 - PROM NAWA.

S6: Advanced Materials and Technologies for Rechargeable Energy Storage

Advanced Cathode Materials for Lithium Batteries I

Room: Coquina Salon E (North Tower)
Session Chair: Payam Kaghazchi, Forschungszentrum Juelich

1:30 PM

(ICACC-S6-016-2023) Earth-Abundant Cathode Active Materials: Research and Development Efforts at Argonne National Laboratory (Invited)

J. R. Croy*[1]; A. Gutierrez[1]; J. Chen[2]; A. Vu[1]; E. Lee[1]; B. Shi[3]; J. Gim[4]; M. Balasubramanian[5]; M. Thackeray[1]

1. Argonne National Laboratory, USA
2. Worcester Polytechnic Institute, USA
3. University of Rochester, USA
4. Argonne National Lab, CSE, USA
5. Oak Ridge National Lab, USA

The U.S. DoE has recently released its vision for the future of batteries in the United States, titled The National Blueprint for Lithium Batteries. This document outlines the key areas of investment most critical to enabling a more secure and independent ecosystem around lithium-based batteries for the U.S. and is heavily influenced by the ideas of sustainable technological development. In this regard, earth-abundant cathode active materials are particularly attractive. Ni is considered an earth-abundant element and it plays a critical role in high-performance vehicle applications. This role will likely continue far into the future, especially if recycling can gain traction. However, concerns over Ni demand and the stability of supply chains have already surfaced and the cost-effectiveness of high-Ni cathodes could remain an issue. Mn is substantially more abundant in the earth's crust than Ni or Co and significantly less expensive. In addition, Mn has a rich history and still promising future in energy storage applications. This presentation will explore research and development efforts at Argonne National Laboratory focused on enabling new designs in high Mn, low Ni, Co-free cathodes towards diversifying the portfolio of viable materials for commercial applications.

2:00 PM

(ICACC-S6-017-2023) Exploration of cathode materials for high energy density rechargeable batteries (Invited)

K. Kawai*[1]; A. Yamada[2]

1. Waseda University, Japan
2. The University of Tokyo, Japan

Efficient electrochemical energy storage devices serve as a key technology in a sustainable society. However, the energy density of state-of-the-art lithium-ion batteries has not reached a level enough to meet industry demands due to the limited performance of cathode materials. The development of large-capacity or high-voltage cathode materials has been a crucial task although their use in commercial rechargeable batteries remains a challenge regarding cycle life, energy efficiency, and cost. Here, we show two classes of cathode materials for high energy density rechargeable batteries: high-voltage Cr-based phosphates and large-capacity anionic redox layered oxides.

2:30 PM

(ICACC-S6-018-2023) Li-rich layered oxides for lithium cells: Pushing positive electrodes to their limits (Invited)

S. Brutti*[1]

1. Università di Roma La Sapienza, Chemistry, Italy

In the last decades industrial and academic research have been focusing on lithium-ion batteries (LIBs). Generally speaking, LIBs are more expensive than other battery chemistries, but they provide the highest power and energy densities. LIBs require further development in terms of safety and performance to establish also in the automotive market and new materials at both electrode sides as well as at the electrolyte side are necessary to overcome the state-of-the-art and the commercial benchmarks. Over-stoichiometric Li-rich layered oxides (LRLO) are a family of positive electrode materials with promizing theoretical performance. The eco-friendly Co-free LRLOs have attracted a lot of attentions, thanks to the improved sustainability, reduced costs and outstanding performance (250 mAh g^{-1}). The crystal structure and cation ordering of LRLO are a matter of controversy: overall, extensive defectivities play a key role in the breakdown of the C2/m lattice symmetry that degrades in the R-3m one. This structural ambiguity is at the origin of the capacity fading in batteries originating from undesired lattice transformations. Here we demonstrate the resilience of the LRLO lattice despite large variations in the transition metals blend and over-lithiation extension, and how the electrochemical performance in batteries are improved by a careful tuning of doping in parallel with the removal of cobalt.

Advanced Cathode Materials for Lithium Batteries II

Room: Coquina Salon E (North Tower)
Session Chairs: Jason Croy, Argonne National Laboratory; Kosuke Kawai, Waseda University

3:20 PM

(ICACC-S6-020-2023) Impact of Doping on Stability of Cathode Materials for Li-based Batteries: A Multiscale Modeling Study (Invited)

P. Kaghazchi*[1]

1. Forschungszentrum Juelich, Germany

By combining density functional theory calculation, thermodynamics consideration, and finite element simulation, impact of doping and substitution on the stability of (potentially) high-capacity Ni-rich layered-oxide cathodes is studied. Simulation shows that a small amount of doping cannot affect bulk properties of materials. However, surface and grain boundary structure can be influenced strongly. For example, B doping leads to the formation of a coating layer on the surface of NCM811 particles. As such, B doping can improve the electrochemical stability of NCM811 by protecting its surface from chemical interaction with electrolyte during charge/discharge. Moreover, it is shown that B can tailor the microstructure of Ni-rich cathodes and thereby enhance their mechanical stability. Mechanism of the B-induced surface oxide formation and its effect on the integrity of the microstructure are discussed. Moreover, one example of a dopant that affects the structure and stability of grain boundaries is presented. Finally, impact of substitution on the stability of layered-oxide cathodes in all-solid-state batteries is discussed.

3:50 PM

(ICACC-S6-021-2023) Structure and Doping of Lithium Rich Layered Oxides as a cathode material for Lithium Ion Batteries

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2. ENEA C.R., Tecnologie Energetiche e Fonti Rinnovabili, Italy

Lithium-Rich Layered Oxides (LRLOs) are opening new frontiers for high-capacity positive electrodes for Lithium-Ion Batteries to meet the challenges of green, sustainable and safe transportation. LRLOs show very high specific capacity that originates from cumulative cationic and anionic redox processes. The cationic redox reactions exploit the oxidation/reduction of the transition metals in the cationic layers. On the other hand, the anionic redox reactions affect the oxygen anionic sub-lattice. In this last process the lithium extraction occurs in parallel with the activation of the reversible O^{2-}/O^{-} redox couple and, unfortunately, with the irreversible O_2 release. Despite the outstanding reversible capacities beyond 220 $mAhg^{-1}$ for hundreds of cycles, many functional features in batteries are still inadequate and hinder their use in a commercial Li-ion device. Here, we analyse the complex structure of LRLOs by exploiting different scattering techniques and different structural models also considering defects. Furthermore, we demonstrate that, by tailoring the transition metal substitution and doping, it is possible to improve the electrochemical behavior and mitigate the structural rearrangements. In this respect we show an optimized layered materials with outstanding electrochemical performance, improved environmental benignity and reduced manufacturing costs.

4:10 PM

(ICACC-S6-022-2023) Design of interfacial thermodynamics to increase stability of Li-based cathodes

R. Castro*[1]

1. University of California, Davis, Material Science & Engineering, USA

Crystal size refinement down to the nanoscale results in unique properties and phenomena in oxide-based materials. However, after decades of nanotechnology, the limited fundamental understanding of the origins of those nano-induced features still refrains a comprehensive design and optimization of devices. In those systems, a significant fraction of the atomic volume belongs to the interfacial regions, a complex chemical environment that governs the system interactions in both positive and negative ways. While interfaces are responsible for phenomena such as the above-mentioned improved ion exchange rate in nano-cathodes, the intrinsic interface excess energies trigger coarsening even at moderate temperatures, limiting service time. This talk discusses how interfacial thermodynamics and its relationships with nanoscale properties offer an effective strategy to understand and design cathode oxides with increased stability. We present recent experimental and atomistic simulation results on the stability of nano-LiMn2O4 and nano-LiCoO2 and their dependence on interface thermodynamics.

S8: 17th International Symposium on Advanced Processing and Manufacturing Technologies for Structural and Multifunctional Materials and Systems (APMT17)

Advanced Composite Manufacturing Technologies, Hybrid Processes II

Room: Coquina Salon F (North Tower)
Session Chair: Csaba Balazsi, ELKH Centre for Energy Research

1:30 PM

(ICACC-S8-014-2023) Catalytic Combustion-type Carbon Monoxide Gas Sensor Compose of Oxide Ion Conducting Solid (Invited)

N. Imanaka*[1]

1. Osaka University, Applied Chemistry, Japan

Carbon monoxide (CO) is well-known highly toxic gas causing a serious health hazard if inhaled, even at low level. One of the serious disadvantages in the case for conventional catalytic combustion-type CO gas sensors (Pt loaded or Pd loaded Al_2O_3) is that they need relatively elevated operating temperatures over 400 degree C for the complete CO oxidation. Since other gases such as methane and volatile organic compounds (VOCs) also burn out at such temperatures, the sensors essentially lacks in selectivity. Recently, we have succeeded in realizing new type of catalytic combustion-type sensor showing appreciably low-temperature operable feature by using 10 wt% Pt loaded $Ce_{0.68}Zr_{0.17}Sn_{0.15}O_{2.0}$ solid composite as the catalyst. They sucessfully demonstrated its operation at as low as 70 degree C, which is more than 300 degree C below compared with conventional ones. Furthermore, by the combination of the superior thermoelectric material of aluminum nitride (AlN)) ceramics powder as an intermediate heat transfer layer combined with the Pt loaded $Ce_{0.68}Zr_{0.17}Sn_{0.15}O_{2.0}$ composite, a novel catalytic combustion-type CO gas sensor was sucessfully achieved, with an excellent sensing performance that drastically accelerated the sensing response at the operating temperature. Some further results will be adderessed in the presentation.

2:00 PM

(ICACC-S8-015-2023) Mechanical properties of potassium and metakaolin based geopolymer under electron irradiation

Y. Yang*[1]; H. Suematsu[2]; K. Niihara[2]; T. Nakayama[2]; T. Le[3]; T. Do[4]

1. Nagaoka University of Technology, Energy and Environment Science, Japan
2. Extreme Energy-Density Reserch Institute, Japan
3. Danang University of Science and Technology, Viet Nam
4. Nagaoka University of Technology, Nuclear System Safety Engineering, Japan

Geopolymers are inorganic polymers composed of AlO_4 and SiO_4 tetrahedral structural units and a three-dimensional network structure. In the decommissioning of JMTR, the geopolymer compaction method for radioactive Al wastes has been considered. For this purpose, the stability of geopolymer under irradiation is important. In this study, leaching and mechanical property changes after electron irradiation were measured. Geopolymer samples were made of EFACO silica, metakaolin powder, potassium hydroxide and potassium silicate solution with molar ratios of Al: Si: K: H_2O =1: 2.1: 0.8: (7, 8, 9,10). 6 Samples were synthesized and divided in to 3 groups, which were cured at RT, 40°C and 60°C for 1 day and then kept at RT. Some samples were irradiated by ETIGO-III at a peak voltage of 2MeV, a current of 5kA and a pulse width of 100 ns for one to four shots. In this research, the potassium and metakaolin-based geopolymer samples with different water contents have been

synthesized. A Vickers indenter was loaded at 1 kgf to measure the hardness. At the same temperature, the Vickers hardness decreases with the increase of water ratio. Under the same molar ratio of water, the Vickers hardness decreases with the increase of temperature. Relative weight change results showed that the higher the water content, the greater the relative weight reduction, but the difference was small.

2:20 PM

(ICACC-S8-016-2023) Novel Wet Electrospinning Inside a Reactive Pre-ceramic Gel to Yield Advanced Nanofiber-Reinforced Geopolymer Composites

Y. Xu*[1]; A. Akono[1]

1. Northwestern University, Civil and Environmental Engineering, USA

Recently, wet electrospinning has been introduced as an efficient way to deposit isolated nanofibers in bulk materials. In wet electrospinning, a liquid bath is adopted, instead of a solid collector, for fiber collection. However, despite several studies focused on wet electrospinning to yield polymer composites, few studies have investigated wet electrospinning to yield ceramic composites. In this paper, we propose a novel in-situ fabrication approach for nanofiber-reinforced ceramic composites based on an enhanced wet electrospinning method. Our method uses electrospinning to draw polymer nanofibers into a reactive pre-ceramic gel, which is later activated to yield nanofiber-reinforced ceramic composites. We demonstrate our method by studying wet electrospun Polyacrylonitrile and Poly(ethylene oxide) fiber-reinforced geopolymer composites, with fiber weight ratio in the range of 0.1–1.0 wt%. Wet electrospinning preserves the amorphous structure of geopolymer while changing the molecular arrangement, and leads to an increase in both mesopores fraction and overall porosity of geopolymer composites. A clear stiffening and toughening effect was observed for Poly(ethylene oxide)-reinforced geopolymer composites. This work demonstrates the viability of wet electrospinning to fabricate multifunctional nanofiber-reinforced composites.

Joining, Integration, Machining, Repair, and Refurbishment Technologies

Room: Coquina Salon F (North Tower)
Session Chairs: Hisayuki Suematsu, Nagaoka University of Technology; Chang-Jun Bae, Korea Institute of Materials Science

3:00 PM

(ICACC-S8-017-2023) Joining and integration technologies for ceramic- and CMC-based components (Invited)

M. Ferraris*[1]

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

Innovation in processing and characterization of ceramic- and CMC-based joined components developed at GLANCE-Glasses, Ceramics and Composites research group at Politecnico di Torino, Italy (www.composites.polito.it) will be presented and discussed. Joined components for energy storage and transformation have been designed, fabricated and characterized in simulated working conditions: recent results will be briefly reviewed: in particular, results on silicon nitride and ceramic membranes joined to metals will be briefly reviewed, together with issues related to the interfacial reactivity and coefficient of thermal expansion mismatch. The combination of advanced design of interfaces and joining materials/technologies, selective matrix removal from the composite surface, laser structuring and mechanical machining of the composite/metal surfaces will be discussed and compared to existing solutions. The work done with the aim of developing reliable and user-friendly international standard test to measure the shear strength of joined components will also be reviewed. Finally, J-TECH@POLITO, Advanced Joining Technology research center at Politecnico di Torino, will be described together with collaboration actions and opportunities for common research activity on joining.

3:30 PM

(ICACC-S8-018-2023) Surface strengthening of ceramics by high-temperature laser shock peening

F. Wang[1]; X. Chen[1]; D. P. DeLellis[2]; A. Krause[2]; Y. Lu[1]; B. Cui*[1]

1. University of Nebraska–Lincoln, USA
2. Carnegie Mellon University, Materials Science and Engineering, USA

A novel process of high-temperature laser shock peening (HTLSP) has been developed for surface strengthening of ceramic materials such as sapphire and SiC. HTLSP at 1200°C can induce a high compressive residual stress on the surface of sapphire while minimize the damage of laser-driven shock waves. Transmission electron microscopy characterizations revealed high dislocation densities near the surface, suggesting that plastic deformation at an ultra-high strain rate was generated by the high shock wave pressure. The HTLSP-induced compressive residual stress can significantly improve the hardness and fracture toughness of sapphire and SiC. The fundamental mechanisms related to the interactions between laser-driven shock waves and ceramics are discussed.

3:50 PM

(ICACC-S8-019-2023) Joining of SiC components by pressureless reaction bonding using SiC loaded suspensions

R. Orta Guerra*[1]; O. Brandt[1]; R. Trice[1]; J. P. Youngblood[1]

1. Purdue University, Department of Materials Engineering, USA

Current demand of SiC ceramic components with complex shapes has pushed the need for reliable joining techniques that can survive high-temperature applications. However, the joining of SiC is challenging because of its strong covalent bonding and corresponding low self-diffusivity. Different methods for bonding SiC have been investigated previously with some of them requiring extensive surface preparation and external pressure application during the thermal bonding cycle. The aim of this work is to present results where SiC has been pressurelessly joined to SiC by reaction bonding using SiC and carbon black loaded suspensions. The approach studied varied the thickness of the applied bonding layer between SiC components and tested its mechanical strength, quantify the crystalline structure, and examine the presence of defects in bonding region. The integrity of the bonding region is also studied after being exposed to a high temperature environment of 1300°C in air.

Novel Forming/Sintering Technologies, Near-net Shaping

Room: Coquina Salon F (North Tower)
Session Chair: Monica Ferraris, Politecnico di Torino

4:30 PM

(ICACC-S8-020-2023) Understanding the role of transient liquid phases on the densification of cold sintered ZnO

A. Jabr*[1]; J. Fanghanel[2]; Z. Fan[2]; R. Bermejo[3]; C. Randall[4]

1. Montan Universitaet Leoben, Department Materials Science, Austria
2. Materials research institute, Materials Science and Engineering Department, USA
3. Montanuniversitaet Leoben, Institut fuer Struktur- und Funktionskeramik, Austria
4. Penn State University, Materials Science and Engineering, USA

Among the different alternative sintering techniques that aim at lowering the sintering temperature, the cold sintering process (CSP) enables densification of ceramics at unmatched low temperatures below 300°C. The process is enabled by a chemomechanical effect which is activated by a compatible transient liquid phase. This study aims at investigating the effect of the liquid phase on densification, transient chemical changes, microstructural evolution and mechanical strength of cold sintered ZnO. A comparative study was conducted using various organic acids (formic, acetic and citric acid). To assess chemical interactions during cold sintering, SEM, TEM, FTIR and TGA analyses were performed. Almost full densification (> 96%) was achieved for samples sintered using formic and acetic acid, in contrast to those sintered with citric acid (< 80%). The lack of densification in the latter is ascribed to the high stability of the formed complexes which inhibit their back transformation to ZnO, highlighting the importance of the precipitation step during CSP. A remarkable impact of the liquid phase chemistry on the strength of cold sintered samples was found, where samples sintered with formic acid exhibit twice the strength of those sintered with acetic acid. The relatively high strength achieved by formic acid is about 40% higher than the previously reported value in the literature.

4:50 PM

(ICACC-S8-021-2023) Influence of the monomer on the sintering of the gelcasted ceramics

L. Gauzere*[1]; C. Besnard[2]; S. Couillaud[2]; J. Léon[2]; J. Heintz[3]

1. ICMCB-CNRS, France
2. Galtenco Solutions, France
3. ENSCBP-Bordeaux INP, ICMCB, France

Within the framework of my PhD, related to the manufacture of alumina ceramic parts of complex shape and/or large size, I study gelcasting, which is a net-shape forming process. It deals with the preparation of a homogeneous suspension, composed of a solvent (usually water), a dispersant, a plasticizer, a monomer, a cross-linking agent and a ceramic powder. The advantage of this process is that the ceramic particles are "frozen" into the desired shape through in-situ polymerization. The monomer initially used in the development of gelcasting was acrylamide (AM). However, this compound is toxic and other monomers have then been considered. I will focus my presentation on the influence of the nature of the monomer on the sintering of alumina samples prepared in this way. I will present the rheological behavior of the slurries as well as the thermal evolution of the samples (TGA, dilatometry, SEM) according to the chemical nature of the monomer (AM, methyl-acrylamide or dimethyl-acrylamide). These results have clearly shown an important influence of the monomer on the final relative density of the sintered pellets (from 88% to 98%) prepared in the same conditions. These different sintering behaviors were analysed in terms of green pellet homogeneity. X-ray tomography analyses will be presented to support this explanation.

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

High Temperature Ceramics for Space Reactor and Advanced Reactor Applications

Room: Ballroom 4 (South Tower)
Session Chair: Christian Deck, General Atomics

1:30 PM

(ICACC-S13-016-2023) Raman spectroscopy imaging of uranium nitride microspheres

E. Lopez Honorato*[1]; R. Seibert[1]; J. M. Kurley[1]; A. Nelson[1]

1. Oak Ridge National Lab, USA

Raman spectroscopy has gained considerable interest in the characterization of uranium-bearing compounds due to its ability to identify small variations in composition, phases, and defects. However, limited data exists on the application of Raman spectroscopy on uranium nitride. In this work, Raman spectroscopy was employed on sol-gel synthesized UN microspheres. It was observed that although group theory suggests that UN is Raman non-active, it has defect-induced first and second-order scattering modes that can be used for its characterization. Raman spectroscopy was used to generate images containing information on chemical composition and phases present in UN microspheres. Through Raman spectroscopy imaging it was possible to identify variations in carbon concentration, the core-shell structure produced during the carbothermic reduction, and the transition from UC1-xNx to UN.

1:50 PM

(ICACC-S13-017-2023) Chemical vapor deposition of ZrC for space nuclear propulsion

E. Lopez Honorato*[1]; R. Heldt[1]; T. J. Gerczak[1]; P. Doyle[1]; W. Cureton[1]; G. Helmreich[1]

1. Oak Ridge National Lab, USA

ZrC is a candidate material for the production of advanced protective coatings for nuclear propulsion fuel elements. In this work, we described the impact of deposition conditions on the microstructure and composition of ZrC deposited on the inner cooling channels of the fuel elements. ZrC coatings were characterized by SEM/EDS, EBSD, Raman spectroscopy, XRD, and X-ray computer tomography. ZrC was deposited with the use of $ZrCl_4$ and methane from atmospheric pressure down to 100 Torr. It was observed that although the composition could be controlled by temperature, $ZrCl_4/CH_4$ ratio, and hydrogen concentration, the uniformity, and quality of the coatings were strongly affected by residence time, pressure, and surface area/volume ratio.

2:10 PM

(ICACC-S13-018-2023) Development of Zirconium Carbide - Uranium Mononitride Ceramic Composites by Spark Plasma Sintering Technique for Space Reactor Applications

N. D. Jerred*[1]; R. D. Scott[1]; S. R. Hamilton[1]; J. M. Zillinger[1]; R. C. O'Brien[2]; C. A. Fife[4]; D. E. Burns[3]

1. Idaho National Laboratory, Advanced Fuels Fabrication, USA
2. Idaho National Laboratory, Special Reactor Concepts, USA
3. Idaho National Laboratory, Space Nuclear Power & Isotope Systems, USA
4. Idaho National Laboratory, Irradiation Testing, USA

The zirconium carbide (ZrC) – uranium mononitride (UN) ceramic composite is of interest as a fuel form for the high temperature hydrogen environments of nuclear thermal propulsion (NTP) systems. The ZrC provides an inert structural matrix to house a dispersion of UN fuel kernels. To fabricate the composite, the spark plasma sintering (SPS) fabrication technique is employed. The sintering process was observed to lead to phase interactions between the matrix and fuel components. The resulting ZrC-UN composition has been analyzed using scanning electron microscopy (SEM) and X-ray diffraction (XRD) techniques and the resulting composition is compared against the fabrication parameters employed, such as sintering temperature, time and UN concentration is investigated. Additionally, the manufacture of a ZrC-UN composite fuel compact for an irradiation experiment in the Transient Reactor Test Facility (TREAT) at Idaho National Laboratory (INL) is discussed.

2:30 PM

(ICACC-S13-019-2023) Large Scale Additive Manufacturing of Silicon Carbide

K. Terrani*[1]; M. Trammell[1]; B. Jolly[1]; G. Garside[1]

1. Ultra Safe Nuclear, USA

Silicon carbide (SiC) additive manufacturing is now possible through a number of techniques. One such technique that yields high purity and fully crystalline SiC, is the combination of binderjet additive manufacturing and chemical vapor infiltration (CVI). This methodology provides ample flexibility in part geometry without any major limitations in manufacturing parts with unsupported overhangs. Most importantly, this methodology stays true to the original computer assisted drawing (CAD) input and does not involve any shrinkage or other size distortion. Ultra Safe Nuclear Corporation has invested in sizable SiC additive manufacturing capabilities via this technology at two locations in the United States. This paper provides an overview of the technology, the large-scale manufacturing capabilities, and the art of the possible with this process.

New Materials and Containment for Neutron Moderators, Reflectors, and Shielding

Room: Ballroom 4 (South Tower)
Session Chair: Hirokazu Katsui, National Institute of Advanced Industrial Science and Technology (AIST)

3:20 PM

(ICACC-S13-020-2023) Advanced Hydrides for Terrestrial and Space Fission Reactors (Invited)

C. Taylor*[1]; T. Nizolek[2]; A. P. Shivprasad[1]; E. Luther[2]; T. Saleh[1]

1. Los Alamos National Lab, Materials Science and Technology Division, USA
2. Los Alamos National Lab, Sigma Division, USA

Moderators are often employed in nuclear reactors to permit higher fuel efficiency and lower U-235 enrichment. For a given level of U-235 enrichment, reducing the quantity of fuel is particularly important for space applications where minimizing reactor size is advantageous, but also for terrestrial microreactor applications. Moderators often consist of hydrogenous materials with a high cross-section for elastic interaction with neutrons. Neutrons slow down into the thermal energy regime upon interaction with the moderator, increasing the probability for fission. Metal hydrides often contain higher hydrogen densities than liquid hydrogen and remain stable to relatively high temperatures, making them ideal candidates for moderator applications. The hydriding reaction, however, causes significant volume expansion (often on the order of 10-20%), which leads to cracking if not carefully controlled. This high propensity for cracking makes fabricating large metal hydride components particularly challenging. This talk will overview recent advances in fabricating metal hydrides and current knowledge of thermophysical and mechanical properties. Metal hydride stability under irradiation will also be discussed. The talk will focus on the two most promising candidates for space and fission applications: zirconium hydride and yttrium hydride.

3:50 PM

(ICACC-S13-021-2023) Solid ceramic breeder materials for fusion and metal hydrides for microreactors (Invited)

C. N. Taylor*[1]; T. F. Fuerst[1]; M. N. Cinbiz[1]

1. Idaho National Laboratory, USA

Ceramics and ceramic-like materials play a pivotal role in both fusion and fission systems as functional materials. In fusion systems, lithium-containing solid ceramics, such as Li_2TiO_3, $LiAlO_2$, $LiSiO_4$, and Li_2ZrO_3, are used to breed tritium through transmutation of lithium where it is harvested and supplied to the fusion fuel cycle. In fission systems, metal hydrides possessing some ceramic-like properties, such as YH_2 or ZrH_2, will be used as neutron moderators. These high temperature moderator materials retain large quantities of hydrogen up to high temperatures, thus enabling the development of compact, high temperature microreactors. For these two material systems, hydrogen's behavior has a crucial effect on the respective fusion or fission reactor design. Solid ceramic breeders and metal hydrides must withstand the harsh fusion and fission nuclear reactor environments, respectively. Legacy irradiation experiments have been performed on each of these classes of materials decades ago. A wide number of potential solid breeders underwent neutron irradiation in the 1980's and those data are presently accessible. Yttrium hydride was irradiated in the 1960's and only anecdotal summaries are available. However, recent irradiations of yttrium hydride provide additional performance data. This study reviews the currently available irradiation experiments and performance of these two material types.

4:20 PM

(ICACC-S13-022-2023) Surface Modification Strategies for Hydrogen Retention in Hydride Moderators

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1. Texas A&M University, Nuclear Engineering, USA
2. Stony Brook University, Center for Thermal Spray Research, USA
3. Los Alamos National Laboratory, USA
4. University of Michigan, Nuclear Engineering & Radiological Sciences, USA

Hydride moderators can reduce the size and improve the power density of advanced microreactors and nuclear thermal propulsion systems, but rapid hydrogen loss at temperatures above 500 °C remains a challenge. Augmentation of the hydride surface with a ceramic hydrogen barrier can slow hydrogen loss. To rapidly identify optimal methods, two strategies for surface modification were pursued in parallel: direct ceramic application with thermal spray and reactive ceramic growth. Coupons of ZrH and YH were sprayed with yttria-stabilized zirconia (YSZ). Meanwhile, similar coupons of ZrH and YH were oxidized in air and pure O_2. Both sets of samples were examined with optical and electron microscopy, as well as x-ray diffraction (XRD). This talk will present the initial results of both approaches and discuss their implications for future development. Historical context for the hydrogen loss problem and brief discussion of alternate approaches will also be given.

4:40 PM

(ICACC-S13-023-2023) Development of Ceramic Matrix Entrained Metal Hydride Shield Composites

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1. Stony Brook University, Materials Science and Chemical Engineering, USA

High temperature superconductor coils in compact fusion reactor require high performance shielding materials to protect them from combined neutron damage and deposited heat from radiation. Here, we propose a two-phase composite shielding design with MgO ceramic as the matrix and metal hydride as the entrain phase. The metal hydride provides excellent moderation and absorption of neutrons while dense, irradiation stable MgO ensures a full encapsulation of metal hydrides to prevent hydrogen loss during high temperature operation. The co-sintering the MgO with temperature sensitive metal hydrides requires substantial reduction of sintering temperature. In this work, we present a strategy in lowing the sintering temperature of MgO by using a bimodal size distribution of MgO feedstocks and a nano LiF sintering additives. The significant reduction of MgO sintering temperature (by ~600°C) enables the low temperature co-sintering of MgO with HfH_2 particles. High density is achieved for MgO-HfH_2 composite shielding above 1000°C. Microstructure characterization demonstrates a full dense MgO matrix with excellent entrainment of HfH_2 particles. XRD and hydride analysis suggest a high retention of hydrogen because of low temperature densification of MgO matrix. This paper will review the processing and thermophysical properties of this class of composite material as well as describing its performance as fusion shield.

S15: 7th International Symposium on Additive Manufacturing and 3-D Printing Technologies

Vat Photopolymerization / Substrate Stereolithography II

Room: Coquina Salon H (North Tower)
Session Chair: Majid Minary, University of Texas at Dallas

1:30 PM

(ICACC-S15-010-2023) High resolution 3D printing and injection molding of transparent fused silica glass (Invited)

F. Kotz-Helmer*[1]

1. Glassomer GmbH, Germany

Fused silica glass is an important material due to its high chemical and thermal stability its outstanding optical transparency and hardness. Due to these properties, fused silica glass is an interesting material for future applications in chemical synthesis or optics and photonics. However structuring of glasses is difficult, especially when high-resolutions are needed structuring is usually done using wet chemical or dry etching using hazardous chemicals. Glassomer has developed a novel way to shape transparent fused silica glass. Liquid photocurable nanocomposites can be shaped using polymeric 3D printing technologies like stereolithography, 2-photonpolymerisation or volumetric printing. Solid thermoplastic Glassomer nanocomposites can be shaped using high throughput methods like injection molding, embossing or roll-to-roll replication. After the shaping process the polymeric Glassomer is converted using debinding and sintering yielding high-quality fused silica glass with high optical transparency (transmission > 90 %) and optical surface quality (Rq < 2 nm). 3D printing and injection molding of complex shaped fused silica glass components will enable a plethora of applications from optics and photonics to medical devices or lab-on-a-chip devices.

2:00 PM

(ICACC-S15-011-2023) Nanocomposites Obtained with Vat Photopolymerization Techniques

L. Gil*[1]; H. A. Colorado L.[1]

1. Universidad de Antioquia, Colombia

This research presents the additive manufacturing of nanocomposites using an LCD 3D desk printer VAT-photopolymerization technology. As reinforcement, copper nanoparticles (CuNP's) were added in 0, 0.5, and 1.0wt% with respect to the resin. Different curing times were used, and samples were tested in tensile tests. The microstructure was investigated using scanning electron microscopy.

2:20 PM

(ICACC-S15-012-2023) Ceramic Stereolithography for Sustainable Manufacturing

F. Spirrett*[1]; S. Kirihara[1]

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

As sustainable manufacturing becomes a high priority in society; industry and academia must adapt to meet new expectations for responsible manufacturing. Additive manufacturing supports sustainability through enabling the use of smart materials, reducing resource requirements, and supporting manufacture on demand. A key benefit of AM is the geometric freedom that is enabled by layer-wise fabrication of 3D parts from digital data. For ceramic materials, complex geometries can be difficult and expensive to form and shape by conventional methods. Ceramic stereolithography (SLA) offers new opportunities for problem solving in industrial manufacturing of technical components. Computational methods such as Computer Fluid Dynamics, the Discrete Element Method, and Ray Tracing analysis were used to reduce the number of experimental iterations required for ceramic SLA process optimisation, reducing the time and resource cost of this step. In this research, photosensitive ceramic pastes were made by dispersing solid ceramic particles in a photosensitive resin. Alumina, Yttria Stabilised Zirconia, and Alumina-Glass composite parts were fabricated by SLA to demonstrate the potential to improve sustainability in manufacturing for various applications, such as YSZ electrodes for aluminium smelting, complex alumina thermoacoustic stacks, and composite alumina-glass for structural applications.

2:40 PM

(ICACC-S15-013-2023) Stereolithographic Additive Manufacturing of Geometrically Modulated Structures

S. Kirihara*[1]; F. Spirrett[1]

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

Stereolithography have been modified as smart processing to create fine ceramic components. In additive manufacturing of lamination technologies can fabricate ceramic solid components according to topological geometry. Two dimensional cross sectional patterns were created through photo polymerization by ultra violet laser drawing on spread resin paste including ceramic nanoparticles, and three dimensional composite models were sterically printed by layer lamination though chemical bonding. As row material of the 3D printing, nanometer sized ceramic particles were dispersed in to photo sensitive liquid resins 50 % in volume fraction. The resin paste was spread on a glass substrate at 50 μm in layer thickness by a mechanically moved knife edge. An ultraviolet laser beam of 355 nm in wavelength was adjusted from 10 to 300 μm in variable diameter and scanned on the pasted resin surface. Irradiation power was changed automatically from 10 to 200 mW for enough solidification depth for 2D layer bonding. The created 3D composite precursor was dewaxed and sintered in the vacuum and air atmosphere to obtain full metal and ceramic components. Through the computer aided smart manufacture, design and evaluation, geometrically modulated periodic and self-similar patterns with graded and fluctuated structures were processed. Their geometric structures were modulated form integer to non-integer dimensions.

Vat Photopolymerization / Substrate Stereolithography III

Room: Coquina Salon H (North Tower)
Session Chair: Dileep Singh, Argonne National Lab

3:20 PM

(ICACC-S15-014-2023) 3D printing alumina using DLP process

M. Minary*[1]

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Direct Light Processing (DLP) is a highly sought-after additive manufacturing method to produce complex and dense ceramic parts. Alumina is one of the most studied ceramic materials incorporated in DLP photosensitive resins, due to its superior hardness and wear resistance. Most studies employ the use of a unitary spherical particle shape but multiple particle sizes to improve the density, which does little to improve other mechanical properties. This article, we demonstrate the fabrication of highly dense alumina ceramics parts incorporating multiple particle shapes facilitated using a low-cost Digital Light Processing (DLP) printer. The high solid loading alumina photosensitive resin developed uses a 1:1 ratio by weight of both platelets and nanoparticles. Thermal post processing was optimized for burnout and sintering, resulting in highly dense ceramic parts. The optimized DLP printing process shows that this scalable additive manufacturing method has the potential to produce low-cost alumina ceramics with improved mechanical properties.

3:40 PM

(ICACC-S15-015-2023) Increasing Wall Thickness of Additively Manufactured Alumina Parts Through Improving the Burnout Schedule

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3. Oak Ridge National Lab, Chemical Sciences Division, USA

The ceramic additive manufacturing (AM) field is growing as the desire for near-net and complex shaped parts are increasing in a variety of industries, such as in the aerospace and heat exchanger industry. One of the methods for ceramic AM is digital light processing (DLP), where a projector is used to cure thin layers of photocurable resins filled with ceramic particulates onto a build plate. This is repeated layer-by-layer until the part is fully printed. One of the challenges of DLP printing is cracking that occurs during the post-processing step of binder burnout, due to the large amount of photopolymers (~40 vol.%+) that need to be burned off before densification. This limits part thickness to a maximum of roughly 10 mm in any direction. Here, we present a thorough investigation of the polymer decomposition stages in order to develop an improved binder burnout schedule for alumina parts printed via DLP. In a study of printing varied geometries with incremental dimensional changes, we report a nearly 100% increase in wall thickness with the improved burnout schedule compared to the manufacturer supplied procedure.

4:00 PM

(ICACC-S15-016-2023) Lithography-based additive manufacturing of piezoceramics and highly dielectric ceramics

M. Schwentenwein*[1]; C. Bae[2]; A. A. Altun[1]; D. Brouczek[1]; C. Hofstetter[1]; S. M. Allan[3]

1. Lithoz GmbH, Austria
2. Korea Institute of Materials Science, Department of 3D printing materials, Republic of Korea
3. Lithoz America, LLC, USA

Lithography-based additive manufacturing (AM) technologies are photopolymerization-based AM technologies and create objects by spatially controlled solidification of a liquid resin by exposure to light. The liquid resin is a mixture of monomers together with a photoinitiator; in the case of lithography-based ceramic manufacturing (LCM) ceramic particles are added at a high solids loading of >40 vol% into the mixture to give a photocurable ceramic suspension. The solidification mechanism - in the case of LCM a radical polymerization mechanism - is also triggered by exposing the slurry to light. For dielectric material systems, different ceramic materials were successfully printed, debinded, and sintered. Characterization of the resulting materials showed that the 3D printed parts exhibited compatible basic electrical properties to the properties of tape cast parts (permittivity, Q factor). This behavior could be demonstrated for different materials with dielectric constant ranging from 10 to 60. For piezoceramic materials, 2 different grades were investigated within the LCM process: classical lead-zirconate-titanate (PZT, soft-doped) and lead-free potassium-sodium-niobate (KNN). Again, it was possible to show, that the resulting 3D printed components had properties at eye-level to their conventionally manufactured analogues with measured d33 values being at 650 and 100pC/N, respectively.

4:20 PM

(ICACC-S15-017-2023) DLP-based additive manufacturing technology for 3D transparent yttria construction

S. Zhang*[1]; C. Gal[2]; Y. Choi[2]; H. Kim[2]; Y. Park[2]; H. Yun[2]

1. University of Science and Technology, Republic of Korea
2. Korea Institute of Materials Science, Republic of Korea

Yttria (Y_2O_3) is a promising transparent ceramic with exceptional transparency, high fracture toughness, hardness, and a high dielectric property making it suitable for various applications such as missile domes, scintillators, and optoelectronic devices. High-quality transparent Y_2O_3 can be produced using a variety of shaping techniques, including dry pressing, and gel casting. However, the geometric control limits of shaping processes would hinder the utilization of Y_2O_3. We herein propose additive manufacturing (AM) as one of the best solutions to solve this structural flaw. In this study, 3D-structured transparent Y_2O_3 ceramic was achieved by DLP-based AM technology assisted by vacuum sintering, and the critical factors governing the transparency were revealed. Detailed slurry conditions were optimized to control the printability of the slurry as well as the homogeneity and density of the green body, which facilitates the densification of transparent ceramics. Zirconia and lanthana were used to control the growth of grains as sintering additives. The sintered transparent Y_2O_3 ceramics reached a relative density of 99.9% with high in-line transmittance by achieving a fine-grained dense microstructure. This study demonstrates that the overcoming of the geometric control limitation of Y_2O_3 by ceramic AM offers excellent promise for expanding the utilization of Y_2O_3 in a variety of applications.

S16: Geopolymers, Inorganic Polymers and Sustainable Construction Materials

Geopolymers made from or with Biological Materials

Room: Coquina Salon C (North Tower)
Session Chair: Cengiz Bagci, Hitit University

1:30 PM

(ICACC-S16-008-2023) Valorization of a fruit waste (Musa paradisiaca) for producing kaolinic and-illitic-based ceramics (Invited)

R. Serewane-Deramne[2]; G. Lecomte-Nana*[1]; G. Tchangbedji[3]; C. Peyratout[1]

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2. Université de Bangui, Central African Republic
3. Université de Lomé, Togo

The proposed research work is in line with the valorization of the peel of a tropical plant fruit regarding the manufacture of low cost silicate ceramics. We aim at studying the sintering of the selected clays and the effect of using the peel waste of Musa paradisiaca (readily available in tropical regions) fruit on the final microstructure and properties of use of samples shaped by unidirectional pressing. The deposit of Nzila in Central African Republic (CAR) has been selected regarding the geographical location, the available amount and the previous use for the production of local hand-made pottery. Two types of clays were used from this deposit, NZ1 and NZ2. A commercial kaolin (BIP kaolin, Imerys) serves as the control or reference raw clay to investigate the fruit waste influence over the sintering behavior. Firsly, various amount of the fruit waste (0, 10, 15, 20 and 30 mass%) have been mixed with kaolin BIP and sintered at 1000°C and 1200°C with a dwelling time of 1h. Secondly, the NZ1 and NZ2 clays sintering behavior has been considered from previous experiments, and used to manufacture samples containing up to 15 mass % of the fruit waste. In the latter case sintering has been performed in the range 1000 to 1200°C with dwelling times of 1h and 2h. The results obtained were correlated to the compressive mechanical strengths obtained for the studied samples.

2:00 PM

(ICACC-S16-009-2023) A review on alternative use of Rice husk and its ash as a source of silica in ceramics (Invited)

S. .*[1]

1. Guru Nanak College Budhlada, Punjab, Agriculture & Food Processing, India

In recent years, industrial waste and its by-products especially from food and agricultural sector is gaining a lot of attention in every aspect viz., economical, ecological, scientific, technological as well as social. Waste from rice-milling industries mainly Rice-husk is a by-product obtained from milling of rice and considered as a valuable and environment friendly by-product if used in ceramics because of high amount of silica present in it. In fuel generation process, rice-husk-ash is produced as main by-product, which can be a big problem for environment if not disposed-off, properly. The rice-husk-ash is a great source of amorphous silica, which makes its practical applications in various fields of ceramics. Keeping this in view, the aim is to discuss comprehensive overview on the production of nano-silica from rice-husk as well as its importance for environmental-safety. It can be summarized that rice-husk/rice-husk-ash is vital source of amorphous silica, which has a great potential to be used as an alternative for conventional sources of silica like quartz. Thus, amorphous silica from rice-husk-ash has been used successfully for the production of value-added ceramics for further practical applications.

2:30 PM

(ICACC-S16-010-2023) Up-cycling of Rice Husk as a Precursor for SiC Reinforcement in Geopolymer Composites (Invited)

C. Bagci*[1]; W. M. Kriven[2]

1. Hitit University, Department of Metallurgical and Materials Engineering, Turkey
2. University of Illinois at Urbana-Champaign, USA

Studies to improve the mechanical properties of geopolymer (GP) composites are generally based on two mechanisms: (i) high temperature in-situ crystalline phases forming in an amorphous GP matrix by heat treatment or (ii) making of GP-composites directly with GP-processing. On one hand well-formed matrix-reinforcement interface is achieved, on the other hand stress-related cracking due to heating and cooling are ongoing in the in-situ method. So, composites of GPs should be simultaneously made by direct addition of reinforcement phases with various types, shapes or sizes at ambient conditions. Rice husk has been used for GPs as a fumed silica alternative. It could also be used as silica precursor for SiC to reinforce GPs and thus contribute to a circular economy by increasing recycling of regional waste. For this end, one mole rice husk and four moles carbon black mixture were used in carbothermal reduction to obtain SiC particles. Synthesized-SiC at a rate of 5-15 % by wt were added to the GP-resins prepared by mechanically mixing a potassium silicate solution with metakaolin. According to relevant ASTM standards, room-temperature three-point flexural and compressive tests were made and Weibull strengths were estimated by processing the test results. When both tests are evaluated with XRD and SEM results of fractured surfaces, 5-10 % synthesized-SiC additions are seen as the working window.

3:20 PM

(ICACC-S16-011-2023) Geopolymer composite bamboo fiber reinforcement for high flexural strength and low water absorption (Invited)

M. G. Sá Ribeiro*[1]; I. P. Miranda[1]; W. M. Kriven[2]; A. Ozer[3]; R. A. Sa Ribeiro[4]

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2. University of Illinois at Urbana-Champaign, USA
3. University of Illinois at Urbana-Champaign, Material Science and Engineering, USA
4. INPA-National Institute for Amazonian Research, Green Building and Engineering Laboratory, Brazil

This work focused on designing an engineered material combining metakaolin-based geopolymer composite with bamboo fiber reinforcements for high flexural strength and low water absorption. Natural fibers are plentiful and renewable resources, biodegradable, and exhibit attractive physical and mechanical properties when used as sustainable reinforcements for composites in construction applications. The type of geopolymer matrix plays an important role in the strength, stability, and durability of the composites. The strength, stiffness, and durability of natural fiber reinforced geopolymers are affected by the type of fiber, fiber processing/treatment and its layup design, and the interaction between fiber-matrix interface. This study evaluated geopolymer composites reinforced with particulates (chamotte, sands) and bamboo fibers which were water-treated or alkali-treated. The composites reinforced with sand particles and bamboo fibers met the standard requirements.

3:40 PM

(ICACC-S16-012-2023) Mechanical Properties of Hemp Hurd Reinforced Geopolymer

G. Jarrold*[1]; E. Oh[1]; W. M. Kriven[1]

1. University of Illinois at Urbana-Champaign, USA

Geopolymer composites reinforced with chopped hemp hurd were produced and evaluated mechanically in 3-point flexure and compression. Potassium-based geopolymer of stoichiometry $K_2O\bullet Al_2O_3\bullet 4SiO_2\bullet 11H_2O$ (Kasolv® from PQ Corp, Malvern PA, USA) and sodium based geopolymer of stoichiometry $Na_2O\bullet Al_2O_3\bullet 4SiO_2\bullet 13H_2O$ (Sodium Metso Beads from PQ Corp, Malvern PA, USA), were fabricated by mixing metakaolin and water glass at high shear of 2000 rpm. Chopped hemp hurd fibers ¼″ in length were washed with 1 M NaOH for 30 min to remove lignin, a plant fat that causes staining and may result in degradation of the geopolymer matrix over time. The fibers were then ultrasonicated in tap water, rinsed in deionized water and dried prior to addition to the geopolymer. Hemp hurd was incorporated in amounts ranging from 2.5 - 10 wt% and 20 wt% of either sand or chamotte was added as a particulate reinforcement. Mechanical evaluation was performed through 3-point flexure testing of 1″x1″x 6″ bend bars and compressive testing of 1″ diameter x 2″ height cylinders. Flexural strengths of ~18.5 MPa and compressive strengths of ~30 MPa were recorded for potassium-based geopolymer composites with chamotte and hemp hurd additives.

Synthesis, Processing, Microstructure

Room: Coquina Salon C (North Tower)
Session Chair: Waltraud Kriven, University of Illinois at Urbana-Champaign

4:00 PM

(ICACC-S16-013-2023) Formation of ferrisilicates during the geopolymerization of laterites: Phases evolution, microstructure and micromechanics (Invited)

E. Kamseu*[3]; A. Akono[1]; R. Kaze[2]; J. Nouping[3]; S. Rossignol[4]; C. Leonelli[5]

1. Northwestern University, Civil and Environmental Engineering, USA
2. University of Yaoundé 1, Department of Inorganic Chemistry, Cameroon
3. Local Materials Promotion Authority, Cameroon
4. University of Limoges, IRCER, UMR 7315, France
5. University of Modena and Reggio Emilia, Department of Engineering Enzo Ferrari, Italy

Natural Iron-rich aluminisolicates, laterites, abondantly available in the tropical area of the World have amorphous to metastable structure with goethite and hematite that can dissolved in alkaline solution together with kaolinite already corroded by iron minerals. Simulating hydrothermal curing conditions in the temperature range between 50 and 100°C, ferro and ferrisilicates are formed in nanosizes. The combined action of the geopolymerisation and the formation of ferro and ferrisilicates are investigated using FT-IR, XRD, MIP, ESEM, TEM and micromechanical characterizations. The composites from laterites based geopolymerisation appeared with low porosity and water absorption, relatively high flexural strength (more than 20 MPa), high indentation modulus (more than 100 GPa) high indenation hardness (9-10 GPa) as well as high fracture toughness (more than 1 MPam$^{0.5}$). The energy saving process used and the characterisatics of the final composites make laterites based geopolymers an interesting prospectives for the future of buildings and construction.

4:20 PM

(ICACC-S16-014-2023) Influence of alkaline and iron precursors on geopolymer matrix at high temperature (1150 °C)

Q. Cligny*[1]; D. Brandt[2]; S. Rossignol[3]

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2. CEA, DAM, France
3. Laboratoire SPCTS, France

Influence of alkaline and iron precursors on geopolymer matrix at high temperature (1150 °C) In order to develop projects that are environmentally friendly, economically viable and sustainable, binders based on geopolymers are favoured. Geopolymer synthesis follow the reaction between aluminosilicate sources with an alkaline solution. Some applications require materials that could be used at high temperature for instance to avoid or resist to fire. The objective of this work is to understand the effect of alkaline content and iron additives on the geopolymer behaviour at high temperature (1150 °C). In this study, different alkaline solution concentrations and two iron sources have been investigated. Firstly, physico-chemical characteristics of the mixture are presented, such as initial viscosity and solution density. Then, characterisation of thermal analysis, dilatometry and mercury porosity were evaluated. Finally, features and mechanical resistance changes before and after a high temperature (1150 °C) cycle were discussed according to structural characterisation. Silicate alkaline nature and concentration govern properties from fresh state to consolidated one. The iron precursors are promising additives to reach materials to be used at high temperature.

4:40 PM

(ICACC-S16-015-2023) Effect of Nanomaterials on the Properties of KNa Activated Metakaolin Based Engineered Geopolymer Composites

R. Abufarsakh*[1]; H. Noorvand[1]; G. Arce[2]; M. Hassan[1]; S. Subedi[1]; S. Sukhishvili[3]; O. D. Huang[3]; M. Radovic[3]

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2. Virginia Department of Transportation, USA
3. Texas A&M University, Materials Science & Engineering, USA

Guided by fiber/matrix micromechanics and fracture mechanics principles, Engineered Geopolymer Composites (EGCs) exhibit pseudo-strain-hardening behavior (PSH) at low fiber content (i.e., 1 to 2 vol.%). To date, fly ash-based EGCs have been studied extensively, yet the compositional space of metakaolin-based EGCs remains vastly unexplored. To this end, the following study investigates the physical and mechanical properties of KNa (i.e., 50 mol.% potassium and 50 mol.% sodium) activated metakaolin-based EGCs reinforced with pristine ultra-high molecular weight polyethylene (UHMWPE) fibers. Aspects of the material composition studied include the effect of nanomaterial type (i.e., carbon nanofibers and carbon nanotubes) and content on the compressive strength and tensile properties. To fully exploit the potential of the material, plasma-treated UHMWPE fibers were also tested. Compressive strength results reveal that the addition of nanomaterials increases the compressive strength, yet nanomaterial content does not have a significant effect. Furthermore, the use of plasma-treated UHMWPE fibers increased the compressive strength of the control (i.e., without nanomaterials) by 13%. Moreover, it is expected that the addition of nanomaterials and the use of treated UHMWPE fibers will increase the tensile strength and tensile strain capacity.

S17: Advanced Ceramic Materials and Processing for Photonics and Energy

Multi-functional Materials II

Room: Coquina Salon G (North Tower)
Session Chairs: Daniel Chua, National University of Singapore; Elisa Moretti, Ca' Foscari University of Venice

1:30 PM

(ICACC-S17-015-2023) Radical Polymer Devices for Energy Storage and Hybrid Solar Cells (Invited)

G. Fanchini*[1]

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

Organic radical polymers where each repeating unit is a stable radical are unique alternatives to semiconducting polymers where repeating units normally exist only in one state and are at the basis of most organic electronics proposed to date. The electrical conductivity of radical polymers stems from the oxidation of a radical site forming a cation (R+) which can on its turn oxidize a neighboring neutral site, thus moving the oxidation even further along the chain in a dynamic redox process. Despite recent claims of p-type polynitroxyl doping, such materials have so far fallen short of any gated electronic devices, such as field effect transistors in which separated p-type and n-type transport is necessary. Here, we will review our work [on electrotunable radical polymers with ambipolar characteristics, in particular poly-(oxo) verdazyls where repeating units may stably exist in three distinct configurations: anionic (R-), neutral (R*) and cationic (R+). We show that reversible disproportionation and comproportionation (2R* <--> R- + R+) along the polymer chain enables both ambipolar and p-type field effect transistors in these systems. The same systems can be utilized in single-layer flow batteries, resistive memory devices and hybrid solar cells. The competitive advantages of tri-stable poly-(oxo) verdazyl radical polymers offset, in part, their behaviour as strongly correlated systems and will be discussed.

2:00 PM

(ICACC-S17-016-2023) Controllable synthesis and development of one dimensional oriented CdS nanoarray photoanodes for efficient photoelectrochemical hydrogen evolution (Invited)

M. Siaj*[1]

1. University of Quebec, Montreal, Faculty of Science, Canada

Photoelectrochemical (PEC) water splitting for hydrogen evolution is a highly efficient and eco-friendly technology in solar energy conversion, but the question still how to build an available PEC system with satisfied photoconversion efficiency. Photoactive materials as the basic component for PEC systems have been extensively studied due to their distinct structure features. Among them, cadmium sulfide (CdS), especially one dimension (1D) oriented CdS nanoarray has attracted considerable attention and is regarded as a promising candidate for PEC hydrogen production due to its suitable narrow band gap (2.4 eV), excellent electrical/optical properties, larger aspect ratio and confined charge transfer path for lower carrier loss. Here, the rational design of CdS-based nanojunctions, like Plasmon-enhanced, Z-scheme and p-n scheme heterostructure, will be presented to overcome these drawbacks through coupling 1D CdS with other components. In parallel, a heterojunction fabrication as a promising strategy that can greatly boost the charge carrier separation and improve the solar-to-hydrogen conversion efficiency of the PEC will be discussed.

2:30 PM

(ICACC-S17-017-2023) Sustainable Carbon Nanomaterials in Green Energy Applications (Invited)

R. Naccache*[1]

1. Concordia University, Chemistry and Biochemistry, Canada

Carbon nanomaterials have garnered significant interest owing to their versatile properties with a vast potential in sensing and imaging applications, in optoelectronics, Catalysis, as well as energy conversion. Their ultra-compact size, low cytotoxicity, low photobleaching/blinking, tunable photoluminescence, combined with simple, environmentally friendly and low-cost synthesis, makes them ideal and cost-efficient candidates for study. In this work, we discuss the synthesis of carbon nanomaterials via a myriad of techniques including solvent mediated and solid-state bottom-up synthesis methods, with simple organic precursors. We focus our efforts on tailoring their physico-chemical and optical properties and exploit them in order to design multifunctional materials for clean energy applications namely conversion of refined and waste oils to biofuels via chemical or light-mediated approaches. We also investigate the potential to prepare these nanosized dots from waste materials as we prioritize sustainability without compromising function and performance in clean energy and biofuel applications.

3:20 PM

(ICACC-S17-019-2023) Optical and magnetic probes of the interplay between spin, charge, and lattice in the multiferroic RMn_2O_5 (Invited)

S. Mansouri[1]; M. Chaker*[1]

1. INRS, Energie materiaux télécommunications, Canada

Multiferroic materials showing strong magnetoelectric coupling have opened new perspectives for controlling the magnetization by an electric field. With such materials, data processing in RAM memories could be carried out with electric fields rather than magnetic fields. These materials have also shown a giant rotating magnetocaloric effect (RMCE) at relatively low magnetic fields paving the way to the implementation of compact and efficient magnetic refrigerators operating at low temperatures. Such an effect is attributed to the magnetocrystalline anisotropy. However, much fundamental research still needs to be performed for understanding the microscopic mechanisms responsible of magnetoelectric and magnetocaloric effects and identify the parameters that govern them. Our research on RMn_2O_5 materials where R=Sm and Nd aims to address these issues. In this presentation, we report a detailed study of Raman and crystal-field excitations in RMn_2O_5 compounds as a function of temperature ranging from 5 to 300 K and under a magnetic field of up to 11 Tesla. Interestingly, our findings revel that the Kramers degeneracy of the ground-state is lifted due to the R^{3+}-Mn^{3+} magnetic interaction in the ferroelectric phase. In this presentation, we also demonstrate that the magnetocrystalline anisotropy in RMn_2O_5 is determined by the quadrupolar charge distribution of 4 f shells.

3:50 PM

(ICACC-S17-020-2023) Double Perovskite Oxides as Bifunctional Catalysts for Oxygen Evolution and Reduction Reactions

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

Metal-air batteries have a much higher theoretical energy density than lithium-ion batteries. They are frequently advocated as the solution for next-generation electrochemical energy storage for applications including electric vehicles or grid energy storage. Optimization of the air catalyst and cathode architecture plays a decisive role in improving the performance of metal-air batteries. Air cathode is the performance-limiting electrode in metal-air batteries because of the sluggish ORR/OER activities of most electrocatalysts. Herein, we develop novel double perovskite oxides and demonstrate their bifunctional electrochemical activity toward oxygen evolution and reduction reactions. The materials synthesized through sol-gel chemistry were characterized for physicochemical by various diffraction and spectroscopic techniques and further evaluated for electrochemical characteristics. A vibronic superexchange interaction resulting in a predominant M^{3+}–M'^{3+} interaction was found to positively impact the electrocatalytic activity by enhancing the current densities and lowering the Tafel slope. The present study's findings deliver a promising pathway for developing novel earth-abundant ceramics and substantiate their suitability as bifunctional catalysts for oxygen evolution and reduction reactions.

S18: Ultra-High Temperature Ceramics

Processing-Microstructure-Property Relationship I

Room: Coquina Salon A (North Tower)
Session Chair: Gregory Thompson, Thompson

1:30 PM

(ICACC-S18-017-2023) Deposition Characteristics and Corresponding Microstructural Evolution in Composite-based UHTC Structures (Invited)

G. Thompson*[1]; M. Large[1]; C. Stotts[2]; C. R. Weinberger[2]

1. University of Alabama, Metallurgical & Materials Engineering, USA
2. Colorado State University, Department of Mechanical Engineering, USA

A series of multilayered UHTC carbide/metallic laminates are examined with respect to their phase stability and mechanical properties. Here, metal carbide films are grown through reactive sputter deposition from a metallic source exposed to different carbon-based precursor gases at various associated partial pressures. The carbide phase is found to be either amorphous or crystalline dependent upon the deposited substrate temperature. Inserted within the carbide phase is a series of metallic layers that provide low temperature toughness. This composite is then ex situ annealed and the stability of the microstructure as a function of temperature and time are assessed and linked to mechanical strength. Through carbon content control and/or bilayer thickness ratios, the composite can convert to a single sub-stoichiometric carbide phase with such experiments linked to a model of the diffusivity and carbide phase stability. Such a phase evolution provides a beneficial balance of mechanical property attributes required for UHTC resiliency over a range of thermo-mechanical conditions.

2:00 PM

(ICACC-S18-018-2023) Micro/nanomechanics of ZrB_2 grains: The effect of anisotropy, temperature and dislocations (Invited)

T. Csanádi*[1]

1. Institute of Materials Research, Slovak Academy of Sciences, Slovakia

Zirconium diboride (ZrB_2) is a promising ultra-high temperature ceramic that can be used as parts and tools exposed to extreme environmental conditions at temperatures exceeding 2000°C in oxidizing atmospheres. Since the deformability of grains plays an important role in macromechanical performance, the deformation and fracture behaviour of ZrB_2 grains of polycrystalline samples were studied by various micro/nanomechanical testing. The ZrB_2 grains were tested by nanoindentation, nanoscratch and microcantilever bending at room temperature and micropillar compression at temperatures up to 500°C. Samples were subjected to small-scale characterization techniques (SEM, EBSD, XRD) before and after the mechanical testing. It was revealed that ZrB_2 grains exhibit orientation-dependent hardness and indentation modulus in the range of 30-40 GPa and 600-660 GPa, respectively. Anisotropic yield and fracture strength, and fracture toughness were observed, which depend on the crystallographic orientation, pre-existing dislocation densities and testing temperature. It was revealed that ZrB_2 grains could show considerable plasticity in some specific cases and the operating slip systems were determined during micro-compression of prismatic pillars. The orientation-dependent deformation behaviours were described by analytical models proposed based on anisotropic dislocation nucleation and slip operation.

2:30 PM

(ICACC-S18-019-2023) Densification, Microstructure, and Properties of Zirconium Diboride (ZrB_2) with Carbon Additions

Y. Zhou*[1]; W. Fahrenholtz[1]; G. Hilmas[1]

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

Commercial Grade B ZrB_2 powder containing carbon additions up to 1.5 wt% were densified by hot-pressing. Particle size and purity of the starting ZrB_2 was examined. Prior to densification particle size was measured by laser light scattering, particle morphology was observed by scanning electron microscopy, and oxygen and carbon contents were measured by inert gas-fusion. Phenolic resin was used as an carbon source and the carbon yield was measured prior to add into ZrB_2. The powders with carbon additions were hot-pressed at 2150C with a pressure of 32MPa. Grain size and morphology were observed by scanning electron microscopy. Bulk densities were measured by Archimedes method. Grain boundaries were observed using transmission electron microscopy to identify impurity phases. Finally, the effect of carbon content on thermal conductivity was also analyzed.

Response in Extreme Environments I

Room: Coquina Salon A (North Tower)
Session Chair: Luca Zoli, CNR ISTEC

3:20 PM

(ICACC-S18-020-2023) Elevated Temperature Flexural Strength of Ultra-High Temperature Ceramic Matrix Composites Manufactured by Sintering, Pyrolysis or Hybrid Processes (Invited)

L. Zoli*[1]; F. Servadei[1]; A. Vinci[1]; P. Galizia[1]; D. Sciti[1]

1. CNR-ISTEC, Italy

There is an increasing demand for improving durability of structural components at temperature exceeding 2000 °C. Ultra-High Temperature Ceramic Matrix Composites (UHTCMCs) are novel materials, which can overcome the limits associated with currently used ceramic matrix composites. The Ultra-High-Temperature Ceramic (UHTC) matrix, based on transition metal borides, carbides, and nitrides, possess melting temperatures that exceed 3000 °C, and good strength and oxidation/ablation resistance even over 2000°C. UHTCMCs are potential materials for manufacturing of leading edges and Thermal Protection System (TPS) in hypersonic vehicles or rocket nozzles and turbine blades for propulsion. In the last ten years, our team developed several processes for manufacturing UHTCMCs with the aim of improving UHTCMCs performance and durability. Here, we explore the thermo-mechanical properties of continuous reinforced C_f/ZrB_2-SiC materials obtained via sintering (Hot Pressing/HP or Spark Plasma Sintering/SPS), via Polymer Infiltration and Pyrolysis (PIP) or via hybrid processes (PIP + HP) through bending tests up to 1500 °C. HT flexural strengths ranged from 100 to 500 MPa as function of the thermal treatment.

3:50 PM

(ICACC-S18-021-2023) Investigation of mechanical properties and melting point of tantalum and hafnium carbo-nitrides

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1. European Commission, Joint Research Center Karlsruhe, Germany

Ultrahigh temperature ceramics (UHTCs) are of interest for the development of new applications working under extreme conditions, such as space vehicles, nuclear reactor or high temperature turbines. Indeed, UHTC show excellent performances to withstand high temperatures, thermal shocks and corrosive environments. Among them, tantalum carbide (TaC) and hafnium carbide (HfC) exhibit very interesting mechanical properties and one of the highest melting points (>3500K). Recently, Hong and van de Walle predicted that the sub-stoichiometric hafnium carbonitride $HfC_{0.56}N_{0.38}$ would be the compound with the highest melting point reported among all materials (>4000K). In this framework, we studied tantalum and hafnium carbo-nitride systems in order to characterise experimentally their mechanical properties and melting point, as they are key properties to know the limits of the use of these materials. Dense samples were prepared by spark plasma sintering under argon atmosphere to avoid oxidation. The final composition, morphology and structure were confirmed by various characterization techniques (XRD, SEM, C/N analysers…). The mechanical properties, e.g. hardness and Young's modulus, were determined by nano and macro indentation experiments. The melting point was determined by a laser heating facility that allows us to reach a temperature high enough to melt ultrahigh temperature ceramics.

4:10 PM

(ICACC-S18-022-2023) Melting point determination of high entropy materials via laser heating and radiation pyrometry

M. Milich*[1]; E. Hoglund[5]; H. B. Schonfeld[1]; M. Qin[4]; D. Robba[2]; L. Vlahovic[2]; K. Boboridis[2]; R. Konings[2]; J. Luo[3]; P. E. Hopkins[1]

1. University of Virginia, Mechanical and Aerospace Engineering, USA
2. EU Joint Research Center-Institute for Transuranic Materials, Germany
3. University of California, San Diego, USA
4. University of California, San Diego, Department of NanoEngineering, Program of Materials Science and Engineering, USA
5. University of Virginia, Materials Science and Engineering Department, USA

High entropy ceramics are a promising new category of material receiving attention from the hypersonics community due to their high strength and hardness, and high temperature stability. Although the properties of these materials have been studied extensively at room temperature, measurements at the ultra-high temperatures these materials are intended to operate at is challenging and has been rather limited in previous works. Using laser heating and radiation pyrometry, we conduct contactless measurement of melting temperature on a variety of high entropy carbides, borides, and two-phase carbide-boride mixtures. We further characterize the materials after heating with energy dispersive spectroscopy (EDS) and perform electron energy loss spectroscopy (EELS) on TEM samples taken from the melt region, to study the effects of high temperature and extreme thermal gradients on the solid solution. We compare the compositional gradients induced by fast-pulsed heating versus slow melting to study the effect of thermal shock and limited diffusion, as well as topological effects such as dendrite formation.

4:30 PM

(ICACC-S18-023-2023) A steady-state laser heating technique to measure the thermal conductivity of ceramics at ultrahigh temperatures and in their molten state

P. E. Hopkins*[1]; M. Milich[2]; S. Bender[1]; H. B. Schonfeld[2]; D. Robba[3]; L. Vlahovic[3]; K. Boboridis[3]; R. Konings[3]; J. Luo[4]; J. Gaskins[5]

1. University of Virginia, USA
2. University of Virginia, Mechanical and Aerospace Engineering, USA
3. Joint Research Centre, Germany
4. UCSD, USA
5. Laser Thermal, Inc., USA

The measurement of the thermal conductivity of materials in extreme environments and in molten states is a continued challenge. Typical transient approaches are applied in a heat front/probe back geometry which can also suffer from the sample instabilities when molten as mentioned above. However, focused laser-based pump-probe techniques, which interrogate a relatively small, localized volume of heated material on the same side in which the laser induces the heating presents a novel avenue for measurements of thermal properties. Here, we present a new method to measure thermal conductivity of materials to high temperatures based on laser heating, in which a continuous wave beam rapidly heats the sample surface to the desired temperature, then the amplitude of the laser beam is perturbed to induce a steady state temperature profile on the sample surface. We analyse the temperature vs. laser power data and extract the thermal conductivity of a variety of materials, including W and Mo standards, which show good agreement with the literature. We then demonstrate this technique on selected high entropy caribes and borides, measuring both thermal conductivity and emissivity up to and through their melting temperatures.

S19: Molecular-level Processing and Chemical Engineering of Functional Materials

Polymer-Derived Ceramics III

Room: Ballroom 3 (South Tower)
Session Chair: Günter Motz, University of Bayreuth

1:30 PM

(ICACC-S19-016-2023) Modification of organosilicon polymers by transition metal complexes towards functional nanocomposites for energy (Invited)

S. Bernard*[1]

1. CNRS, IRCER, France

The scientific and technological challenges of energy-related fields are mainly associated with the emergence of new, advanced knowledge and fundamental understanding of materials. In the different categories of materials, ceramics offer a unique combination of physical and chemical properties making them key contributors for energy production/conversion and storage applications. The Polymer-Derived Ceramics (PDCs) route offers new preparation opportunities in ceramic sciences. In particular, the organosilicon polymer backbone can be used as a highly reactive platform to link or coordinate lower-molecular weight transition metal (TM) complexes. The as-obtained pre-ceramic polymer will promote upon heat-treatment the precipitation of metallic or ceramic (TM carbides, nitrides and/or silicides) phases in a ceramic matrix composed of Si, C, N and/or O elements that contribute to dispatch PDC towards the energy field. In this talk, we will present our last results on the modification of polysilazanes with metal-organic species to offer functionalities to the materials obtained after the thermo-chemical conversion of precursors. As a proof of concepts, we will investigate the optical selectivity and electrocatalytic properties of final materials, namely nanocomposites, according to the composition of the transition metal-based phase.

2:00 PM

(ICACC-S19-017-2023) Pyrolysis of silicone resin in reactive CO_2 environment: A comprehensive study of the polymer-to-ceramic transformation

G. D. Soraru*[1]; R. Campostrini[1]; A. Zambotti[1]; M. Biesuz[1]

1. University of Trento, Industrial Engineering, Italy

The composition and properties of polymer derived silicon oxycarbide glasses can be controlled by the pyrolysis atmosphere. Pyrolysis in inert atmosphere leads to silicon oxycarbide and free carbon, $SiOC/C_{free}$, while reactive H_2 or CO_2 atmospheres result into stoichiometric SiOC and SiO_2/C_{free}, respectively. In this study we investigate the pyrolysis process in flowing CO_2 of a methyl-substituted silsesquioxane resin, $CH_3SiO_{1.5}$. For comparison the same precursor was also pyrolyzed in N_2. The work was performed recording, in both CO_2 and N_2 atmospheres, TG-MS analysis and FT-IR spectra at different stages of the pyrolysis process. The characterization of the resulting ceramic, i.e. $SiOC/C_{free}$ and SiO_2/C_{free} was completed via ^{29}Si MAS-NMR and by studying their high temperature XRD evolution. Results show that CO_2 reacts with Si-CH_x-Si to form $SiO_2+C_{free}+H_2O$. The XRD analysis shows that the high temperature crystallization of the material pyrolyzed in CO_2 does not differ substantially from the one treated in inert N_2 gas. This work was supported by the Italian Ministry of Education, University and Research (MIUR) within the program PRIN-2017 "Nanostructured Porous Ceramics for Environmental and Energy Applications".

2:20 PM

(ICACC-S19-018-2023) On the oxidation of polymer derived silicon carbide ceramics by in situ NAP-XPS and NMR

J. O. Garcia*[1]; H. Jain[2]; G. Singh[3]; R. Thorpe[5]; J. Roberts[4]; W. Breyer[4]

1. Lehigh University, Materials Science and Engineering, USA
2. Lehigh University, International Materials Institute for New Functionality in Glass, USA
3. Kansas State University, Mechanical and Nuclear Engineering Dept., USA
4. Lehigh University, Chemistry, USA
5. Lehigh University, Institute for Functional Materials and Devices, USA

The study of polymer-derived ceramics (PDCs) has developed significantly in the last decade. However, there are limited studies investigating oxidation reactions as they occur on the surface and interior of these materials. We aim to study the effects of oxidation by different gases on the structure of a carbosilane PDC, synthesized via crosslinking at 150°C and pyrolysis at 800°C. The resulting ceramic is studied in situ by use of Near-Ambient Pressure (NAP) XPS system to examine the oxidation products as a function of time and temperature. Initial observations suggest that a surface oxidation reaction occurs within the C-C, C-Si, and C-Si-O region at temperatures above 500°C. Solid-state NMR is used ex situ to examine the bulk structure after treatment at select temperatures, unraveling the average chemical changes that occur before and after oxidation.

2:40 PM

(ICACC-S19-019-2023) Design, doping strategy and characterization of Polymer Dervied SiAlON

M. Kumari*[1]

1. CNRS, IRCER, France

White light-emitting diodes are a breakthrough technology with great photoluminescence efficiency, low energy consumption and non-toxicity with several applications like LCD backlights and display technologies. The crystal structure, chemical composition, and shape/size of photoluminescent materials must be strictly regulated due to the demanding conditions involved with the widespread usage of LEDs. Recent research has revealed that rare-earth-doped oxynitride is potential of photoluminescence because of their excellent thermal and chemical stabilities. By equivalently substituting Al-O for Si-N in the hexagonal crystal structure of β-SiAlON, the chemical formula of this material is $Si_{6-z}Al_zO_zN_{8-z}$ (where z is the number of Al-O pairs that replace Si-N pairs and ($0 \le z \le 4.2$)). The larger emission and excitation wavelengths make SiAlON suitable host material for high luminescence efficiency but it must have high phase purity and homogenous crystal size distribution. In this paper, we investigate the polymer derived ceramics (PDCs) route for the designing of β-SiAlON. We expect a high compositional and structural homogeneity for the envisioned materials and a relatively low temperature of preparation. The evolutive material is characterized at each step of SiAlON preparation and, as a proof of concept doping effect with rare-earth element is explored.

Polymer-Derived Ceramics IV

Room: Ballroom 3 (South Tower)
Session Chair: Samuel Bernard, CNRS

3:20 PM

(ICACC-S19-020-2023) Synthesis of UHTC powders using wet or dry process (Invited)

S. Lee*[1]; H. Lee[1]; S. J. McCormack[2]

1. Korea Institute of Materials Science, Republic of Korea
2. University of California, Davis, Materials Science and Engineering, USA

Among ultra-high temperature ceramics (UHTC), ZrB_2, ZrC, HfB_2, HfC and TaC have received a special attention in the recent years due to the combination and tenability of their chemical and physical properties. In addition, Ta-Hf-C system was reported to have extremely high melting points. The synthesis of various UHTC 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, the synthesys of UHTC nano-powders which have the desired properties were performed using the Metal organic framework (MOF) method, organic steric entrapment method and plasma method. High purity UHTC nano-powders were successfully synthesized by the carbothermal reduction of oxide powders at 1400-1900°C. The sizes of the synthesized powders were in the range of 50-300nm. The effects of synthesis temperature and carbon contents on the phase composition and microstructures of the synthesized powders were analyzed.

3:50 PM

(ICACC-S19-021-2023) Effect of ultra-fast ceramization of polymer-derived SiOC aerogels and their application as anodes for Na-ion batteries

A. Zambotti*[1]; M. Melzi d'Eril[2]; M. Graczyk-Zajac[2]; E. Ionescu[2]; G. D. Soraru[1]

1. University of Trento, Industrial Engineering, Italy
2. Technical University Darmstadt, Germany

In the last year, the ultra-fast pyrolysis of polymer-derived ceramics (PDCs) became object of interest, since it might result in parallel or alternative reactions to conventional pyrolyses, leading to ceramic systems with unexpected microstructures and properties. In this project, a vinyl-modified polysiloxane-derived aerogel was subjected to an ultra-fast ceramization ($>100°C\ s^{-1}$) up to 1000°C, and it was deeply characterized through FTIR, micro Raman and NMR spectroscopies, SEM, N_2 physisorption, and mass spectrometry, unveiling the differences with the same aerogel treated at $5°C\ min^{-1}$. The obtained results will be discussed, featuring a difference in the atomic arrangement of silicon, as well as in the dimensions of the free carbon nanodomains present within the SiOC matrix. The porosity of the ultra-fast pyrolyzed aerogel seems to be affected only in its macroporous range. Finally, the two SiOC aerogels were employed as anodes for Na-ion batteries, both showing remarkably steady capacities in the 160-110 $mAh\ g^{-1}$ range and efficiencies close to 97%.

4:10 PM

(ICACC-S19-022-2023) Functional Bioglass-C Nanocomposite Scaffolds by Masked Stereolithography of a Preceramic Polymer-based Nanoemulsion (Invited)

F. M. Stabile[2]; H. Elsayed[1]; E. Bernardo*[1]

1. Unniversity of Padova, Department of Industrial Engineering, Italy
2. CETMIC, Argentina

Silicone resins are attractive both as precursors of silicate bioceramics and as feedstock for additive manufacturing technologies, including stereolithography. The two aspects may be successfully combined operating with simple silicone-based blends, consisting of a silicone polymer mixed with photocurable acrylates. The inclusion of a Ca salt, in nanoemulsion within the silicone-containing blend, enables the obtainment of homogeneous printed scaffolds, later converted into crack-free fully amorphous ceramic composites, by firing at 700 °C, in N_2 atmosphere. According to the quasi-molecular of CaO distribution, the new material features a fully amorphous matrix resembling 70S30C (70% SiO_2 and 30% CaO) bioglass, including pyrolytic carbon. The latter phase is intended to provide extra functionalities, such as enhanced absorption of IR light, useful for disinfection purposes. Preliminary results of the extensions of the nanoemulsion methodology to direct ink writing of silicone-based pastes are also presented.

4:40 PM

(ICACC-S19-023-2023) Heterometallic Actinide Alkoxides as Molecular Precursors for the Formation of Ternary Actinide Oxide Nanomaterials

A. Lichtenberg*[1]; A. Raauf[1]; S. Mathur[1]

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

The application of (mixed-) metal alkoxide precursors for the formation of phase pure binary or ternary metal oxide phases and nanomaterials is well established for s-, p- and d-block metals and the lanthanides. Rare examples for the formation of actinide metal oxides derived from actinide alkoxides were previously demonstrated by magnetic field assisted chemical vapor deposition of UO_2 thin films by using $U(O^tBu)_6$ as precursor or through hydrolysis of $U(ditox)_4$ (ditox = $^tBu_2MeCO^-$) to obtain UO_2 nanocrystals and nanofilms. Although syntheses of ternary actinide oxides, such as MUO_3 and M_2UO_4 (M = Li, Na, K, Rb, Cs), MUO_4 (M = Sr, Ba, Cr, Mn, Fe, Ge, Pb) or $MThO_3$ (M = Sr, Ba), have been successfully established through solid state, melt or multiple source precursor methods, they are facing synthetical difficulties or phase inhomogeneity. Several heterobimetallic actinide alkoxides of thorium(IV), uranium(IV) and uranium(V) with monovalent alkali metals (M(I) = Li, Na, K, Rb, Cs), bivalent group 14 metals (M(II) = Ge, Sn, Pb) and trivalent metals (M(III) = Al, Fe) have been synthesized. Their application as single source precursors in hydrolytic, solvolytic or solvothermal processes have shown clean conversations to targeted $AnMO_x$ (An = U, Th; M = alkali, bivalent group 14 and trivalent metal cations) ternary actinide oxide phases in form of nanocrystalline powders.

Poster Session A

Room: Ocean Center Arena

5:00 PM

(ICACC-P002-2023) 3-D Printing of Interdigitated Battery Electrodes

D. H. Nguyen*[2]; A. S. Almansour[1]; M. Singh[3]; M. C. Halbig[2]; Z. Tuchfeld[2]

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

The movement towards electric aircraft and higher performing rechargeable batteries seeks to find a solution to improve the energy and power densities of lithium-ion batteries. This can be done through advancements in electrochemical performance and manufacturing processes. We developed procedures for preparing 3D printable inks for Lithium Iron Phosphate (LFP) cathodes and Lithium Titanate Oxide (LTO) anodes with the addition of conductive carbon nanoparticles to enhance the electrical conductivities of battery electrodes. The cathode and anode ink ratios were optimized to contain around 61 wt.% solid loading of the lithium iron phosphate powder and the carbon nanopowder while maintaining the necessary ink rheology for 3D printing. Ink rheology was tailored with use of compatible additives such as solvents, dispersants, defoamers, binders, viscosifiers, and plasticizers. A trade study was conducted to demonstrate the effect of various processing parameters in 3D printing of interdigitated electrodes with a height of approximately 0.700 mm and line widths of approximately 0.300 – 0.400 mm. This trade study gave valuable information about the 3D printer setup and operation for the benefit of future additive manufacturing activities.

(ICACC-P003-2023) Room temperature synthesis of Al^{3+}-doped γ-Ga_2O_3 nanoparticles by direct oxidation of Al-Ga alloy using ultrasound irradiation

T. Yamanaka*[1]; Y. Hayashi[1]; J. Fukushima[1]; H. Takizawa[1]

1. Tohoku University, School of Engineering, Japan

Gallium oxide (Ga_2O_3) is a wide-bandgap ceramics with five polymorphs (α, β, γ, δ, ε). γ-Ga_2O_3, which is one of the polymorphs of Ga_2O_3, shows excellent photocatalytic activity and fluorescence. However, γ-Ga_2O_3 can only be synthesized by limited homogeneous aqueous solution methods, such as hydrothermal method because γ-Ga_2O_3 transforms to β-Ga_2O_3 under high-temperature condition (600 °C or above). Hydrothermal method is not environment friendly, although this method is effective technique for synthesis of various ceramics nanomaterials. For example, high-temperature and high-pressure conditions are required for hydrothermal method, and metal salts, which are often used as raw materials for hydrothermal method, generates counter-anions (Cl^-, $(NO_3)_3^-$, SO_4^{2-}, etc.) needed to be removed after the reaction. There is a need for better processing for synthesis of ceramics nanomaterials without environmental burden. Thus, we investigated the fabrication of γ-Ga_2O_3-based nanoparticles using gallium (Ga)-based alloy and ultrasound at near room temperature. In the case of aluminum (Al)-Ga alloy, Al^{3+}-doped γ-Ga_2O_3 nanoparticles, which shows large specific surface area (181 m^2/g), can be obtained at 60 °C. In poster session, we will present about the mechanism of oxidation of Al-Ga alloy by ultrasound irradiation in detail.

(ICACC-P004-2023) Ion Gated Thermoelectricity with Thin Film Oxides for the Internet of Things

C. Nunez Lobato*[1]; N. Pryds[2]

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

In the context of searching for alternative energy sources to power the network of the Internet of Things (IoT), miniaturized energy harvesters have been suggested as solutions to capture renewable energy from one or more energy sources. In this regard, thin films of complex oxides, such as in SrTiO3(STO)-based materials, are a viable option for thermoelectric IoT micro-harvesters. However, challenges remain to both increase their low efficiency and characterize the thermoelectric properties of these perovskite materials in a device. In this work we explored a solid-state platform with Nb-doped STO thin films that enables characterization of the thermoelectric parameters describing the figure of merit (zT) in the epitaxial film, thereby measuring electrical conductivity (σ), Seebeck coefficient (S) and thermal conductivity (κ). An ionic gating technique was further applied to the Nb:STO semiconductor, allowing field effect control of key properties of the material, particularly on σ, S and its carrier density. Merging the field of iontronics with thermoelectrics opens in this sense a promising strategy applicable to a variety of materials and configurations such as e.g. gating using solid electrolytes together with alternative device designs.

(ICACC-P005-2023) Influence of Spark Plasma Sintering Technique on Thermoelectric Properties of Sodium Tungsten Bronze

S. Maneeyom*[1]; M. Ohtaki[1]

1. Kyushu University, Department of Applied Science for Electronics and Materials, Japan

Metal oxides are one of the promising thermoelectric materials due to their high-temperature stability, low cost, and non-toxicity. Sodium tungsten bronze (STB) possessing high electrical conductivity is one of the candidates. However, its thermoelectric properties has not been throughly examined. In this study, STB precursors prepared by a reduction reaction of Na_2WO_4 were sintered at 800 °C by hot pressing (HP) and spark plasma sintering (SPS) techniques. Cubic STB phase was confirmed by XRD. Relative densities of

HP- and SPS-sintered samples were 98.2% and < 70%, respectively. SEM-EDS observed Na, W, and O elements were in all samples with a similar Na:W ratio of around 0.47:1, confirming that the difference between HP and SPS techniques unaffected the crystal phase and chemical composition of the samples. The HP-sintered sample showed the largest grain sizes (~ 2 μm) among all the samples. On the other hand, the SPS-sintered samples were characterized by cubic particles of several hundred nanometers in size and high porosity, resulting in low thermal conductivity. The lowest k_{total} and $k_{lattice}$ were observed on the SPS-sintetred sample at 800 °C with the value of 6.57 and 0.69 W/mK, respectively. As a result, the dimensionless figure of merit (ZT) of the SPS-sintered sample was higher than that of the HP-sintered sample by 54.7%, and the maximum ZT was 0.019 at 800 °C

(ICACC-P006-2023) Fabrication of Oxide Multilayer Thermoelectric Generators

B. Capraro*[1]; D. Schabbel[1]; R. Loehnert[2]; A. Bochmann[2]; A. Ibrahim[2]; J. Topfer[2]

1. Fraunhofer IKTS, Germany
2. Ernst-Abbe-Hochschule Jena, Germany

Oxide-based thermoelectric multilayer generators (TEG) are fabricated using the ceramic multilayer process with oxide thermoelectric green tapes and printed metal layers. This technology is an interesting option enabling miniaturization and allowing for easy mass fabrication of multilayer TEGs. We demonstrate the fabrication of transverse multilayer thermoelectric generators (TMLTEG) with charge transport perpendicular to the heat flow direction. These generators consist of layers of tape-cast p- or n-type thermoelectric oxides in combination with metal layers printed at a certain angle with respect to the heat flow direction to create anisotropic thermoelectric properties. Applications include autonomous sensor systems with low power consumption. We report on the tape casting of thermoelectric oxides of $Ca_3Co_4O_9$, La_2CuO_4 and $CaMnO_3$ powders including optimization of the casting slurry composition and lamination behavior. We also report on screen-printing and testing of various precious metal paste systems as well as on the co-firing behavior of oxide tapes with metal layers. We demonstrate the layer stacking and fabrication of multilayer transverse generators using various thermoelectric oxides.

(ICACC-P007-2023) Enhanced Thermoelectric Properties of Metal Nanoparticles Incorporated Skutterudite Composites

M. Lee[1]; M. Kang[1]; G. Lee[2]; J. Cho[1]; Y. Lim[3]; W. Nam*[1]

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We report the effect of the incorporation of metal nanoparticles on thermoelectric properties in n-type filled skutterudite $In_{0.4}Co_4Sb_{12}$ (SKD). The SKD powder was prepared by a combination of induction melting and melt-spinning. A high-energy ball milling was employed to decorate Ag nanoparticles on the surface of SKD powder, and bulk composites (SKD-x wt% Ag, x = 0.05 ~ 0.2) were prepared by a spark plasma sintering (963 K, 50 MPa, 15 min). The electrical conductivity (σ) of the composites decreased with increasing the Ag content while the Seebeck coefficient (S) increased due to the low-energy charge carrier filtering effect by introducing a Schottky barrier at interfaces between the SKD matrix and Ag nanoparticle. The decrease in the carrier concentration by incorporating Ag nanoparticles led to an increase in mobility, which is desirable for power factor optimization, and the maximum power factor ($S^2\sigma$) of 46.6 x 10^{-4} W/mK^2 was achieved at 517 K for the sample with x = 0.05. Furthermore, the lattice thermal conductivity in the composites was effectively reduced due to an additional phonon scattering at the interfaces. Simultaneous control of electric and thermal properties in the composites could be realized by incorporating Ag nanoparticles in the SKD matrix, leading to an enhancement of ZT.

(ICACC-P008-2023) Evaluation of Thermal Conductivity and Melting Temperature of Single and Multi-Component Rare-Earth Disilicates

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In recent years, rare-earth silicates have become the industry standard for coating state-of-the-art SiC ceramic matrix composites (CMC) for gas turbine engine components, due to their low volatility, high melting point, and thermal shock resistance. Current research is focused on designing rare-earth silicate based environmental barrier coatings (EBC) with improved resistance to CMAS (CaO-MgO-Al2O3-SiO2), steam, and crack formation, while maintaining high temperature stability. In this work we compare the high temperature performance of a variety of single and multi-component rare-earth disilicates by measuring their melting point using laser heating and radiative pyrometry. We also measure their thermal conductivities using time-domain thermoreflectance (TDTR), a non-destructive optical pump-probe technique that relates change in surface reflectance to change in temperature. A multilayer thermal model is then fitted to the transient decay in surface temperature to determine the thermal properties of the material. These thermal conductivity and melting point measurements are used to evaluate the material's feasibility as an EBC in a high temperature environment.

(ICACC-P009-2023) Al_2O_3 aerogel-modified EB-PVD TBCs with enhanced CMAS corrosion resistance

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The threat of calcium-magnesium-aluminium-silicate (CMAS) corrosion remains a longstanding challenge for thermal barrier coatings application. Here, we address this issue by designing an Al_2O_3 aerogel-modified EB-PVD TBCs structure. The ceramic aerogel serving as a melt trap and Al reservoir, enables fast crystallization of CMAS melt at the infiltration front, which thereby suppresses melt injection and prevents TBCs structure from thermochemical degradation. In addition, the Al_2O_3 aerogel-modified TBCs structure is customizable to withstand different corrosion intensities. The superior CMAS resistance, along with enhanced sintering resistance, confers this novel architecture with robust stability in a corrosive environment. This work provides a design pathway for novel TBCs architecture with excellent resistance toward both CMAS corrosion and sintering.

(ICACC-P010-2023) High-temperature oxidation resistance of polymer-derived CMCs

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The feasibility of coating commercial carbon fibers with polysilazane preceramic polymers are demonstrated. Pyrolysis of functionalized C-fibers leads to the formation of polymer-derived SiCN/CF ceramic matrix composites (CMCs), which was confirmed by SEM and X-ray based spectroscopy. The effect of SiCN content on microstructure and mechanical properties of the composites was investigated. High amount of ceramic yield was observed for the samples prepared in

Ar environment. The high amount of surface oxygen was observed in the ceramic-coated fibers after oxidation test at 800-1500 °C, however all the samples retained ceramic coatings on the surface. The PDC coated fiber mats were observed to be resistant to oxidation resistance until at least 1500 °C. The fracture strength of the uncoated c-fibers was also improved after SiCN deposition. The ultimate bending strength and flexural modulus of the SiCN/CF composites were measured to be 236 MPa and 83 GPa, respectively.

(ICACC-P011-2023) Numerical and experimental studies on the preparation of ceramic coatings by aerosol deposition method

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Aerosol deposition (AD) method is a dry, high-velocity spray coating technique that can be used to deposit dense coatings at room temperature directly from ceramic powders via a room temperature impact consolidation mechanism. Without the necessity of heat treatment, it is capable to retain the original properties of ceramics and metals, and prohibit interfacial material reactions. In this research, the flow characteristics of gas and particle phases inside and outside the nozzle were numerically evaluated using computational fluid dynamics (CFD), which provided a guideline for setting the processing parameters. The particle impact velocity and landing locations which are hard to measure and can severely affect coating formation were computed. The particle impaction process was further simulated to better understand the deposition mechanism. Using AD equipment, Al_2O_3 and ZrO_2 powders were successfully deposited on different substrates, including copper, glass and polymer, for evaluation of their performance in insulating and corrosion prevention. The thickness, roughness and microstructure of these coatings were investigated by various characterisation methods. Further SEM, FIB and TEM tests on coating samples were performed to validate the possible deposition mechanisms derived from the simulation results.

(ICACC-WWP4-2023) Thermal Environmental Barrier Coatings and their Deposits-Induced Degradation

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Core power and efficiencies of gas-turbine engines have increased in recent years. Protective coatings in the engines include thermal barrier coatings (TBCs) and environmental barrier coatings (EBCs). TBCs are porous and have strain-tolerance to accommodate the coefficient of thermal expansion (CTE) mismatch with the substrate. But above all, they have low thermal conductivities to provide insulation to the internally-cooled superalloy. In contrast, EBCs must be dense and have a good CTE match with the underlying CMC component that is not internally cooled. These ceramics all suffer from enhanced deposits-induced, high-temperature degradation. Many deposits are calcia-magnesia-alumina-silicates (CMASs) from natural sources and deposit on the surface of the cearmics. Chemical interaction between the coating and the melt results in the thermo-chemo-mechanical failure. The different strategies that are used to mitigate the CMAS-induced degradation of the coatings will be discussed. Increasing pollution in the atmosphere raises concerns of enhanced degradation of the protective coatings in engines. In addition to CMAS, the atmosphere contains sulfates and sea salt from ocean water. All of these corrodents can melt together and form deposits of new compositions. These combined corrodents likely cause degradation via multiple mechanisms. Recent results from the synergistic degradation will be discussed.

(ICACC-P013-2023) Spatially Resolved Thermal Conductivity Mapping of CMAS Reacted EBCs

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In addition to the high temperatures experienced in gas turbine engines, components must also withstand corrosion to environmental debris including sand, water, and gas combustion products. When reacted with calcium-magnesium aluminosilicate (CMAS), state-of-the-art SiC turbine blades recede and their lifetimes are significantly reduced. To protect the blades from corrosion, environmental barrier coatings (EBCs) are applied, and adhered to the blades with a silicon bond coat. Ytterbium disilicate (YbDS) has become the standard coating material for protecting SiC blades. In these experiments, we use steady-state thermoreflectance (SSTR) to spatially map with micron resolution the thermal conductivity of YbDS EBCs, reacted with CMAS at 1316 °C for 4, 24, and 96 hours. SSTR is an optical pump-probe technique for measuring thermal conductivity of microscale material systems, where a low power "probe" beam monitors the change in reflectivity of a metal transducer on the sample surface in response to a modulated heating "pump" laser with varied power. By applying a mathematical model that relates the applied heat flux to the steady-state temperature rise, we calculate thermal conductivity. We corroborate the observed variations in thermal conductivity with SEM micrographs, that show changes in EBC composition as the YbDS reacts with CMAS, as well as the formation of YbMS due to the high reaction temperatures.

(ICACC-P014-2023) 3D Printing of Anticancer Drug-loaded 3D Scaffolds for Liver Cancer Treatment

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Primary liver cancer is the third leading cause of cancer deaths. Partial or total liver transplantation provides a solution for treating liver cancer but faces donor shortage. Traditional chemotherapeutic drugs through oral or intravenous administration for treating cancer have off-target toxic effects. To overcome these problems, localized environment-responsive chemotherapeutic technologies are attractive. 3D printed hydrogel scaffolds offer advantages for the local administration of anticancer drugs. In this study, natural polymers were chemically modified for 3D printing of hydrogel scaffolds. Both methacrylamidation of gelatin and oxidation of alginate were performed. They were used for 3D printing of hydrogel scaffolds incorporated with an anticancer drug, doxorubicin hydrochloride (DOX). Rheological properties of hydrogels were investigated and their printability was assessed; and printed scaffolds were studied using various techniques. 3D printed scaffolds exhibited high shape fidelity. In vitro drug release experiments showed that the released amount of DOX in pH7.4 buffer was much lower than that in acidic buffer (pH5.4), demonstrating pH-responsive drug release behavior. Using HepG2 cells for culture, results of in vitro biological experiments showed that compared to the control, DOX-loaded scaffolds greatly reduced the survival rate of HepG2 cells, indicating good anticancer effects.

(ICACC-P016-2023) Chemical Imaging of Li-rich Disordered Rocksalt-type Vanadium Oxide Particles Using Hard X-ray Spectroscopic Ptychography

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Li-rich disordered rocksalt (DRS) type cathode materials are promising ones for high-capacity Li-ion batteries. The Li-rich DRS-type materials are often synthesized by mechanical milling (MM) and may have heterogeneous structures with various chemical states. Heterogeneous structures will lead to capacity fade with heterogeneous delithiation reaction. X-ray spectroscopic ptychography, the combination of XAFS spectroscopy and ptychography imaging, can visualize chemical state distribution in the bulk sample with a spatial resolution of several 10 nm. In this study, we performed chemical imaging of Li-rich DRS-type material synthesized by MM before and after delithiation reaction using hard X-ray spectroscopic ptychography, and investigated relationships between the heterogeneous structure and the delithiation reaction at the particle level. We prepared $Li_{10/7}V_{6/7}O_2$ particles using MM and those delithiated by chemical oxidation. X-ray spectroscopic ptychography measurements were performed at BL29XUL in SPring-8. We obtained chemical state maps from reconstructed spatially resolved V K-edge XAFS spectra and phase images. Then, the chemical state maps were processed into clustering analysis to classify multiple domains with different chemical states. We found the presence of domains indicating that the delithiation reaction was suppressed.

(ICACC-P017-2023) Compositional Studies of Spinel-Structured High-Entropy Oxides For Next-Generation Anodes

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High-entropy oxides (HEOs) have been studied as potential anodes in next-generation lithium-ion batteries (LIBs). Composed of equimolar amounts of five or more metal oxides, HEO systems offer compositional flexibility that allows tuning of system properties. HEO systems with both rock salt and spinel structures have been shown as conversion-type anodes with high discharge capacities (> 550 mAh/g) and good stability. A major concern with these systems is the unsustainable use of Co as a redox-active component. In this presentation, we will explore several approaches to removing Co from the $(Ni_{0.2}Co_{0.2}Mn_{0.2}Fe_{0.2}Ti_{0.2})_3O_4$ HEO system, using both redox-active (Cu, Zn, Cu/Zn mixture) and inactive metals (Mg, Ca, Al). HEO nanopowders were synthesized using a liquid-feed flame spray pyrolysis (LFFSP) system. The resulting powders offer submicron sized particles that are atomically mixed at these length scales. When replacing Co with redox-active metals, anodes with discharge capacities of > 600 mAh/g were produced, and when paired with a comparative economic analysis, were found to be a more cost-effective anode system than the original Co-containing system. The system with Al was found to offer superior rate performance to other systems despite a sacrifice in discharge capacities. With this work, we hope to provide a greater picture of the influence of HEO composition on system properties.

(ICACC-P018-2023) Fabrication of $Li_{1.3}Al_{0.3}Ti_{1.7}(PO_4)_3$ Protective Coating Sol of Ni-Rich Cathode Materials for All-Solid-State Lithium Batteries

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Currently, lithium-ion secondary batteries are used in many fields from portable electronic products to electric vehicles (EVs) and power storage systems (ESS). Existing $LiNi_xCo_yMn_zO_2$ (NCM, $x \geq 0.6$) has high discharge capacity and energy density and is widely used as a cathode active material, but its performance is greatly reduced due to low cycling stability and side reaction between electrode/electrolyte. Therefore, many studies are in progress to reduce the interfacial resistance of the Ni-rich cathode materials. Surface modification suppresses the destruction of the crystal structure of NCM and improves the electrochemical performance, and many studies are being conducted. A Li^+ conductor is often used as a coating material, the Li^+ coating can improve the performance of the NCM by suppressing side reactions with the electrolyte and increasing Li^+ diffusion. In this study, $Li_{1.3}Ai_{0.3}Ti_{1.7}(PO_4)_3$ with high ionic conductivity and chemical stability by using sol-gel method was prepared for a coating sol, coated on the NCM. The composite cathode was investigated for crystal structure using XRD, and the LATP coating layer on the NCM were observed through a SEM. LATP was confirmed by EDS and ICP-MS. In addition, the cell performance was evaluated by assembling an all-solid-state battery with a composite cathode/solid electrolyte/aerogel composite anode.

(ICACC-P019-2023) Boosting Li-Ion Rate Capability and Efficiency Stability Enabled by MoS_2 Nanosheets in Polymer-Derived Silicon Oxycarbide Freestanding Electrodes

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Transition metal dichalcogenides (TMDs) such as MoS_2 have continued to generate interest in the engineering community because of their unique layered morphology—the strong in-plane chemical bonding between transition metal atoms sandwiched between two chalcogen atoms and the weak physical attraction between adjacent TMD layers provides them with not only chemical versatility but also a range of properties that can be unlocked upon exfoliation into individual TMD layers. Nonetheless, many issues, including fast capacity decay arising due to volume changes and TMD's degradation reaction with electrolytes at low discharge potentials, have restricted use in commercial batteries. One approach to overcome barriers associated with TMDs' chemical stability functionalization of TMD surfaces by chemically robust precursor-derived ceramics or PDC materials, such as silicon oxycarbide (SiOC). Herein, we report the synthesis of such a composite in which MoS_2 nanosheets are in SiOC matrix in a self-standing fiber mat configuration. Morphology and chemical composition of synthesized material was established by the use of electron microscopy and spectroscopic technique. When tested as LIB electrodes, the SiOC/MoS_2 fiber mats showed improved cycling stability over neat $MoSe_2$ and neat SiOC electrodes.

(ICACC-P020-2023) Electrochemical Performance of Lithium Argyrodite Li_6PS_5Cl Electrolytes Prepared via Wet Milling

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Sulfide-based solid electrolytes such as lithium argyrodite (Li_6PS_5Cl) are typically prepared by mechanical milling and subsequent annealing processes. The lithium argyrodite electrolytes prepared via dry ball milling process exhibit a room temperature (25°C) ionic conductivity exceeding 10^{-3} S/cm. However, the dry ball milling process is long and tedious. In wet ball milling, the precursors are mixed in non-dissolving organic solvents. Wet ball milling offer advantages over dry ball milling in terms of improved homogeneity, shortened duration, and scalability. In this study, Li_6PS_5Cl solid electrolyte powders were synthesized via wet ball milling and post-annealing. Organic solvents such as hexane, heptane, toluene, and xylene are used during the wet milling process. The phase evolution, powder morphology, and electrochemical properties of the wet-milled Li_6PS_5Cl powders and electrolytes are studied. Compared to dry milling, the processing time is significantly reduced via wet milling. The nature of the solvent does not affect the ionic conductivity significantly; however, the electronic conductivity changes noticeably. The study indicates that xylene and toluene can be used for the wet milling to synthesize Li_6PS_5Cl electrolyte powder with low electronic and comparable ionic conductivities.

(ICACC-P021-2023) The solution-based synthesis of Li_6PS_5Cl solid electrolyte for facile lithium ion conduction in the cathode electrode of all-solid-state batteries

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An all-solid-state battery (ASSB) using a sulfide-based solid electrolyte is advantageous for the extended driving range and thermal safety of the transportation. However, in order to ultimately use ASSB as energy storage devices for the transportation, more research is needed on solid electrolytes (SE), which are core materials directly related to battery performance. Among the properties required for a sulfide-based SE, there are typically high ionic conductivity, stability in a wide potential window, and electrochemical stability to lithium metal anodes. Good continuous adhesion due to its mechanical properties is very important for effective lithium ion conduction and reduction of cell resistance. In this study, the particle size of the SE prepared by the liquid-phase synthesis process, which is advantageous for mass synthesis, can be effectively controlled and a new process is proposed. In addition, based on the understanding of solutionization in the liquid phase synthesis process, it was possible to obtain a SE having a crystal structure with reduced impurities. When the SE with improved particle size and purity was introduced into the ASSB, it was confirmed that the interfacial formation state in the positive electrode was improved, and the cell performance was improved.

(ICACC-P023-2023) Superhydrophobic IP-PDMS micro-hoodoo arrays fabricated by two-photon polymerization direct laser writing

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Superhydrophobic surfaces have an apparent contact angle (CA) with water >150 deg and a CA hysteresis <10 deg. Such surfaces occur naturally in e.g. lotus leaves and often have up to six levels of structure hierarchy. Engineered superhydrophobic surfaces often have only one or two hierarchical levels. For example, nanograss textures are known to increase the CA with water. Periodic arrays of mushroom-like structures, also known as micro- or nano-hoodoos, can be engineered to display superomniphobic properties, i.e. to repel both water and oily liquids. Such structures have been previously fabricated by two-photon polymerization direct laser writing followed by surface modification or replication. Here, we present the fabrication and characterization of superhydrophobic microarrays which consist of micro-hoodoo structures arranged in a hexagonal lattice. Furthermore, we report the surface wettability in relation to the size of individual microstructures and the lattice constant, i.e. pitch. Tightly packed micro-hoodoo surfaces, i.e. with a top diameter of 30 μm, and a pitch of 100 μm, were superhydrophobic, with a CA ~160 deg and a CA hysteresis of ~5 deg. Increasing the pitch led to a decrease of the CA. To the best of our knowledge, this work is the first demonstration of micro-hoodoo structures fabricated by a single step process, i.e. direct laser writing of PDMS.

(ICACC-WWP3-2023) Lead-free (1-x)$BaZr_{0.08}Ti_{0.92}O_3$/(x)$CoFe_2O_4$ nanocomposite obtained by SPS

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Obtaining nanostructured composite materials with chemical integrity of their phases has received great scientific and technological importance in recent years, mainly when we refer to composites with enormous potential for designing lead-free magnetoelectric devices. In this scenario, Spark Plasma Sintering (SPS) appears to have high applicability for obtaining dense ceramics and retaining grain size due to the type of heating by the pulsed electric current, using high heating rates, and sintering under pressure. Therefore, the present study focuses on obtaining lead-free nanocomposites (1-x)$BaZr_{0.08}Ti_{0.92}O_3$/(x)$CoFe_2O_4$ (BZT/CFO)($0.0\leq x\leq 1.0$) and the influence of sintering by SPS on the physical and microstructural properties. Accordingly, nanopowders of BZT/CFO composites obtained in situ by the novel one pot Pechini assisted by microwave method were used. After sintering by SPS, high densification levels (above 93%) and chemical integrity of the constituent phases were achieved and analyzed by XRD, EDS, and EDXRF. Regarding the microstructures, analyzed by SEM, it was possible to observe a homogeneous distribution with a low degree of percolation and high dispersion of the BZT and CFO phases in all nanocomposites, in addition to the retention of the average grain size, below 300 nm, providing the efficiency of SPS sintering in obtaining lead-free magnetoelectric nanocomposites.

(ICACC-P025-2023) Boron – challenging and exciting element

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Elemental Boron is a very fascinating element due to its unique structure and its polymorphism. It occurs in several allotropic forms; most are quite difficult to access. Its complex chemistry makes preparation of pure boron very demanding and requires well-designed production processes determined by its increased reactivity at high temperatures. Crystalline boron is used in permanent magnets (Nd-Fe-B), refractory boride synthesis, sputtering targets, or neutron shielding in nuclear applications, whereas larger volumes of amorphous boron are required in pyrotechnics (airbag igniters, flares, propellants), explosives, and advanced ceramics. An increasing demand for purer and finer qualities is the challenge for today's manufacturers. Höganäs Germany, a renowned boron producer since more than 60 years has accepted that challenge and took the opportunity to further improve their broad boron portfolio. Based on a state-of-the-art production facility, progress has been made to optimize lot to lot consistency and to reach an outstanding quality level. The smart combination of preparation and purification techniques available from our versatile technology platform enabled for sustainable product improvements on some distinct grades. Latest results of chemical and physical powder characterization will be discussed, with an emphasis on the improvements achieved and their potential benefits on the use of those products.

(ICACC-P026-2023) Structure and properties of spark plasma sintered bulk alumina ceramics with graded microstructure

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Alumina (Al_2O_3) is of interest for use in parts in which high mechanical strength, hardness, and electrical insulation are required, however, its brittleness can limit its applications. Introducing graded microstructure in materials can tailor traditional properties and give rise to specific functionalities. We fabricated a bulk Al_2O_3 with graded microstructure by spark plasma sintering (SPS), by packing layers of 1000, 500, 200, 80, 200, 500, and 1000 nm Al_2O_3 powder particles into a graphite die and sintered for 2 min at 1600 °C, obtaining a relative density of 99.6%. The grain size of 1000, 500, 200, and 80 nm layers after SPS was 75±50 μm, 54±36 μm, 19±9 μm, and 10±4 μm, respectively. The hardness was measured in each layer, obtaining 22.1±1.6 GPa and 17.5±2.5GPa for the middle layer and the outermost layer, and young's modulus of 432 and 281 GPa respectively, showing a trend in improving the mechanical properties for each layer when decreasing grain size. Other properties such as compression resistance and insulation properties, for example, are still to be explored to understand graded microstructure influence on Al_2O_3, however, the manufacturing method for sintering a bulk Al_2O_3 grain size gradient was successfully described by using the SPS technique obtaining a potential new graded material and a new outcome of ceramics engineering.

(ICACC-P028-2023) Correlation between uniaxial pressing and initial sintering stage for YSZ8

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Ceramic Science brings challenges in the processing step. In an overview, it is possible to understand pressing and sintering as transformations that involve energy exchange. The literature indicates that uniaxial pressure has not a linear correlation with the green density. It is found at a transition point where there is an abrupt increase in density. It is expected that energy exchange during the uniaxial process influences pre-sintering and initial stage sintering characterization. This work analyzes uniaxial pressing and densification behavior for YSZ8. The ceramic powders were prepared by milling for 5 hours with 1%wt. of PVB as binder. The deagglomerated powders were compacted on a cylindrical die and uniaxially pressed from 5 to 12.5 MPa. The pellets were sintered for 4 hours at 600°C and 900°C aiming for a systematic correlation between pressing parameters and densification in the initial stage of sintering. Further analysis has been performed with X-ray radiation for structural properties. The curve green density vs uniaxial pressure shows a transition behavior between the initial and intermediate stage of compactation close to 7 MPa. It was possible to identify greater densification in high temperatures and pressure as expected. The density, sintering temperature, and pressure correlation indicate an improvement in sinterability by pressure values close to the transition point.

(ICACC-P029-2023) Effect of sintering pressure on the synthesis of superhard tungsten tetraboride by SPS

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Tungsten tetraboride-WB_4 or debated as tungsten triboride-WB_3 has been of interest as a candidate for inexpensive superhard material(1). Despite various inconsistent perspectives concerning the microstructure of this compound in the theoretical studies, it has been reported to be synthesized by numerous methods such as arc-melting, hot-press, and spark plasma sintering (SPS)(2,3,4). Among them, SPS has been employed to obtain various types of ceramics with high controllability of microstructure, which can additionally enhance the Vickers hardness by grain boundary strengthening. Variation of content ratios and sintering temperatures has been investigated to acquire desirable compositions and phases. However, the effect of sintering pressure has never been reported in previous literatures. Herein, we attempt to sinter dense pellets of tungsten borides by SPS at various W:B ratios, sintering temperatures, and sintering pressures. The combined effects of the above factors on acquired phases, density, and Vickers hardness are reported. The results show that the enhancement of sintering pressure provides a dense pellet of tungsten tetraboride without decomposing to tungsten diboride-a softer phase.

(ICACC-P030-2023) Synthesis and Characterization of Soybean Feestock-based Composite Systems

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Biomass has emerged as an imporant resource for materials synthesis. Soybean, for example, is an important crop in North Dakota. Soybean is am important feedstock for processing oil from the product. Soybean meal (SM) is referred to as the residue after the extraction process. Currently, SM is used as a animal feed. Clearly, the valorization of SM will give additional revenue streams to farmers and other stakeholders. In this poster presentation, we will present synthesis and characterization of novel materials designed from SM.

(ICACC-P031-2023) Co-sputtered W/Fe interlayers for joining tungsten to steel

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Tungsten has been selected as a structural material in the DEMO fusion power plant as refractory, low activation and high Z material. However, its use is restricted to the high temperature parts which had to be connected to the low temperature parts made of the reduced activation ferritic/martensitic steel EUROFER97. The joint of these materials is very difficult because of several reasons. Despite fairly low temperatures of up to 600°C, the different coefficients of thermal expansion of tungsten and steel cause the formation of excessive stresses during cooling from the in-service to room temperature, which may cause premature de-bonding of the joint. This work reports about the preparation of W/Fe co-sputtered layers between tungsten and steel to obtain graded interlayers able to minimize CTE mismatch, then subsequently joined to steel by hot pressing. SEM imaging, SEM-EDS compositional analysis, XPS, HR-TEM have been used to characterize the W/Fe co-sputtered layer. W tiles co-sputtered with the W/Fe layers have been hot pressed to steel tiles: morphology and mechanical strength (Lap-shear tests in compression at room temperature) have been investigated and compared for joints with and without Fe/W co-sputtered layer. High heat flux tests on both kind of joints have been performed to assess their behaviour against cyclic thermal loads.

(ICACC-P109-2023) Preparation of High Entropy Titanite and Zirconate Pyrochlores and Thermodynamic/Radiation Stability Study

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This study is intended to examine the efficacy of a new class of oxides for radwaste storage, specifically the recently conceived high-entropy oxide (HEO) pyrochlore. The objective is to develop an advanced nuclear waste form capable of immobilizing multiple dissimilar and long-lived radionuclides. We have established a consistent method to fabricate high entropy titanite and zirconate pyrochlores. Conventional solid state sintering and sparking plasm sintering are used to synthesize HEO pyrochlores with various compositions. Through trial and error, we are able to develop a method that consistently produced samples with a pyrochlore structure. This consistency is necessary to conduct further stability studies on thermodynamic and radiation damage on the structure to assess its viability for future use in nuclear waste forms. To investigate thermophysical and thermodynamic properties of HEO pyrochlores, the derived enthalpies of formation of pyrochlore phases using melt solution calorimetry, will suggest whether formation is thermodynamically favorable. In this study, ion beam irradiation (600 keV Ar ion) is also used to produce displacement damage and simulate alpha decay damage from self-radiation damage in nuclear waste storage.

(ICACC-P032-2023) Manufacturing of ceramic structure by a newly developed extrusion 3D printer with optimized ceramic pastes

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In this work, various 3D ceramic-based structure was produced via a newly developed extrusion-based three-dimensional (3D) printer as well as optimized ceramic pastes. The 3D printer was designed to be fully integrated and scalable with reasonable price. Several ceramic pastes for the 3D printer were also developed with different powder sizes, solid contents, and additives. For example, alumina-based paste was prepared with Methyle Cellulose and Polyethylene glycol as binder/surfactant. After printing, the printed pieces were sintered at different temperatures, and then dimensional stability tests were conducted to know the contraction behavior during sintering process. An alumina-based sintered part with 99.7% of relative density showed well-defined α-Al_2O_3 phase and more than 340 MPa of bending strength. The effect of the powder size, solid content and additives of the ceramic paste on the sintered product was also investigated systematically.

(ICACC-P033-2023) Conformal Additive Manufacturing of Zirconia and its Application

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Zirconia is a ceramic material which is prominently used for power generation, electrical applications and in bioengineering as it has better thermal insulation, electrical insulation, wear resistance, temperature strength and corrosion resistance than other ceramic materials. It also has extremely high melting point but this along with its brittle nature makes Additive Manufacturing (AM) of zirconia difficult. AM is a manufacturing process of making parts by depositing materials generally layers by layers onto a horizontal plane by using slices from a 3D model. In this study, we have presented a multi-step AM process of highly dense zirconia using a piston-based material extrusion method. Unlike conventional planar AM process, which slices the 3D model into horizontal layers and deposits material, we have presented a conformal AM process. A conformal AM is a 3D printing approach which slices 3D model into layers based on substrate's surface topology and deposits materials layer upon layer. This approach was then used to 3D print structures on a freeform surface (cylindraical, spherical and others) with dynamically changing z-values based on substrate's surface topology. This AM method accounts for better surface finish, increased mechanical properties with enhanced interconnection between layers in comparision with Planar AM approach. Several fabricated samples through conformal AM method were introduced.

(ICACC-P034-2023) Direct Ink Writing (DIW) of log-pile structures based on $BaTiO_3$

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Usually, ceramics are fabricated and consoidated by sintering powder compacts, but structures with complex geometries are difficult to produce due to limitations in the mould design. The solution to these problems came up with the evolution of Additive Manufacturing (AM). Additive manufacturing has the potential to fabricate complex parts with near net shape which seems to be impossible by the conventional methods available (especially for ceramics). Our interest falls particularly in Direct ink writing/Robocasting of $BaTiO_3$ which is one of the most widely used functional ceramics that finds its application in positive temperature coefficient (PTC) resistors, multilayer ceramic capacitors (MLCC), ferroelectric memories and dielectric capacitors due to its excellent ferroelectric, dielectric and piezoelectric properties. Ink with very high solid content was optimized with desirable rheological properties so that it can be extruded through plastic tips with a diameter of 0.25 mm or more. Log-pile structures were printed using a commercially available 3D printing machine (Delta Wasp 2040 Turbo 2). Thereafter, the green body was kept at room temperature for few hours before it was ready to be sintered. Several sintering strategies has been used to sinter crack-free, fully dense components. Furthermore, electrical properties will be evaluated for energy-harvesting applications.

(ICACC-P035-2023) In-situ Self-Healing and Self-Glazing Geopolymer Composites for high temperature Applications

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This study investigates in-situ self-healing potential of geopolymer, specifically, the impact of various cations (sodium and potassium) on the physical evolution of geo-polymeric materials. Geopolymer are prepared by mixing metakaolin and alkaline solution (water glass-WG) as an aqueous solution of either sodium silicate and/or potassium silicate. The geopolymer of composition $1M_2O.1Al_2O_3.4SiO_2.11H_2O$ which M could be Na, K, or mixture of both in determined ratios. Five different geopolymer samples were made containing (i) 100 wt% Na WG, (ii) 75 wt% Na and 25 wt% K WG, (iii) 50 wt% Na and 50 wt% K WG, (iv) 25 wt% Na and 75 wt% K WG and, (v) 100 % K WG with the calculated amount of metakaolin. The prepared samples were heat-treated up to 1000C for 2, 6 and 10 hours to evaluate the degree of crystallization and magnitude of in-situ self-healing and self-glazing. In the case of $K_2O.Al_2O_3.4SiO_2.11H_2O$ leucite starts to crystallize at 950C and melts at 1693C and for sodium $Na_2O.Al_2O_3.4SiO_2.11H_2O$, the geopolymer crystallizes into glass plus nepheline ($Na_2O.Al_2O_3.2SiO_2$) which melts at 700C (after forming at 1000C). The results show the formation of glassy phases which can fill the crack gaps and obstruct the propagation and could make a glazing surface for geopolymer. However, sample (ii) with 6 hours heat-treatment exposure showed the best healing and glazing properties.

(ICACC-P036-2023) In Situ Crystallization of Jadeite from Sodium Geopolymer

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The overall goal of this project is to investigate the synthesis of jadeite $NaAlSi_2O_6$ through the crystallization of geopolymers. Geopolymer synthesis allows for straightforward compositional control through solution chemistry and the solidification reaction can take less than 24 hours. Crystallization of the X-ray amorphous geopolymers has been used to produce various ceramics including silicon carbide, silicon nitride, and SiAlON composites. In this work Na-geopolymer crystallized under high pressure using the Paris-Edinburgh cell and a resistive graphite heater at beamline 16-BM-B at the Advanced Photon Source, Argonne National Laboratory. This combination of equipment with in-situ energy dispersive x-ray diffraction allows the exploration of synthetic conditions that are difficult to create elsewhere. Typical jadeite syntheses require around 1000°C at 3.5 GPa. However, successful synthesis was shown here at temperatures as low as 500°C at 3.5 GPa or 700°C at 1.5 GPa. The ability to monitor the system in situ with the high brilliance of X-ray synchrotron radiation allowed accurate determination of the conditions required to develop the crystalline jadeite phase. Jadeite is one of the hardest minerals, ranking 6.5-7 on the Mohs Hardness Scale. Unlike other minerals with similar hardness, the microstructure of interlocked crystals gives exceptional resistance to fracture and makes jadeite one of the toughest minerals.

(ICACC-P037-2023) Using Recycled Plant Fibers in Geopolymer Matrix for Thermal Insulation in Buildings

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Energy required to regulate indoor temperature is a pressing concern, especially in recent years of changing climate. Space heating comprises 40% of energy use in typical North American homes. Passive temperature regulation in form of design of space and choice of building materials have been gaining interest to reduce energy consumption. Fiber composites made from locally sourced plants and geopolymer were produced to aid in thermally insulating buildings, as a non-structural material. Geopolymer was formed by high shearing of metakaolin and solution of an alkali silicate. Three widely available commercially cultivated plants – Cannabis sativa, Miscanthus sinensis, and Zea mays – were used in varying percentages, with a batch of fibers having gone through alkali treatment while the other batch was left as is. Fibers were collected from non-edible portions of the plants after harvest. Resulting samples were tested for mechanical strength and thermal conductivity. In comparison to common insulation materials, geopolymers with plant fibers were similar in thermal conductivity while lower in consumption of energy during manufacture, and posed less health risk during installation of the material into a built space. Properties showcased by geopolymerized plant fibers and ease in production makes it a safer and economic alternative to the existing thermal insulation materials used in buildings.

(ICACC-P038-2023) Geopolymer cements made of construction and demolition waste

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This research presents a geopolymer cement developed with construction and demolition waste, prepared with sodium hydroxide and sodium silicate as activators. The main goal is to produce a solution that reduces pollution related to the construction and demolition wastes at Colombia. Diverse sample formulations were manufactured, and compressive strength, density, scanning electron microscopy, and x-ray diffraction characterization tests were included in this research in order to understand the structure-property relations of the developed formulations.

(ICACC-P039-2023) Production and use of cellulosic nanofibers from Guadua spp to reinforce cement composites

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In this work we studied Guadua spp, popularly known as bamboo. The native bamboo was crushed and subjected to two types of cellulosic pulping, after which the pulp was bleached by chemical methods. We also produced samples of mortar with different levels of nanofiber addition and compared the results of physical and mechanical tests of the different batch compositions studied. Cellulosic bamboo nanofibers were evaluated for the effects of their addition on the mechanical and physical properties of the mortar. The pulp was characterized by analysis of cellulose content, infrared spectroscopy-FTIR, X-ray diffraction-XRD, thermogravimetry and transmission electron microscopy-TEM. Mortar samples were molded with 6 different levels of nanofiber addition, determining their water absorption, void ratio, specific mass, compressive strength, and tensile strength. Chemical analyses defined the best pulping and bleaching method, with the highest final cellulose content and transmission electron microscopy confirmed the morphology of the nanometric dimensions of the fibers. The results showed good characteristics of bamboo nanofiber for use as reinforcement of cementitious composites. Based on the results, the nanostructured material has the potential to be applied in civil engineering.

(ICACC-P040-2023) Solid Wastes Utilization in Construction Materials

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The cost of construction materials is increasing day by day because of high demand, scarcity of raw materials, and high price of energy. From the standpoint of energy saving and conservation of natural resources, the use of alternative constituents in construction materials is now a global concern. For this, the extensive research and development works towards exploring new ingredients are required for producing sustainable and environment friendly construction materials. Growth of population, increasing urbanization, and rising standards of living due to technological innovations have contributed to increase the quantity of a variety of solid wastes generated by industrial, mining, domestic and agricultural activities. Moreover, the recycling of solid wastes in civil engineering applications has undergone considerable development over a very long time. The utilization of fly ash, blast furnace slag, phosphogypsum, recycled aggregates, red mud, Kraft pulp production residue, waste tea, etc., in construction materials shows some examples of the success of research in this area. The present review investigates the potential use of various solid wastes in the production of construction materials.

(ICACC-P041-2023) Characterization of Bacillus sp. HK-1, Healing Concrete Cracks

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The bacteria-based self-healing method is one of the effective techniques for closing concrete cracks. The mechanism induces various solid forms based on calcium by enzymes secreted microbially. Sporosarcina pasteurii is well known, forming calcium carbonate even in tough conditions. Nevertheless, searching for a more robust strain is still attractive because the bacteria should survive even in harsh conditions such as high Alkaline, low water activity, long-term, and high temporal Temperature. In this study, we isolated bacteria newly from Calthemite growing slowly below the ceiling of the apartment basement. Urea was the selection pressure to isolate target bacteria, and colonies were yellowish and round. The new strain was designed as Bacillus sp. HK-1 and physiological characteristics were evaluated using experimental protocols forming calcium carbonate. Urease production was evaluated via colorimetric methods and solidified carbonate was observed. The isolated species HK-1 was applied to the cracked concrete and observed for 90 days. As a result, the gap between cracks was solidified and filled with white materials. The isolated strain is expected to help reduce the maintenance cost of the building materials and allow them to be used for a more extended period, thus helping to respond to carbon neutrality positively.

(ICACC-P042-2023) Effect of curing temperature on the resilience of marginal excavation, construction and demolition waste mixed with hydraulic cement

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Using marginal demolition and construction residues in the form of excavation clays and tailings, the aim of this research is to determine the relationship between the curing temperature and the resilience of a soil formulation combined with hydraulic cement. The resilient modulus (AASHTO T307-99) of cylinders was measured in three clayey tailings mixed with cement, cured at different controlled temperatures: 10°C, 25°C and 40°C. We then correlate the effect of curing temperature on the resilience of the materials. Likewise, the aim is to identify variables that affect the results such as plasticity, thermal conductivity, fatigue, compressive strength and indirect tensile strength at 7 days of age.

(ICACC-P043-2023) Addition Effect of Recycled Concrete on Permeable Concrete

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This paper investigates the physical and mechanical properties of pervious concrete using shrinkage and demolition waste, more specifically concrete block waste, as coarse aggregate. The mechanical performance and permeability of pervious concrete are shown with respect to the volume fraction of recycled and natural aggregate used in each sample, the aggregate particle size, the aggregate to cement ratio, and the water to cement ratio. The replacement percentages of recycled aggregate volume used were: 0%, 50% and 100%. The two nominal maximum diameters used were 9.5mm and 12.7mm, and the two water-cement ratios used were: 0.28 and 0.34. The characterization of the aggregates as well as the proportions of the mixes was conducted in properties such as workability, void content, unit weight, compressive strength, and permeability. Results show very low strengths and very high permeabilities, confirming that mechanical strength decreases with increasing permeability.

(ICACC-P044-2023) Influence of Weathered Polyethylene Terephthalate on Performance of Cement-polymer Composites

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In terms of material-based problems, circular economy-based solutions are a near requirement. This is especially true for two very broadly used, manmade material categories: Portland cement-based concrete; and plastic waste. To date, infrastructure material scientists have had ample success with the utilization of waste plastic in Portland cement-based construction materials (e.g., concrete, mortars, etc.). However, limited research has been conducted on waste plastic as a cement additive. Therefore, this work has sought to understand the role that crystallinity (i.e., weathering) of waste polyethylene terephthalate (PET) has on the resulting mechanical performance of the cement matrix when waste PET is utilized as a cement additive. Waste PET was modified via: artificial weathering using the one-of-a-kind NIST SPHERE; and micronization using cryogenic grinding. At each modification step, the structures of various waste PET were characterized via differential scanning calorimetry and Fourier-transform infrared spectroscopy. After grinding, particle characterization was performed. Lastly, compression testing was used to evaluate the effect of PET inclusion in the cement-polymer composite matrix. Overall, the results of this work will help to provide the broader construction materials community an indication on the viability of waste plastic, specifically PET, as a cement additive.

(ICACC-P045-2023) Effect of WC on the Microstructure and Properties of Zirconium Diboride

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Tungsten carbide (WC) additions, up to 5 wt%, were added to commercial zirconium diboride (ZrB_2) powder. Densification was promoted by the addition of 0.5 wt% carbon. The powders with WC additions were hot-pressed at 2150ºC with a pressure of 32 MPa. Bulk densities were measured by the Archimedes method. Scanning electron microscopy was used to determine grain size and morphology, as well as whether any second phases were present. The crystalline phases present were determined by x-ray diffraction. Properties, including hardness, elastic modulus, strength, and thermal conductivity, were measured and will be discussed.

(ICACC-P046-2023) Comparison of Oxidation Behavior of C_f / MC-SiC (with M = Hf, Zr) Composites in an Oxyacetylene Torch and in an Arc Plasma Torch

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The rapid development of space vehicles needs the elaboration of a new kind of materials able to resist to extreme conditions. For thermal-structural materials, the most severe conditions take place in atmospheric reentry vehicles and/or combustion chambers. Those two environments lead to the occurence of several physical and chemical reactions due to the high temperature, high flux and oxidation phenomena. One of the best candidates is the Ceramic Matrix Composites (CMC) especially CMC with Ultra High Temperature Ceramic (UHTC) as matrix. This kind of materials presents many advantages. The melting temperature of UHTC like TaC, HfB_2, HfC or ZrC is above 2500 °C. Moreover, they exhibit high hardness, and good ablation resistance. In this study, C_f / HfC – SiC and C_f / ZrC – SiC composites are elaborated by Reactive Melt Infiltration (RMI) process using hafnium and zirconium silicide (HfSi2 and ZrSi2). Samples are exposed to severe conditions using an Oxyacetylene Torch and an Arc Plasma Torch. Temperatures higher than 2000°C are reached. Post-mortem microstructural analysis (Electron Probe Micro Analysis and SEM/EDS) allows to compare sample degradation and to understand oxidation mechanisms.

(ICACC-P047-2023) Enhanced high temperature stability of UHTC modified C_f/SiC composite fabricated by liquid silicon infiltration

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Ultra-High Temperature Ceramic (UHTC) is a promising refractory material for aerospace fields such as hypersonic vehicle and weapons. The UHTC exhibits high performance against oxidation and ablation due to its high melting temperature. The carbide and boride ceramics and their composites are well-known as UHTC and there are many researches on them to achieve outstanding stability against the harsh environment of thrusting system. However, the single component UHTC has a weakness on thermal shock that induces micro-cracks. In this study, the carbon fiber reinforced silicon carbide ceramic matrix which is light and has high mechanical strength is introduced to overcome the drawbacks. Liquid silicon infiltration process is a facile method to fabricate C_f/SiC composite. Furthermore, UHTC powder was added in C_f/SiC matrix to prevent oxidation of carbon fiber and reduce the amount of residual silicon which was left by liquid silicon infiltration method. The stability of C_f/SiC-UHTC composite has been tested under an oxyacetylene torch flame for up to 5 minutes. The oxidized surface and microstructure of the composite were characterized by 3D scanning, XRD and SEM.

(ICACC-P049-2023) Infiltration of Porous Ultra-High Temperature Ceramics for Active Cooling

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Aliya Kaplan and Carolina Tallon Department of Materials Science and Engineering Advanced Manufacturing Team Virginia Polytechnic Institute and State University Blacksburg, Virginia, 24061 Space and hypersonic travel produce extreme thermal and ablative conditions on different vehicle components which most materials cannot survive. Ultra-high temperature ceramics (UHTCs) are unique in their high melting temperatures, thermal resistance, and mechanical strength making them suitable candidate materials for some of the key components of hypersonic vehicles. However, as a result of restrictions in the manufacturing of UHTCs, most of the current state-of-the-art thermal protection systems have relied on passive heat management strategies, such as heat sinks or sacrificial materials. Recent advancements in the manufacturing of UHTCS have been made such that they can now be formed into complex geometries and intricate microstructures and architectures, so they can support new active cooling approaches. This research proposes a novel active cooling approach involving the infiltration of aligned porous UHTCs formed via ice-templating to provide several heat transfer mechanisms during extreme thermal flight conditions. The heat transfer during the process will be quantified and changes to the microstructure of these materials as a result of the infiltration will be characterized.

(ICACC-P050-2023) $Ti_3C_2T_x$ MXene-Zirconium Diboride Based Spark Plasma Sintered Ultra-High Temperature Ceramic Composites

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$Ti_3C_2T_x$ MXene is a two-dimensional (2D), solution-processable, high aspect ratio, with reported stiffness (333 GPa) highest among solution-processable 2D materials. In this project, we have investigated a one-pot, surfactant-free, aqueous mixing method to develop homogeneous ZrB_2-Ti_3C_2 MXene green bodies and evaluated their sintering behavior. We present the phase transformation of Ti_3C_2 MXene to TiC_y at the grain boundaries, the interface interactions between the ZrB_2-TiC_y grains and their densification mechanism. A nominal relative density of ~96% is achieved when 0.5 wt.% Ti_3C_2 MXene in ZrB_2 was sintered at 1900C with 50 MPa pressure in inert atmospheres. We discuss the intermediate phase formation and evolution and their effect on the mechanical properties of the resulting UHTCs. This study lays the groundwork for 2D MXenes as compatible materials for UHTC applications that can be used as precursors for carbides in ultra-high temperature applications such as hypersonics, nuclear, and extraterrestrial travel

(ICACC-P051-2023) Chemical Vapor Deposition of Phase-Pure Uranium Dioxide Thin Films from Uranium(IV) Amidate Precursors

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Homoleptic uranium(IV) amidate complexes have been synthesized and applied as singlesource molecular precursors for the chemical vapor deposition of UO_2 thin films. These precursors decompose by alkene elimination to give highly crystalline phase-pure UO_2 films with an unusual branched heterostructure.

(ICACC-P052-2023) Highly efficient theranostic nanocarriers with a dual therapeutic approach against triple-negative breast cancer

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Up to date, triple-negative breast cancer is responsible for more than 15 % of new breast cancer cases per year. Lacking hormone expression of targeting receptors, TNBC shows aggressive heterogenous tumor biology with a high risk of reoccurrence and metastasis and, therefore, therapeutic options for patients are limited. In this study, a targeted dual-purpose therapeutic nanocarrier is developed by integrating complementary functionalities and payloads into one nanocarrier through covalent conjugation strategies. The nanocarriers demonstrated an outstanding radiochemical yield of > 98 % and serum stability in human serum during 3 h (^{68}Ga) and 120 h (^{177}Lu). In vitro studies using 3 different TNBC cell lines with FA-DOTA@ $mSiO_2$ carriers demonstrated a 43% cell internalization suggesting specific targeting of folate receptor in TNBC cells. Apoptosis assays showed combined delivery of radiations and DOX induced significant cell death to TNBC cells earlier at different time points. Our data suggests that dual-functions nanocarriers has great potential in targeted delivery of radiations and drug doses for TNBC tumor treatment and points out further studies to evaluate its preclinical therapeutic efficacy.

(ICACC-WWP1-2023) Enhanced Hardness of Thin-Film High-Entropy Transition Metal Ceramics

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Group 4-6B transition metal carbides, nitrides, and borides possess favorable refractory properties such as high hardness and high melting temperatures, which are useful for applications in advanced armors, cutting tools, and spacecraft thermal protection systems. High entropy transition metal ceramics (TMCs) are multicomponent materials that experience a high degree of configurational entropy compared to binary TMCs, resulting in phase stability with complex chemistries. This results in enhanced thermal and mechanical properties. Physical vapor deposition of thin films through magnetron sputtering provides the ability to kinetically freeze-in high entropy ceramics, providing an efficient synthesis process to explore this vast composition space. This research investigates the ability to enhance the hardness of high entropy TMCs through chemical engineering and nanostructural manipulation.

(ICACC-WWP2-2023) Investigation of Glasses and Glass-Ceramics formed in the Ga_2O_3 – B_2O_3 Binary

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Gallium oxide (β-Ga_2O_3) is an ultrawide bandgap semiconductor material with applications in high powered electronics, sensors, and optoelectronic devices. During Czochralski crystal growth of β-Ga_2O_3, volatilization of components is a critical concern when attempting to control dopant composition and crystal quality. Liquid encapsulation (LEC) with B_2O_3 (a layer of molten B_2O_3 over the Ga_2O_3 melt) is a potential solution. One concern with LEC is that interactions between Ga_2O_3 and B_2O_3 are poorly understood, and B_2O_3 could potentially contaminate the Ga_2O_3 melt. This study examines the Ga_2O_3-B_2O_3 binary series of quenched glasses and heat-treated glass ceramics to illuminate the relationship of Ga_2O_3 and B_2O_3 in a melt. X-ray diffraction (XRD), optical microscopy, Raman microscopy, and other characterization techniques are utilized to understand the resulting glass structures, including phase separation and crystallization. Crystal structures and formation processes are discussed. As such, this study informs the viability of LEC of β-Ga_2O_3.

(ICACC-WWP5-2023) Ammonolysis and Sintering of Complex Nitride Compounds

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Nitride ceramics are of increasing interest for a wide variety of applications, and the unexplored chemical space is enormous. The nitride thin film community has devoted immense efforts to developing new nitride alloys, particularly those with wurtzite or zinc blende structures, but work in powder-derived nitride ceramic alloys and compounds remains limited. We are specifically interested in developing powder-derived ceramics of perovskite nitrides that our group recently demonstrated in thin film form: $LaWN_3$, $CeMoN_3$, and $CeWN_3$. Their refractory and severely oxyphilic nature preclude direct calcination of precursor nitrides or simple nitridation of metallic precursors, so we build on previous work on the formation of transition metal nitrides from precipitation reactions of transition metal chlorides followed by ammonolysis. These prior efforts showed nitridation reactions from metallic ions in polymeric hydrocarbons lower reaction temperatures and time to form ceramic powders compared to use of binary precursors. However, prior work has explored limited chemistries for ternary nitrides using this synthesis approach. In this work, an ammonolysis reactor is used to study the synthesis and sintering of complex nitride compounds. We explore the extensibility of this technique to other chemistries with a focus on transition metal and lanthanide series cations.

(ICACC-WWP6-2023) Magnetic Field-assisted Chemical Vapor Deposition of $MgFe_2O_4$ Films for Photoelectrochemical Water Splitting

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Single-phase magnesium ferrite films ($MgFe_2O_4$) were grown by magnetic field-assisted chemical vapor deposition (CVD) of mixed-metal precursor $[MgFe_2(O^tBu)_8]$ as a function of the applied field strength (B = 0.0, 0.5 and 1.0 T). The formation of monophase $MgFe_2O_4$ deposits was confirmed by X-ray diffraction and photoelectron spectroscopy. The cross-sectional analysis (FIB-SEM) of the film revealed an increased densification and crystal growth, upon application of the magnetic field when compared to zero-field deposition. The $MgFe_2O_4$ films deposited under zero-field and field-assisted conditions were used as electrodes in a photoelectrochemical (PEC) water-splitting reaction. All the three samples showed a stable performance and photocurrent values, however, the photocurrent was found to gradually decrease with increasing applied magnetic field (0 T: 5.74 $\mu A/cm^2$, 0.5 T 2.33 $\mu A/cm^{2,}$ and 1 T: 1.33 $\mu A/cm^2$ at 1.23 V (vs. RHE)), which is possibly due to change in absorption properties and crystal orientation, decreasing photo absorption intensity provided by the UV-vis results and the latter being evident in the disappearance of (220) peak in $MgFe_2O_4$ films grown under the influence of the external magnetic field.

(ICACC-WWP7-2023) 20 years dispersing carbon nanophases into ceramics: Results, problems, and solutions

P. Rivero-Antunez*[1]

1. Universidad de Sevilla, Spain

For decades, low dimensionality carbon nanophases were excellent aspirants for the reinforcement of ceramic matrix composites. However, despite the hugely devoted effort in this field, the effect of carbon nanotubes or graphene as toughening agents is questionable, as reviewed in this work. Recently, novel approaches such as sol-gel and reactive SPS were tested but no major reinforcement was observed. Furthermore, this work suggests major causes of the absence of conclusive results, being the lack of dispersion and homogenization of the carbon one of the most important issue hindering mechanical improvements, as the aggregates act as defects. We performed structural computer modelling to confirm the massive density of reinforcing elements per unit of volume typically used by the researching community and how these aggregates could be revealed by SAXS. Finally, this work suggests that very low and perfectly dispersed carbon contents are required for the achievement of effective reinforcement.

Wednesday, January 25, 2023

Emerging Materials and Sustainable Manufacturing Technologies in a Global Landscape: Symposium in Honor of Dr. Tatsuki Ohji

Tatsuki Ohji Honorary Symposium 3

Room: Coquina Salon D (North Tower)
Session Chairs: Alexander Michaelis, Fraunhofer IKTS;
Palani Balaya, National University of Singapore

8:30 AM

(ICACC-HS-015-2023) Additive Manufacturing of Porous Geopolymers for Environmental Applications (Invited)

P. Colombo*[1]; G. Franchin[1]; M. D'Agostini[1]; M. Muracchioli[1]

1. University of Padova, Industrial Engineering, Italy

Geopolymers possess a chemical composition similar to zeolites, intrinsic micro- and mesoporosity and have ion-exchange capabilities; these features make them highly interesting for environmental applications as sorbents and catalysts for multiple reactions. Moreover, the possibility of room to low temperature consolidation, low CO_2 emissions and the fact that they can be obtained from waste make geopolymers attractive from the processing point of view. Our group has been exploring Direct Ink Writing as an additive manufacturing 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 macro-porosity above 50% vol were fabricated and characterized in terms of microstructure, mechanical properties, specific surface area and permeability. Specific compositions and functionalization protocols were developed for selected applications, including biodiesel production catalysis, CO_2 adsorption, water purification from cationic and anionic pollutants as well as microbes.

9:00 AM

(ICACC-HS-016-2023) A Brief Perspective in Green Manufacturing of Ceramics and Composites (Invited)

S. Gupta*[1]

1. University of North Dakota, Mechanical Engineering, USA

Sustainability has become an important parameter in design considerations. Based on sustainability, circular economy based materials design has become vital for materials development. In this presentation, I will present some of the recent development in my research group in designing of advanced materials. Some examples are greening of ceramics, bioplastics, biomass utilization, lignin-based materials etc. I will present different case studies to illustrate these examples.

9:30 AM

(ICACC-HS-017-2023) Purification Processes Utilizing the Ion-Exchange Reactivity of Nanometric Metal Oxides (Invited)

A. Apblett*[1]; C. Perkins[1]

1. Oklahoma State University, USA

Nanocrystalline calcium zirconate and scheelite, $CaWO_4$, undergo unexpected ion exchange with aqueous heavy metals lanthanides, and actinides, sorbing the dangerous metal ions and releasing benign calcium ions into solution. Selectivity is for larger metal ions over smaller ones and is especially high for oxocations such as uranyl ions. Sorption capacities are extremely high – exceeding 100% by weight in several cases. The novel reagents have been used to remove heavy metals such as lead and cadmium and radionuclides such as strontium and uranium from water, strontium from milk, and calcium from magnesium pharmaceutical solutions. Mining the ocean for uranium is also possible.

10:20 AM

(ICACC-HS-018-2023) Advanced Ceramics and Coatings for Mining, Mineral, Oil & Gas Production and Power Generation (Invited)

E. Medvedovski*[1]

1. Endurance Technologies Inc., Canada

The use of advanced ceramics, composites and coatings in mining, mineral and oil & gas production, refinery and power generation constantly grows owing to the development of advanced materials with improved properties and their technologies according to specific application requirements. Particular demands for advanced materials with high reliability and complex shapes or for coatings on complex shape steel components and long tubing with inner surface protection for a variety of applications require novel and optimized processing. The materials include oxide and non-oxide ceramics, ceramic-ceramic and ceramic-metal composites, coatings on metallic components where functional application properties can be achieved. Some principles of selection and manufacturing of these materials, specifically for wear-corrosion environments, are considered. The examples of successful development and processing of wear, corrosion and thermal shock resistant ceramics, composites (e.g., fine grained materials or materials with specially designed grain sizes and phase distribution) and coatings are discussed. The technology importance on industrial components manufacturing will be demonstrated.

10:50 AM

(ICACC-HS-019-2023) Development and Testing of a Ceramic Heat Exchanger (Invited)

D. Singh*[1]

1. Argonne National Lab, USA

The need for ceramic thermal components, such as heat exchangers, are becoming increasingly important in manufacturing and power generation sectors as application temperatures preclude metals and alloys. In this regard, we have been developing ceramic heat exchangers based on silicon carbide for its its excellent mechanical strength, good chemical resistance, and high thermal conductivity. Additive manufacturing approach was used to fabricate the lab scale prototypes based on the design developed based on conjugate thermal and stress analysis. Heat transfer of the ceramic prototype heat exchanger was experimentally investigated at elevated temperatures up to 550 °C. The experimental data were compared to the predictions from simulation models showing good agreement. This material is based upon work supported by the U.S. Department of Energy's Office of Energy Efficiency and Renewable Energy (EERE) under the Solar Energy Technologies Office Award Number 34240 at Argonne National Laboratory operated under Contract no. DE-AC02-06CH11357 by the UChicago Argonne, LLC.

11:20 AM

(ICACC-HS-020-2023) Recent trends of advanced ceramics industry and Fine Ceramics Roadmap 2050 (Invited)

H. Takemura*[1]

1. Japan Fine Ceramics Association, Japan

Japan's fine ceramics (advanced ceramics) industry, which reached US$30 billion of production in 2018 (an annual growth rate of 6.3%), accounts for 40% of the global market. The mission of the Japan Fine Ceramics Association (JFCA), which consists of 116 corporate members related to fine ceramics is to further promote the development of this industry. In this paper, we describe the recent trends of fine ceramics industry and the latest JFCA's activities including publication of the FC Roadmap 2050 (2021 edition). This roadmap addresses fine ceramics technologies and products that will meet increasingly diverse needs of society, market, and industry in the year 2050.

5th Pacific Rim Engineered Ceramics Summit

5th Pacific Rim Engineered Ceramics Summit V

Room: Coquina Salon B (North Tower)

Session Chairs: Jingyang Wang, Shenyang National Laboratory for Materials Science, Institute of Metal Research, Chinese Academy of Sciences; Yamato Hayashi, Tohoku University

8:30 AM

(ICACC-PACRIM-014-2023) Mechanochemical Synthesis of Fluoride Ion Conducting Ceramics for Electrochemical Applications (Invited)

A. Mineshige*[1]

1. University of Hyogo, Japan

In the present study, fluoride ion conducting behavior in ceramics with fluorite-type and its related structure was reinvestigated in order to think of a strategy for the development of fast ion conductors. Especially, enhancing the ionic conductivity and lowing the activation energy for conduction by using a mechanochemical synthesis was examined. We prepared fluorite-type BaF_2-based fluorides via a mechanical milling process by changing milling media, weight ratios of ball to powder (B/P), and milling time. Obtained electrical conductivities for pure BaF_2 and BaF_2-based fluorides were largely scattered over several orders of magnitude although the chemical compositions are the same. The conductivity was suggested to be enhanced due to the additional introduction of fluorine vacancies and/or interstitials in the lattice during the milling process. Furthermore, by employing the mechanical milling method, we succeeded in obtaining the highest conductive fluoride ion conductor below the freezing point. These BaF_2-based electrolytes are successful candidates for the electrolyte materials in high performance electrochemical devices. This work was supported by RISING2 (JPNP16001) and RISING3 (JPNP21006), commissioned by the New Energy and Industrial Technology Development Organization (NEDO).

9:00 AM

(ICACC-PACRIM-015-2023) Towards developing safe and fast chargeable lithium-ion battery (Invited)

P. Balaya*[1]

1. National University of Singapore, Department of Mechanical Engineering, Singapore

State-of-the-art Li-ion battery technology using graphite anode is not able to meet safety requirements for fast charging of EVs due to micro-shorting by dendrite growth of lithium causing thermal runaway. Battery technology seeks new concepts in materials design to overcome current safety limitations especially during high power operations. Currently, industries provide fast chargeable battery technology using $Li_4Ti_5O_{12}$ coupled with NMC cathode materials assuring safety. However, $Li_4Ti_5O_{12}$ anode has limited storage capacity (170 mAh/g) and expensive. On the other hand, TiO_2 anatase is inexpensive with specific storage capacity of 335 mAh/g. In this talk, we present our work on developing fast chargeable Li-ion battery using mesoporous TiO_2 (meso-TiO_2) coupled with mesoporous $LiMnFePO_4$. Meso-TiO_2 shows enhanced storage capacity of 265 and 107 mAh/g at C/5 and 30C respectively. Mg^{2+} substituted Mn-rich $LiMn_{1-x}Fe_xPO_4$ shows improved storage performance with less polarization, less volume expansion, improved structural and thermal stability and enhanced Li diffusion. We will present synthesis strategies for the above cathode and anode materials, which exhibit unique morphology of micron-size mesoporous secondary particles comprising interconnected primary nanoparticles showing impressive storage and rate performances with long cycle.

9:30 AM

(ICACC-PACRIM-016-2023) Advanced ceramic research in Slovak - Czech cross-border region (Invited)

D. Galusek*[1]

1. Alexander Dubcek University of Trencin, FunGlass, Slovakia

Advanced ceramic industry is virtually non-existent in the Slovak Republic. Situation is better in the Czech republic, with a variety of smaller-scale ceramic operations. This hampers experimental development in both countries. However, fundamental research of ceramic materials has a long tradition in both countries. There are 5 institutes active in the field of ceramics in Slovakia, and in the cross border region in Czech republic. This contribution has an ambition to provide a thorough overview of their activities, and to highlight their achievements. The IIC SAS in Bratislava works in non-oxide ceramics (nitrides, and carbides), with focus on their mechanical properties, UHTC, and nitride-based ceramic luminophores. The Department of Inorganic Materials of the STU in Bratislava focuses on additive manufacturing of oxide ceramics, including bioceramics by FFD. The FunGlass Centre in Trencin has a variety of activities ranging from transparent oxide ceramics, through ceramic composites with eutectic microstructures, to additive manufacturing of ceramics structures, including bioactive scaffolds. The IMR SAS in Kosice is recognized for its contribution to the fundamentals of mechanical properties of ceramics, and high-entropy non-oxide ceramics and ceramic coatings. CEITEC Brno focuses on fundamentals of sintering and adviced sintering methods, such as HIP and SPS in preparation of transparent ceramics.

5th Pacific Rim Engineered Ceramics Summit VI

Room: Coquina Salon B (North Tower)
Session Chairs: Atsushi Mineshige, University of Hyogo; Palani Balaya, National University of Singapore

10:20 AM

(ICACC-PACRIM-017-2023) Directionally solidified high-entropy $RE_3Al_5O_{12}/Al_2O_3$ eutectic with outstanding crystallographic texture formation capability (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

Crystallographic texture control is the critical challenge for directionally solidified eutectic (DSE) ceramics. This lecture will present a high-entropy $RE_3Al_5O_{12}/Al_2O_3$ DSE ceramic that shows outstanding crystallographic texture formation capability. Based on comprehensive observations of phase composition and eutectic microstructure characteristics, the single crystal phases of high-entropy $RE_3Al_5O_{12}$ (RE = rare earth) and Al_2O_3 are obtained within a much-shortened growth distance than those of the single rare earth containing counterparts. The rapid formation of stable crystallographic texture is significant for microstructure control in DSE processing and uniformity of property along the whole material.

10:40 AM

(ICACC-PACRIM-018-2023) SDGs oriented processing of nano metal related materials from ceramics powder (Invited)

Y. Hayashi*[1]

1. Tohoku University, School of Engineering, Japan

In recent years, SDG-oriented processes have been required in materials synthesis. And, a variety of high-value-added technologies as nanotechnology and the high value-added products as nano metal particle related materials using it can be requested various fields. Nanoparticles is one of the most important nano metal related materials because nanoparticle manufacturing is an essential component of nanotechnology. We developed a new nano metal particle related materials synthesis method that achieved by sonochemical solid-liquid processing. A ceramic material, metal oxide, was used as the metal source. Metal oxides are inexpensive, safe, and low emission raw materials because they are composed of oxygen and metal atoms. This new synthesis method is with the ultrasonic as non-equilibrium reactor and the metal oxide and alcohol based solvent are used for the raw material. We have synthesized nano metal related materials by ultrasound in liquid-solid slurry and controlled morphology of products. The metal oxide simply was reduced into metal and morphology of metal nanoparticles was changed by various conditions. This fabrication method is extremely clean and safety processing, so it can also be applied to food and medicines, etc.

11:10 AM

(ICACC-PACRIM-020-2023) Tailoring the structure of electrospun nanofibers for specific functionalities (Invited)

G. Grader*[1]

1. Technion - Israel Institute of Technology, Chemical Engineering, Israel

Ceramic nanofibers with complex structures and surface functionalities are required for improved performance in diverse applications. Fiber architecture offers a built-in directionality as well as reduction of mass transport limitation and accessibility to a large functional surface. Porous or hollow fiber structures provide additional inner surface, opening opportunities for impregnation, and creation of new interfaces within the porous body. Electrospinning is an effective method to produce continuous nanofibers of practically any material. Obtaining the desired morphology in such fibers is the main challenge to be addressed in this presentation. Typically, electrospun nanofibers are synthesized from solutions that contain the pre-ceramic precursors, solvents and polymers. The desired ceramic phase is obtained after drying, polymer removal and precursor's decomposition by thermal treatment. During this stage, fibers shrinkage, deformation, and phase and morphology changes occur. The presentation will focus on creation of nanofibers with diverse surface and inner structures from variety of materials. Formation of lamellar, hollow and belt architectures for specific applications will be discussed.

FS2: Materials for Thermoelectric and Thermionic Energy Conversion

Topological Thermoelectric Materials, Theories and Computations

Room: Flagler A (South Tower)
Session Chairs: Jon Goldsby, NASA Glenn Research Center;
Gabi Schierning, Bielefeld University

9:00 AM

(ICACC-FS2-016-2023) Thermoelectric and topological insulator properties of compacted Bi_2Te_3 nanoparticles with high residual porosity (Invited)

G. Schierning*[1]; S. Izadi[1]; A. Bhattacharya[3]; S. Salloum[2]; J. Han[3]; L. Schnatmann[1]; I. Ennen[1]; A. Hütten[1]; S. Schulz[4]; M. Mittendorff[3]

1. Bielefeld University, Eyperimental Physics, Germany
2. University of Duisburg-Essen, Chemistry, Germany
3. University of Duisburg-Essen, Eyperimental Physics, Germany
4. University of Duisburg-Essen, Chemistry and CENIDE, Germany

For a little more than a decade, we have known that many excellent thermoelectric materials such as bismuth telluride, Bi_2Te_3, belong to the class of topological insulators. These materials are characterized by the existence of charge carriers with special properties such as extremely high mobility at the crystal surfaces. Although nanoparticles are often used especially for the optimization of thermoelectric properties, little is known about the interplay between the topologically protected surface charge carriers and the "normal" charge carriers from the bulk. In this study, we therefore compacted Bi_2Te_3 nanoparticles to produce samples with high residual porosity. These show a pronounced signature of topologically protected surface charge carriers in low-temperature transport, i.e. weak anti-localization. Evaluation of the magnetotransport data with the Hikami-Larkin-Nagaoka model yields coherence lengths of about 200 nm, which is significantly larger than the average grain size in the studied samples. Additional studies using terahertz spectroscopy allow us to quantify the fractions of surface and bulk charge carriers. According to these data, nanoparticulate samples of Bi_2Te_3 still show high proportions of surface charge carriers in transport even at room temperature, which in turn is likely to influence their thermoelectric properties.

9:30 AM

(ICACC-FS2-017-2023) Lower bounds on thermal conductivity and thermal diffusivity from fundamental physical constants

K. Trachenko*[1]

1. Queen Mary University of London, Physics, United Kingdom

I will discuss how the insights from the liquid theory led to the derivation of the lower bounds for kinematic viscosity and thermal diffusivity. Both lower bounds are governed by the fundamental physical constants and correspond to the global minima on the phase diagram involving solids, liquids and gases. This result answers the long-standing question discussed by Purcell and Weisskopf of why viscosity never drops below a certain value. Our result also shows that the same question and answer apply thermal properties and thermal conductivity and thermal diffusivity. This has practical implications including for thermoelectricity, energy conversion and insulation.

9:50 AM

(ICACC-FS2-018-2023) Assessment of Silicon Germanium Proprieties for Direct Energy Conversion Using Density Functional Theory (Invited)

J. C. Goldsby*[1]

1. NASA Glenn Research Center, Chief, Ceramic and Polymer Composites, USA

Radioisotope power systems allow for decades-long spacecraft operation, critical for a deep space mission. Silicon-Germanium (SiGe) based alloys are the traditional thermoelectric materials used in radioisotope power generation. These materials harvest enthalpy from the decay of radioactive sources such as plutonium. Solid-state power conversion devices like thermoelectrics depend solely on their operation's temperature gradients Computational methods offer an efficient and systematic manner to study and design SiGe heritage materials and determine new formulations. SiGe calculations were carried out to determine this study's relevant electronic and mechanical properties. Comparisons are made between predicted and experimental measurements of Seebeck coefficients. The Zacharias and Giustino approach were used to calculate the phonon-induced change of the band structure as a function of temperature. The calculations were carried out using a projector augmented wave (PAW) method using a commercial code MedeA incorporates the Vienna Ab-initio Simulation Package (VASP) as the computational engine. The calculation was based on density functional theory using the GGA-PBE exchange-correlation functional and optimized mesh. This study makes predictions and comparisons between experimental and theoretical data of electrical, structural, and crystallographic properties

S1: Mechanical Behavior and Performance of Ceramics & Composites

Mechanical Testing, and Characterization of Ceramics and Composites

Room: Ballroom 5 (South Tower)
Session Chairs: Ji Yeon Park, Korea Atomic Energy Research Institute; Emmanuel Maillet, GE Research

8:30 AM

(ICACC-S1-026-2023) Fabrication and Characterization of MAX Phase Materials for High Temperature Applications

B. Ma*[1]; P. Chaugule[1]; M. Du[1]; D. Singh[1]

1. Argonne National Laboratory, USA

MAX phase materials exhibiting high thermal conductivity and superior mechanical strength at high temperatures (>1000°C) are promising for many applications, such as solar power receivers for the next generation concentrating solar power (CSP) systems. We fabricated MAX phase Ti_3SiC_2 parts of high phase purity from Ti, Si, and TiC powder mixture by spark plasma sintering (SPS) process. Fracture surface scanning electron microscopy revealed dense homogeneous structure with plate-shaped MAX phase Ti_3SiC_2 grains of 0.5 mm thick and 2 to 5 mm in size. The resulting bulk materials contain a volume fraction of ≈90% Ti_3SiC_2 phase and ≈10% TiC phase as determined by X-ray diffraction analysis. We measured bulk density of 4.56 $g\bullet cm^{-3}$ by Archimedes' principle, which corresponds to 100% of the theoretical value. Flexure strength of 519 ± 32 MPa and Vickers hardness of 6.4 ± 0.8 GPa were observed at room temperature. Thermal conductivity of ≈41 W•m^{-1}•K^{-1} was determined by light flashing analysis. Details of fabrication conditions and characterization results will be presented. This work was supported by the U.S. Department of Energy, Solar Energy Technology Office, under Contract DE-AC02-06CH11357.

8:50 AM

(ICACC-S1-027-2023) Dynamic compression induced phase transitions of ZrW_2O_8, $(Cu_{0.2}Co_{0.2}Mg_{0.2}Ni_{0.2}Zn_{0.2})O$, and $(La_{0.2}Ce_{0.2}Pr_{0.2}Sm_{0.2}Y_{0.2})O_2$

S. Bishop*[1]; D. R. Lowry[1]; A. Peretti[1]; C. Riley[1]; M. Blea-Kirby[1]; J. Park[1]; P. Kalita[1]; M. Knudson[1]; A. Sarracino[1]; S. Murray[1]

1. Sandia National Laboratories, USA

In this presentation, high strain rate and pressure induced phase transitions of ZrW_2O_8 and high entropy oxides (HEOs) of $(Cu_{0.2}Co_{0.2}Mg_{0.2}Ni_{0.2}Zn_{0.2})O$ and $(La_{0.2}Ce_{0.2}Pr_{0.2}Sm_{0.2}Y_{0.2})O_2$ will be discussed. These materials are of interest for thermo-mechanical environmental barriers due to the negative expansion coefficient of ZrW_2O_8 and tunable thermal properties of HEOs. The new facilities of the Dynamic Compression Sector at Argonne National Laboratory enable in situ X-ray diffraction to examine crystallographic structure at high strain rates and pressures using a powder gun flyer to impact the sample target. In this work, phase transitions (e.g., amorphization) under dynamic (ns) conditions of the above materials are shown and the role of metastability of ZrW_2O_8 and $(Cu_{0.2}Co_{0.2}Mg_{0.2}Ni_{0.2}Zn_{0.2})O$ will be discussed, in addition to comparisons with existing quasi-static compression phase transition measurements. Sandia National Laboratories is a multimission laboratory managed and operated by National Technology & Engineering Solutions of Sandia, LLC, a wholly owned subsidiary of Honeywell International Inc., for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525.

9:10 AM

(ICACC-S1-028-2023) Fracture Mechanisms of High Entropy Ceramics at Micro- and Nano-size Level

P. Hvizdoš*[1]

1. Institute of Materials Research, Slovak Academy of Sciences, Slovakia

High entropy materials are interesting class of material systems with, several types of atoms mixed in a single-phase structure. In ceramics, bulk high entropy carbides and/or nitrides are particularly novel materials, where at least four different transition metals are present, usually in equal molar ratios. The whole crystallographic structure is stabilized by the configurational entropy. These materials are being developed mainly as structural materials for extreme environment applications. Recently, single-phase high-entropy carbides have been synthesized with the aim to develop structural materials, which broaden the limited set of ultra-high temperature ceramics (UHTCs). This contribution presents fabrication of a high purity, (Hf-Ta-Zr-Nb)C bulk carbide ceramic system by means of a field assisted sintering technology. The optimized materials were prepared with suitable grain structure. Subsequently, their mechanical properties and fracture behavior were studied at micro- and nano-size levels by means of nano-indentation and strength testing. For the latter, micro-pillars and micro-cantilevers were prepared by focused iom beams techniques at carefuly selected sites. Then compression (micro-pillars) and bending (micro-cantilevers) tests were carried out. Besides the strength data the specimens were fractographically examined and fracture (micro) mechanisms were observed and identified.

9:30 AM

(ICACC-S1-029-2023) Structure, mechanical characteristics and high temperature stability of sintered under high and by hot pressing ZrB_2- and HfB_2– based composites

T. Prikhna*[1]; A. Lokatkina[1]; R. A. Haber[2]; Z. Ayguzer Yasar[2]; P. Barvitskyi[1]; V. Moshchil[1]; M. Karpets[1]; O. Borimskyi[1]

1. Institute for Superhard Materials of the National Academy of Sciences of Ukraine, Ukraine
2. Rutgers, The State University of New Jersey, Materials Science and Engineering, USA

The pure ZrB_2 and HfB_2 and their composites with SiC and Si_3N_4 were densified using pressure of 4.1 GPa at 1800 C as well as hot pressing at 30 MPa. Their crystal structure, mechanical characteristics, and high-temperature stability in a vacuum and in the air were studied and compared. The results showed that the stability in the vacuum of pure ZrB_2 and HfB_2 occurred to be essentially higher than that of the materials with SiC additions. On the other side, their composites demonstrated essentially higher values for mechanical properties. In addition, high-pressure sintered materials manifested better mechanical characteristics compared to hot-pressed materials. The use of high pressures at comparatively low temperatures and short sintering time makes it possible to obtain mentioned materials with improved mechanical properties as compared to all other known methods. The composite material prepared from HfB_2-30 wt.% SiC mixture demonstrated mechanical characteristics (density r=6.21 g/cm^3, microhardness Hv(9.8 N) = 38.1±1.4 GPa, and fracture toughness K_{1C}(9.8 N) = 8.2±0.2, MH×m$^{0.5}$ which are essentially higher than that of pure HfB_2 sintered in the same conditions (r=10.79 g/cm^3, Hv(9.8 N)=21.3±0.84 GPa, K_{1C} (49 N)=7.2±0.9 MH×m$^{0.5}$. Pure HfB_2 demonstrated Young modulus E=984 GPa and poisson ratio μ=0.146.

10:10 AM

(ICACC-S1-030-2023) Method for Weibull analysis of Ball-on-3-Ball strength data with non-uniform specimen sizes

K. Breder*[1]; E. Buchovecky[1]

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

The Ball-on-3-Ball (B3B) test enables strength measurements on small cast ceramic discs in their as-made state. Weibull analysis of the strength data is complicated by variations in the specimen dimensions and the lack of an analytical solution for the 3D stress state. We present a novel analysis method that utilizes finite element analysis (FEA) to determine the effective area and volume of each specimen as a function of the Weibull shape parameter. A Maximum Likelihood Estimator (MLE) optimization procedure is then used to determine the Weibull parameters that best describe the entire data set, fully accounting for differences in the specimen sizes. For validation, alumina discs of 4 mm diameter and 0.4 mm thickness were prepared and tested in a Ball-on-3-Ball fixture. For comparison, A-sized and B-sized flexure bars were prepared from the same alumina billets using similar grinding procedures. The B3B strength data were analyzed using the new FEA-MLE method and the flexure test data were analyzed as per ASTM C1239. At least 30 valid tests were completed for each set. The results showed that scaling between the different flexural samples worked well, and scaling between these and the B3B test could reasonably be performed. The FEA-MLE procedure is now routinely used for B3B analysis and has been extended to other test configurations.

10:30 AM

(ICACC-S1-031-2023) Testing and size scaling of ceramic tubes and tube sectors

E. Buchovecky*[1]; K. Breder[1]

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

Statistical strength data from lab-scale tests was rescaled to predict the strength of long SiC roller tubes (L=2900mm, OD=41.6mm, ID=27.5mm) in service. Test specimens were made by cutting full-length tubes into 100 mm sections then cutting each section lengthwise into four 90° sectors. Each sector specimen was tested in a purpose-built 4-point flexure fixture with spans of 75/37.5 mm. Statistical analysis of the strength data was performed via a method that uses finite element analysis (FEA) to determine effective area and effective volume functions for each specimen then applies maximum likelihood estimator (MLE) optimization to determine the Weibull distribution describing the entire data set. The measured Weibull strength was scaled to predict the equivalent strength of full-size rotating rollers in service for both surface-distributed and volume-distributed flaw populations. During analysis of the FEA results it was found that the contact pressure between the load pin and specimen increases the tensile stress in the specimen directly beneath the load pin. This shifts the location of the maximum stress from the midpoint of the specimen to a point beneath the load pin. The amount of 'stress amplification' due to the load pin depends the specimen thickness and the mismatch between the load pin major diameter and the ID of the specimen. These factors were accounted for in the FEA/MLE Weibull analysis procedure.

10:50 AM

(ICACC-S1-032-2023) Fracture behaviour of alumina-based ceramic architectures with tailored microstructures

J. Schlacher*[1]; Z. Chlup[2]; T. Csanádi[3]; A. Hofer[1]; R. Bermejo[1]

1. Montanuniversitaet Leoben, Department of Materials Science, Austria
2. Czech Academy of Sciences, Institute of Physics of Materials, Czechia
3. Institute of Materials Research, Slovak Academy of Sciences, Slovakia

Mimicking biological materials (i.e. nacre) has been established as a remarkable approach to design multilayer ceramic architectures with improved mechanical reliability. Recent research activities have demonstrated the possibility of embedding layers with tailored in-plane compressive residual stresses within a layered architecture for providing an effective barrier against crack propagation. Moreover, orienting the microstructure (texturing) along the [0001]-direction within the embedded layers can be exploited to further enhance the fracture resistance of layered ceramic designs. In order to assess the potential of microstructure texturing for demanding applications (i.e. high-temperature environment), the fracture behaviour of alumina ceramics with textured microstructures is investigated under uniaxial bending over a wide temperature range (up to 1200°C). The exceptional damage tolerance of these materials at room and elevated temperatures may be mainly attributed to crack deflection events along the weaker basal grain boundaries within the textured microstructure regions. To verify this, the (micro-scale) fracture toughness of individual textured alumina grains and grain boundaries has been measured through micro-cantilever bending tests. These investigations may enhance the understanding of toughening mechanisms acting during the fracture process of novel layered ceramics.

11:10 AM

(ICACC-S1-033-2023) Enhanced contact damage tolerance of multilayer alumina ceramics with tailored microstructures

A. Jabr*[1]; J. Schlacher[2]; A. Hofer[3]; R. Bermejo[4]

1. Montan Universitaet Leoben, Department Materials Science, Austria
2. Montanuniversitaet Leoben, Austria
3. Montanuniversitaet Leoben, Materials Science, Austria
4. Montanuniversitaet Leoben, Institut fuer Struktur-und Funktionskeramik, Austria

Contact damage is a major cause for rejection of ceramic parts. This work demonstrates strategies to enhance contact damage tolerance of alumina ceramics. A bioinspired strategy involves texturing the microstructure to deflect cracks along the elongated grains. Further improvement is achieved by embedding textured layers in a multilayer architecture to induce compressive residual stresses, acting as a "protective shield" against crack propagation. The resistance of such layered alumina system has been investigated under Hertzian contact loading and compared to the corresponding monolithic equiaxed and textured alumina. Acoustic emission detection was used for monitoring the materials response. It was found that a textured microstructure can accommodate the damage below the surface by shear-driven, quasi-plastic deformation instead of the classical Hertzian cone cracking in equiaxed alumina. In the multilayer system, a combination of both mechanisms, namely Hertzian cone cracking on the top surface layer and quasi-plastic deformation within the embedded textured layer, was identified. Further propagation of cone cracks at higher loads was hindered owing to the combined action of the textured microstructure and compressive residual stresses. These findings demonstrate the potential of embedding textured layers as a strategy to enhance the contact damage tolerance of ceramics.

11:30 AM

(ICACC-S1-034-2023) Thermal Expansion of Si_3N_4 via Neutron Diffraction

G. Swift*[1]

1. Southern Illinois University Carbondale, School of Mechanical, Aerospace, and Materials Engineering, USA

Time-of-flight Neutron Diffraction data were used to obtain diffraction spectra for silicon nitride from 20°C to 1200°C. These spectra were refined using the Rietveld analysis method. Lattice parameters obtained as a function of temperature from refined diffraction data allowed determination of thermal expansion coefficients for silicon nitride over this temperature range. Because silicon nitride has a hexagonal crystal structure, there are two lattice parameters, and thus two independent components in the thermal expansion tensor. Bulk measurements obtain only an average, physical thermal expansion. Using ND diffraction data allows the CTE tensor to be determined. These values were compared to physical, bulk expansion measurements.

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

CMAS-related Degradation and Mitigation Strategies II

Room: Flagler C (South Tower)
Session Chairs: Ravisankar Naraparaju, DLR - German Aerospace Center; David Poerschke, University of Minnesota

8:30 AM

(ICACC-S2-029-2023) Recession behavior of CMAS resistance Environmental Barrier Coating system in hot gas environment

N. Hosoya*[1]; N. Yamazaki[1]; T. Nakamura[1]; H. Kakisawa[2]; K. Shimoda[2]; M. Watanabe[2]

1. IHI Corporation, Japan
2. National Institute for Materials Science (NIMS), Japan

Silicon carbide fiber reinforced silicon carbide matrix composites (SiC/SiC CMC's) are attractive materials for use in aero engines hot section components. Environmental Barrier Coatings (EBCs) are essentials to prevent CMC's degradation against various damages such as water vapor oxidation and steam recession. Especially in high temperature such as 1400°C environment, we should pay attention for both these degradations and CMAS corrosion. Rare earth oxide based EBCs has been developed for CMAS resistance and CMC coupons were prepared coated with this EBC by atmospheric plasma spraying (APS). High speed gas stream test was conducted to evaluate recession behavior of this EBCs under steam velocity at 150 m/s at 1400°C for 100 h. Recession rate was low and the protective surface layer was formed at the surface of the coupon. Developed EBC showed good results for both CMAS-resistance and steam recession.

8:50 AM

(ICACC-S2-030-2023) Exploring CMAS-Steam Synergy on Yb-silicate Degradation

C. Luckhardt*[1]; E. J. Opila[2]

1. University of Virginia, Department of Materials Science & Engineering, USA
2. University of Virginia, Materials Science and Engineering, USA

CMAS degradation studies of EBCs are frequently conducted in high-temperature ambient air environments. However, steam produced in the combustion environment may lower CMAS viscosity, resulting in accelerated infiltration of the EBC. This work assesses the synergistic effects of steam on CMAS-EBC interactions. Dense spark plasma sintered phase-pure $Yb_2Si_2O_7$ and mixed-phase, nominally 20 vol% coupons of Yb_2SiO_5 in $Yb_2Si_2O_7$, specimens were exposed to approximately 28 and 40 mg/cm^2 loads of CMAS (Ca33-Mg9-Al13-Si45 in single cation mol%) at 1300°C for 4, 24, and 96h. Exposures were conducted in 90% steam/balance O_2, dry-O_2, both flowing at approximately 1.7 cm/s, or stagnant lab air. Specimens were exposed to each condition in triplicate to assess statistical significance of the environment on CMAS wetting behavior and infiltration. Post-exposure specimens were examined in plan view and cross-section with XRD, SEM, and EDS. Plan view features of interest include molten CMAS perimeter, swollen CMAS infiltrate area, apatite ring formation, and apatite formation outside of the ring. Cross-section features of interest include quantifying CMAS infiltration depth, reaction depth when applicable, and blister crack zone. The high-temperature steam environment produced similar CMAS degradation microstructures as observed in other environments but increased infiltration depths in phase-pure $Yb_2Si_2O_7$ after exposure for 24 hours.

9:10 AM

(ICACC-S2-031-2023) Impact of Surface Roughness on the Wettability of Molten Synthetic Sand on Thermal Barrier Coatings

A. Wright*[1]; A. Ghoshal[2]; M. Murugan[1]; L. Bravo[1]

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

Contact angle measurements, wettability, and surface roughness are key factors in the pursuit of a sandphobic coating. Several varying coating and bulk material chemistries were characterized for their surface roughness and subjected to molten synthetic sand at 1260 °C to evaluate the wettability. A relationship between the chemistries, roughness, and contact angle was drawn. An increase in surface roughness tends to decrease the wettability of sand on thermal barrier coatings, but a few chemistries show the opposite trend. A discussion about their wettability behavior is discussed along with parallels to water on hydrophobic and hydrophilic surfaces. In addition, the reactivity of the coatings is considered. Lastly, surface modification techniques such as laser ablation will be highlighted as future work.

9:30 AM

(ICACC-S2-032-2023) Study of CMAS infiltration and evaporation behaviour under water vapour/sulphur oxide conditions in EB-PVD 7YSZ (Invited)

R. Naraparaju*[1]; M. Xabier[2]; M. C. Galetz[2]; U. Schulz[1]

1. DLR - German Aerospace Center, Materials Research, Germany
2. DECHEMA, Germany

The infiltration behaviour of CaO-MgO-Al_2O_3-SiO_2 (CMAS) in Electron Beam Physical Vapour Deposited (EB-PVD) 7 wt.% yttria stabilized zirconia (7YSZ) under controlled atmospheres (dry, wet and sulphurizing) was studied in detail. The microstructure of the CMAS-infiltrated 7YSZ and the CMAS residue on top were analysed to interpret the corrosion mechanisms. One of the major goals was to investigate the stability and the infiltration of $CaSO_4$ into the 7YSZ columns. At the testing temperature of 1250°C, $CaSO_4$ was stabilized only under the SO_2 atmosphere within the TBC coating and only in $CaSO_4$-containing CMAS. Hydroxylation of CMAS elements was found under wet conditions, which changed the local CMAS composition and infiltration behaviour. These chemistry changes have provoked acidity changes in the CMAS which directly affected their aggressivity and viscosity. Thermodynamic equilibrium predictions on the volatility of CMAS components and the anhydrite stability under various atmospheres were used to understand the experimental findings and the underlying mechanisms.

10:20 AM

(ICACC-S2-033-2023) Computational and Experimental methods for characterization of Thermal and Environmental Barrier Coatings

A. Ghoshal*[1]; A. Wright[1]; L. Bravo[1]; M. Murugan[1]; R. Koneru[1]; A. Flatau[1]

1. US Army Research Laboratory, USA

This paper presents DEVCOM ARL's research methods for investigating thermal and environmental barrier coatings for applications in extreme thermal environments. The computational methods incorporate multiphase turbulent gas dynamics without and without particulate entrainment models under confined and external high speed aerodynamics. Currently ARL is working with its research partners to develop full scale engine simulation with large scale eddy simulation (LES) and conjugate heat transfer (CHT) to accurately model the aerothermal loads that are imposed on the hot section components with thermal and environmental barrier coatings. Accurate life models of the hot section structural components depend upon tight coupling of the fluid structure interaction, appropriate aerothermal load transfer, structural and material models under the appropriate thermal and pressure ranges. The experimental methods include investigating chemical kinetics and diffusion coupling of T/EBC with CMAS, relevant thermal cyclic and thermal shock experiments in box furnace and at ARL Hot particulate ingestion Rig (HPIR)/ atmospheric jet burner rig. Contact angle experiments to ascertain the CMAS kinematic spread phase and infiltration dynamics within the T/EBC multilayer systems. The coatings development also include optimizing the spray system parameters, understanding the flowability of the spray powder and powder chemistries.

10:50 AM

(ICACC-S2-034-2023) Corrosion behavior of gadolinium silicates with CMAS for environmental barrier coatings

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1. Kyushu University, Interdisciplinary Graduate School of Engineering Sciences, Japan
2. Kyushu University, Interdisciplinary Graduate School of Engineering Science, Japan

Environmental barrier coatings (EBCs) prevent the oxidation of ceramic matrix composites (CMCs), which are used as components in gas turbines. However, EBCs deteriorate more rapidly in real environments, molten silicate deposits accelerate the deterioration of EBCs. In this study, the Gd_2SiO_5 and $Gd_2Si_2O_7$ performed high temperature corrosion behavior on synthetic calcia-magnesia-alumina-silica (CMAS) for EBCs. Corrosion behavior was investigated by sintered gadolinium silicates with CMAS and heating them to 1400°C for 2, 12, and 48 h. HT-XRD results analyzed that at 1300°C, CMAS and gadolinium silicates chemically reacted to form $Ca_2Gd_8(SiO_4)_6O_2$ (apatite). The content of the main Ca element in the formation of apatite is dominant. The reaction layer becomes thicker vertically as the heat-treatment time increases, and the thickness of the reaction layer has increased following an parabolic curve. It was analyzed that $Gd_2Si_2O_7$ had a higher effect on reaction layer formation than Gd_2SiO_5.

11:10 AM

(ICACC-S2-035-2023) Improvement of Resistance to CMAS Corrosion of Environmental Barrier Coating by Using Rare Earth-Based Oxides

N. Yamazaki*[1]; S. Kanazawa[2]; T. Nakamura[1]; T. Ito[3]; M. Tanaka[4]; S. Kitaoka[3]

1. IHI Corporation, Japan
2. IHI Corporation, Japan
3. Japan Fine Ceramics Center, Japan
4. Japan Fine Ceramics Center, Japan

Ceramic Matrix Composites (SiC_f/SiC CMCs) are expected to be capable as high-pressure turbines in aero engines under high temperatures with critical attacks by environmental factors, such as water vapor oxidation, erosion, and CMAS corrosion. In such cases, Environmental Barrier Coatings (EBCs) are essential parts for the protection of the SiC/SiC CMCs. As CMAS corrosion is one of the severe factors, the reaction mechanism of CMAS should be investigated further to understand effective materials systems for the EBCs. Sintered materials were fabricated as a sample of EBCs and the reaction under CMAS corrosion was analyzed. The correlation was evaluated specifically between the amounts of various oxides added to Rare Earth and the reaction process. Some effective materials were identified in those tests with sintered samples and they were deposited by thermal spraying. The result from examination of the deposited materials were compared with the sintered on the view of the reacted form. We proposed an effective microstructure based on the results that has the potential to work as protection layer toward CMAS corrosion.

11:30 AM

(ICACC-S2-036-2023) Rare-earth disilicate systems for T/EBC application: Synthesis and high temperature interaction with CMAS

C. Y. Guijosa Garcia*[1]; R. Naraparaju[1]; P. Mechnich[1]; U. Schulz[1]

1. DLR - German Aerospace Center, Institute of Materials Research, Germany

Thermal and environmental barrier coatings (TEBCs) that are intended to be used on top of ceramic matrix composites (CMCs) require complex properties such as high temperature phase stability, toughness, inertness to water vapor attack, and protection against silica deposit (CMAS) attack. Multi component rare earth disilicates $RE_2Si_2O_7$ (REDS) such as mixtures of Y, Yb, Er, Ho, Lu, and Sc have been identified as promising TEBC materials due to their high-temperature phase stability, low thermal conductivity, and coefficients of thermal expansion similar to SiC-CMCs ($4.5x10^{-6}$). A novel synthesis method using high-energy milling and solid-state reaction has been successfully developed for producing multi-component REDS powders rapidly. The current work also focuses on understanding the thermochemical reactions between single-component Y, Yb, Er, Ho, Lu, and Sc disilicates as well as multi-component REDS with different CMAS sources at 1350 and 1400 °C for 1h using REDS+CMAS powder mixtures, respectively. Results showed the successful synthesis of monoclinic RE disilicatephases (β and γ types). Structural and chemical analysis confirmed the formation of REDS (Yb, Lu, Sc) reprecipitates, reactive crystallization products as apatite (Er), and intrinsic crystallization products such as diopside. Additionally, Y and Ho thermochemical reactions will be discussed.

11:50 AM

(ICACC-S2-037-2023) CMAS Induced Stiffening of Magnesia-Alumina Spinel Thermal Barrier Coatings

M. Vreeswijk*[1]; N. Jones[1]

1. University of Cambridge, Department of Materials Science and Metallurgy, United Kingdom

The efficiency of turbine engines increases as the temperatures of the gas stream is raised. The introduction of new higher-efficiency gas turbine engines requires novel materials to manage temperatures. High-temperature thermal barrier coatings (TBC) act as a temperature stable barrier protecting the metallic hardware within the engine. The magnesia-alumina spinel (MAS) system has been proposed as an alternative abradable TBC to yttria-stabilised zirconia (YSZ), due to its improved phase and microstructural stability. However, little is known about the interaction of MAS coatings with molten CaO-MgO-Al_2O_3-CaO (CMAS) deposits, which are known to be detrimental to the lifetime of other TBC coatings. This study characterises the interaction between CMAS and MAS and contrasts this with the interaction between CMAS and YSZ. To achieve this CMAS was applied to free standing MAS and YSZ coating samples, which were subsequently heat treated at temperatures between 1000 and 1300 deg. C, allowing the deposit to interact with the coating material. The effect of CMAS on the coating's Young's modulus was assessed and the phase evolution of the coating system was characterised using XRD and SEM methods. Significant differences in the deposit-coating interactions were observed between the two systems, based on these observations, a mechanism for the stiffening induced by CMAS in MAS will be proposed.

S3: 20th International Symposium on Solid Oxide Cells (SOC): Materials, Science and Technology

Novel Processing

Room: Ponce de Leon (North Tower)
Session Chairs: Sebastian Molin, Gdansk University of Technology; Dario Montinaro, SOLIDpower SpA

8:30 AM

(ICACC-S3-027-2023) Towards the Next Generation of 3D printed Solid Oxide Cells and Stacks (Invited)

A. Tarancón*[1]; N. Kostretsova[2]; R. S. Pavlov[2]; L. Bernadet[2]; M. Lira[2]; M. Núñez[2]; A. Sabato[2]; A. Morata[2]; M. Torrell[2]

1. IREC / ICREA, Spain
2. IREC, Spain

Hydrogen technologies are crucial for the foreseen transition towards a more sustainable society based on renewable energy. The most efficient of those technologies is based on high temperature operation devices such as Solid Oxide Cells. Solid Oxide Fuel and Electrolysis Cells have been developed during many decades and, nowadays, reached a level of maturity for a mass market entry. Conventional manufacturing technologies such as tape casting or screen printing enabled the existing generation of high temperature solid oxide cells. This successful story ended up in a feasible technology currently reaching the MW scale and thousands of hour of continuous operation. Complementary, novel fabrication approaches are recently presented pushing the limits of some classical constraints such as simple shape or slow startup opening new application scenarios. In this talk, we will present recent advances in 3D printing of solid oxide cells and stacks for improving the performance and durability of currently existing designs.

9:00 AM

(ICACC-S3-028-2023) Study and optimisation of thin film based barrier layer for large area Solid Oxide Cells

M. Torrell*[1]; L. Bernadet[1]; J. Segura[4]; D. Montinaro[2]; A. Morata[1]; A. Tarancón[3]

1. IREC, Adv. Materials for Energy, Spain
2. SOLIDpower SpA, Italy
3. IREC / ICREA, Spain
4. ID16B, European Synchrotron (ESRF), France

Nowadays, the state of the art SOC cells, using LSCF perovskites as oxygen electrode, includes a barrier layers of gadolinium doped ceria (Ce1-xGdxO2) to block the reaction between Sr and Zr during the cell fabrication and operation. However, obtaining a the optimal microstructure, thickness and attachment of the barrier layer using typical deposition techniques such as screen-printing presents several issues. Usually the presented CGO barrier layers are porous, when sintered at temperatures 1200°C or present undesired CGO-YSZ inter-diffusion when sintered at temperatures >1200°C. A complete study of different operated CGO barrier layers by different characterization techniques such as SEM, TEM, Raman, EPMA-WDS, X-Ray synchrotron fluorescence nano-probe allows to understand and deeply discuss the impact of the barrier layer on the oxygen electrode degradation. The improvement of the electrolyte/oxygen electrode interface by the fabrication of an enhanced barrier layer is a key point to further improve the solid oxide cells performance and durability. The differences between standard screen-printed CGO interlayer and pulsed laser deposition (PLD) deposited barrier layers in large area cells is here discussed.

9:20 AM

(ICACC-S3-029-2023) Fabrication of Micro-Tubular Metal Supported Solid Oxide Fuel Cells

S. K. Sahu*[1]; D. Panthi[1]; Y. Du[1]

1. Kent State University, USA

Although SOFC is a great technology for the production of electrical energy with good efficiency and minimal environmental impact, its successful commercialization has been hindered due to the high cost associated with SOFC production and its limited long-term stability. Metal supported SOFC is looked upon as a potential candidate for the rapid commercialization of SOFC technology. Recently there have been several efforts towards the fabrication of MS-SOFC, but the processes are complicated and expensive. The goal of this work is to develop micro-tubular MS-SOFC using cost-effective fabrication techniques. In this work, the tubular metal support was fabricated using 3D printing technology. Successive coatings of the SOFC components such as the anode functional layer (AFL; Ni-3mol%Y_2O_3 stabilized ZrO_2), electrolyte (EL; Sc_2O_3 stabilized ZrO_2) and the cathode functional layer (CFL; $(La,Sr)(Co,Fe)O_3$) were developed on porous metallic support using dip coating. Additionally, to prevent the delamination an intermediate layer (between metal support and anode) consisting of stainless steel powder, Ni, and YSZ was introduced. After coating of the individual layers, they were fired in a reducing atmosphere at 1000 °C followed by co-sintering of metal/anode/electrolyte multilayers at 1350 °C. Microstructure of the sintered cells was characterized using scanning electron microscopy/energy-dispersive X-ray spectroscopy.

9:40 AM

(ICACC-S3-030-2023) Laser-drilling of gas channels for solid oxide cells

S. Molin*[1]

1. Gdansk University of Technology, Laboratory of Functional Materials, Faculty of Electronics, Telecommunications and Informatics, Poland

In this work, we show the electrochemical and microstructural results concerning the modification of commercial solid oxide cells (type ASC-400B, produced by Elcogen) by femtosecond laser drilling. The laser processing has been performed on sintered cells. The femtosecond laser drilling technique results in high quality of the ablated surfaces, where the ablation/removal of the ceramic layers occurs fast and the removed materials do not affect the other cell parts. We have fabricated gas channels with different geometries (depth, thickness, number/density of holes) and evaluated their influence on the electrochemical performance of the cells. Femtosecond laser drilling can be used post-production to tailor-made cells with improved gas transport properties. Acknowledgement: The project has been funded by National Science Centre, grant agreement 2020/38/L/ST8/00513.

10:20 AM

(ICACC-S3-031-2023) Advanced Materials and Processes for Highly Efficient Protonic Ceramic Electrochemical Cells (Invited)

K. Lee*[1]

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

Protonic ceramic electrochemical cells (PCECs) are highly efficient and sustainable energy devices to convert chemical fuels into electric power and vice versa on demand at reduced temperature below 600 °C. However, PCECs are suffering from issues to form secondary phases of perovskite electrolyte materials because of extremely processing conditions with high temperature and long annealing time, leading to significant decrease on electrochemical performance of PCECs. Furthermore, at low temperatures, the poor catalytic activity of PCECs occurs by the slow kinetics at the air electrode. To address these issues, we attempt new approach with ultra-fast sintering process, which effectively reduces the sintering time within 5 min. In addition, we develop newly doped $BaCoO_3$ air electrodes to enhance the oxygen permeability and phase stability, thus enhancing their activity and durability. In this presentation I will show our recent progress in the development of highly efficient PCECs.

10:50 AM

(ICACC-S3-032-2023) Fabrication of spinel ceramics for SOFC support and fuel reforming applications

A. Veronese*[1]; J. T. Irvine[1]

1. University of St Andrews, United Kingdom

Spinel oxides based on the abundant elements Mg, Cr, Mn, Fe and Ni are promising reforming catalysts for pyrolysis gasses and tar. Their activity comes from redox activity of the Mn^{2+}/Mn^{3+} couple, good coking resistance and the exsolution of active metallic phases in reducing conditions. Such spinel ceramics could be ideal supports for SOFC running on bioderived fuels, providing a barrier to electrode contamination, mechanical support, and a catalytic substrate for fuel reforming. However, the faceted grain morphology of the spinels hinders their sintering, making the manufacturing of tough and mechanically robust ceramics challenging, especially with the levels of porosity required for catalyst applications. To facilitate sintering, the approach of reactive sintering of compositionally inhomogeneous powders was studied. The two ceramic systems investigated were single-phase spinel, fired from a mixture of spinel powders with different compositions and dual-phase magnesia/spinel composites.

11:10 AM

(ICACC-S3-033-2023) Additive manufacturing of high performance protonic ceramic fuel cells

M. Asghar*[1]; P. Lund[1]

1. Aalto University, New Energy Technologies Group, Department of Applied Physics, Finland

Protonic ceramic fuel cells (PCFC) have been rapidly developing as a potential technology. The low temperature (400-600°C) of the cell is enabled due to high ionic conductivity (>0.1 S/cm) with a low activation energy (< 50 eV) of proton conducting ceramic electrolytes. The additive manufacturing has the potential to revolutionize the manufacturing of PCFCs since it can fabricate both the dense and porous structures with good mechanical and electrochemical properties. In this work, we fabricated PCFCs through a hybrid of extrusion-based 3D printing utilizing $BaZr_{0.4}Ce_{0.4}Y_{0.1}Yb_{0.1}O_{3-\delta}$ and $BaZr_{0.3}Ce_{0.5}Y_{0.15}O_{3-\delta}$ as electrolytes, their composite with NiO as anode, and their composite with $Ba_{0.5}Sr_{0.5}Co_{0.8}Fe_{0.2}O_{3-\delta}$ and $Ba_{0.5}Sr_{0.5}Co_{0.8}Fe_{0.2}O_{3-\delta}$ as cathode. The rheological properties of the printable pastes were investigated in detail with the dynamic light scattering, viscometer, tensiometry, differential scanning calorimetry and thermal gravimetric analysis. Furthermore, the cells were characterized with current-voltage measurements and electrochemical impedance spectroscopy. In addition, other spectroscopic and microscopic measurements (HR-TEM-EELS, SEM-EDX, XPS) were conducted to better understand the mechanisms in the cells. Finally, single-layer cells utilizing proton conductors were printed and a systematic study was conducted to optimize their sintering temperature for optimal fuel cell performance.

11:30 AM

(ICACC-S3-034-2023) Novel designs to improve the performance of solid oxide cells by 3D printing technologies

Z. Zhou*[1]; V. K. Nadimpalli[2]; D. B. Pedersen[2]; V. Esposito[1]

1. Technical University of Denmark, Department of Energy Conversion and Storage, Denmark
2. Technical University of Denmark, Department of Mechanical Engineering, Denmark

3D printing technologies have distinct advantages in fabricating complex porous structures because of their almost unconstrained design freedom and precise forming for many materials, especially metals and ceramics. In this work, we show a few examples. We optimized the porous metal supports for SOCs with straight gas channels using laser powder bed fusion. The novel proposed design aims to decrease the tortuosity of gas channels and facilitate gas diffusion, promoting reaction efficiency. Ceria-based multi-coatings are formed on metal supports' regular inner surface by combining electrophoretic deposition and infiltration methods for superior oxidation resistance. We show a novel monolithic SOC with integrated forming by digital light processing (DLP) as a second case study. It is self-supported by the electrolyte fabricated with yttria-stabilized zirconia (YSZ) by DLP-based vat photopolymerization. Then the anode and cathode are formed on the two sides of the electrolyte by coating and infiltration methods. This design aims to increase the active area within the unit volume as much as possible, achieving high volume power density while avoiding connecting and sealing single cells as conventionally.

11:50 AM

(ICACC-S3-035-2023) Symmetrical solid oxide electrolysis cells with gradual porosity produced by the freeze tape casting technique

D. Cademartori[1]; E. Mercadelli[2]; A. Sanson[2]; A. Gondolini[2]; M. Carpanese*[1]

1. University of Genoa, DICCA, Italy
2. CNR-ISTEC, Italy

Solid oxide electrolysis cells (SOECs) show favourable thermodynamics and reaction kinetics, allowing for unmatched conversion efficiencies. These features, combined with other advantages, make them key devices within networks based on renewable sources. Although the performance of SOECs has strongly improved over the past recent years, they still suffer from instability issues that cast doubt on the use in the long term to produce hydrogen. It can be affirmed that the mechanisms of degradation is mainly due to the mechanical instability of the standard electrodes, where a building up pressure causes delamination, especially at high operating current densities. In this work, the technique of the freeze tape-casting is described to produce a button cell with a symmetrical structure and a gradual electrode porosity, discussing the approach that led to the freezing conditions especially optimized. The performance of the cell thus obtained was compared with that of a cell with standard porosity electrodes, by Computational Fluid Dynamics (CFD) modelling, demonstrating the advantages that the open and gradual porosity produces in term of the gas accumulation during the electrolysis operation. The result evinces the advantages that such a hierarchical porosity can have on the life of the cell, preventing eventual mechanical instabilities.

S6: Advanced Materials and Technologies for Rechargeable Energy Storage

Advanced Anode Materials for Lithium Batteries I

Room: Coquina Salon E (North Tower)
Session Chairs: Dominic Bresser, Helmholtz Institute Ulm; Prashant Kumta, University of Pittsburgh

8:30 AM

(ICACC-S6-023-2023) Reliability of Li Metal Protection Layers (Invited)

R. Dominko*[1]; N. Pavlin[1]; J. Bobnar[1]; U. Kosir[1]; B. Genorio[2]

1. National Institute of Chemistry, Slovenia
2. FKKT, University of Ljubljana, Slovenia

A new cell design with protected edges results in reliable results of the effectiveness of coatings developed for the stabilization of metallic lithium. Different strategies to protect metallic lithium have been examined in our lab, like different coatings of the polymer ionic liquids, modified graphene oxide, or modified nanocrystalline cellulose. Using cellulose is considered a sustainable approach. 3mm thick cellulose layers effectively prevent metallic lithium's constant exposure to electrolyte. The beneficial influence was confirmed with long-term lithium stripping and deposition, long-term cycling in carbonate and ether-based electrolytes, ex-situ focused ion-beam scanning electron microscope (FIB-SEM), operando ATR-IR spectroscopy and EIS.

9:00 AM

(ICACC-S6-024-2023) Enabling High-Rate Lithium Metal Anodes by Tailored Structures and Interfaces (Invited)

E. D. Wachsman*[1]

1. University of Maryland, USA

Oxide-based solid-state Li-batteries (SSLiBs) have the potential to be a transformational and intrinsically safe energy storage solution due to their non-flammable ceramic electrolyte that enables the use of high-capacity Li metal anodes and high voltage cathodes for higher energy density over a much wider operating temperature range. However, their progress has been limited due to electrode/electrolyte interfacial issues. In particular for Li-metal anodes concerns over dendrite formation/propagation and the requirement for elevated temperature and high stack pressure are still prevalent. To eliminate these concerns a rational design of tailored structures and interfaces in Li-metal anodes will be presented. In addition, progress toward full cells using these tailored structures and interfaces will be presented.

9:30 AM

(ICACC-S6-025-2023) Silicon-Based Anode Materials for Advanced Lithium and Sodium Ion Batteries (Invited)

R. Riedel*[1]

1. TU Darmstadt, Materials Science, Germany

The increase in energy density and power density requirements for lithium and sodium ion batteries leads to continuous research for new electrode materials. Various insertion materials have been proposed as negative electrodes for rechargeable batteries. In Li-ion batteries, the highest theoretical capacity can be obtained when using metallic lithium (3862 mAh/g) or elemental silicon forming lithium rich alloys (3578 mAh/g), however these materials are not commercialized due to safety reasons and rapid capacity fading, respectively. At present, mostly graphitic materials are used due to low price and high reversibility despite relatively low capacity (372 mAh/g), instability during long time cycling and inadequacy high power applications. Therefore, new materials, which are economically interesting but demonstrate higher capacity, longer life time and better high rate capability are urgently required to meet the technological demands of our future electromobility and energy storage applications. In this context, novel anode materials based on ceramic nanocomposites comprised of ternary SiCN and SiOC systems are discussed and evaluated in terms of their electrochemical performance with respect to application in Li- and Na-ion batteries.

Advanced Anode Materials for Lithium Batteries II

Room: Coquina Salon E (North Tower)
Session Chairs: Eric Wachsman, University of Maryland; Robert Dominko, National Institute of Chemistry

10:20 AM

(ICACC-S6-026-2023) Alternative Charge Storage Mechanism for Alkali Metal-Ion Anodes (Invited)

D. Bresser*[1]

1. Helmholtz Institute Ulm, Germany

Li-ion batteries (LIBs) form the key electrochemical energy storage technology for suites of applications. Nonetheless, concerns about Li-based resources has triggered search for alternative battery technologies like sodium-ion batteries (SIBs) and potassium-ion batteries (PIBs). However, several classic electrode active materials proven as excellent Li^+ host, such as graphite, do not sow the same charge storage mechanism for Na^+, while reversibly hosting K^+. Herein, a new class of active materials will be presented showing essentially the same charge storage mechanism for all three alkali-metal cations – apart from the expected "size effect". This charge storage mechanism involves reversible solid-solution-type insertion of the alkali-metal cations into a metal oxide host and complete reduction of metal dopants to the metallic state. As a result, the specific capacity can be doubled and even tripled, leading to exceptional power densities in alkali-ion full-cells.

10:50 AM

(ICACC-S6-027-2023) Emergent Electrochemically Active Materials for High Energy Density Li-S Batteries (Invited)

P. N. Kumta*[1]

1. University of Pittsburgh, Mechanical Engineering and Materials Science, USA

Lithium – ion batteries have made tremendous strides since Sony commercialized the first Li-ion battery in 1990. While lithiated transition metal oxides and carbon are still the flagship electrodes, recent efforts are directed at high energy density Li-air and Li-sulfur (Li-S) batteries. Li-S systems are of significant interest due to the promise of attaining 500 Wh/kg, targeted for next generation electric vehicles. The system albeit suffers from poor Li transport, more importantly, soluble polysulfides, and dendrite formation in Li metal anodes posing inferior cell performance and more importantly, major safety hazards. This presentation will discuss the recent results achieved in identifying new sulfur confinement cathodes with the ability to confine > 5 mg/cm^2 sulfur loadings combined with incorporating first principles theory identified functional electrocatalysts and Li-ion conductors permitting high capacity (>1000-1200 mAh/g). Additionally, progress made in new dendrite free anode materials and current collectors will also be outlined. Promise of these new systems to achieving the 500 Wh/kg target will also be discussed.

11:20 AM

(ICACC-S6-028-2023) Electrochemical performance of polymer-derived SiOC/GNP- composites for high-performance Li-ion batteries

D. K. Panda*[1]; G. Jella[2]; N. Sapkota[3]; A. M. Rao[3]; S. Ravindran[2]; R. Bordia[1]

1. Clemson University, Materials Science and Engineering, USA
2. Birla Institute of Technology & Science Pilani, Hyderabad Campus, Mechanical Engineering, India
3. Clemson University, Department of Physics and Astronomy, USA

Amorphous silicon-oxycarbide (SiOC) ceramics have a high theoretical capacity and good structural stability, which makes them suitable as anode materials for lithium-ion batteries (LIBs). SiOC, however, has poor electronic conductivity, poor transport properties, poor cycle stability, and limited rate capabilities. It is therefore urgent to develop an anode material based on SiOC that could mitigate the limitations listed above. A carbon-rich SiOC (SiOC-I) and a silicon-rich SiOC (SiOC-II) ceramic was synthesized to improve electrochemical performance. For the first time, carbon nanotube-based bucky paper was paired with SiOC-I or SiOC-II as the anode in a Li-ion battery. Electrochemical performance of SiOC-II/GNP composites was improved when mixed with graphene nanoplatelets (GNPs). In comparison to monolithic SiOC-I, SiOC-II, or even GNPs, the composite anode (25 wt.% SiOC and 75% GNP) achieved a higher specific capacity and cycling stability (744 mAh/g at 0.1C rate). The improved electrochemical performance can be attributed to increased electronic conductivity, a lower charge-transfer resistance, shorter ion diffusion lengths, and reduced agglomeration. SiOC-II/GNP demonstrated excellent cycling stability, achieving 344 mAh/g after 260 cycles at 0.5 C-rate.

11:40 AM

(ICACC-S6-029-2023) Suppressing Li-dendrite formation at Li Metal Anode by Metall Oxide Protecting Nano-Coatings with Application in Sustainable Energy Storage

D. Patrun*[1]; M. Wilhelm[3]; S. Mathur[2]

1. University of Cologne, Inorganic/Materials Chemistry, Germany
2. University of Cologne, Institute of Inorganic Chemistry, Germany
3. University of Cologne, Germany

The numerous applications of batteries call for continuous improvement and new technologies to meet the demand for energy storage. Today, batteries are indispensable since they are part of modern life, from applications in energy storage for renewable energy to smart devices. Solid-state batteries (SSBs) built the next generation of renewable power generation technology, as they promise to produce far better energy densities utilizing lithium metal as the anode material rather than graphite while being significantly more robust and lighter. However, the formation of Li-dendrites is a known problem in this context. SSBs using solid electrolytes can potentially stop this dendrite growth, preventing a short circuit in the battery. However, engineering the electrode/electrolyte interface is unavoidable to improve the performance of the battery. Therefore, we explored state-of-the-art materials such as metal oxide and different metal chalcogenide nanostructurd coatings on the lithium metal anodes to improve the cell performance. The coatings were obtained by various gas phase deposition methods, such as plasma-enhanced chemical vapor deposition (PECVD) and atomic layer deposition (ALD). The investigated materials and the electrode/electrolyte interface were characterized for their structural and mechanical changes concerning battery performance.

S8: 17th International Symposium on Advanced Processing and Manufacturing Technologies for Structural and Multifunctional Materials and Systems (APMT17)

Microwave Processing, SPS, Flash Sintering, High Pressure Assisted Sintering

Room: Coquina Salon F (North Tower)
Session Chairs: Enrico Bernardo, University of Padova;
Anne Leriche, Université Polytechnique Hauts-de-France

9:00 AM

(ICACC-S8-022-2023) Nanostructured rutile TiO_2 ceramics fabricated by High Pressure Spark Plasma Sintering: effect of high pressure on physical densification phenomena (Invited)

S. Cottrino*[1]; T. Gaudisson[2]; E. Ferrara[1]; Y. Le Godec[3]; S. Le Floch[2]

1. MATEIS Laboratory, Material, France
2. Lumière Matière Institut, France
3. IMPMC laboratory, France

Rutile is the most common, stable polymorph form of titanium oxide TiO_2 at all temperatures. Nanocrystalline TiO_2 has attracted increasing interest as a ceramic. For example, the doping of rutile TiO_2 with a small amount of niobium is known to induce a large increase of the electrical conductivity, broadening its technological interest towards new emerging fields such as the thermoelectric conversion of waste heat. In this work, high-density bulk TiO_2 was fabricated from a nanopowder of anatase TiO_2 by high pressure spark plasma sintering (SPS) technique. It is shown that by applying high pressure (76 MPa to 1 GPa), densification and phase transition start at lower temperature and are accelerated. Thus, it is possible to dissociate the two densification steps (anatase then rutile) and the transition phase during the sintering cycle. Regardless of the applied pressure, grain growth occurs during the final stage of the sintering process. However, twinning of the grains induced by the phase transition is enhanced under high pressure resulting in a reduction in the crystallite size. Thus high pressure SPS technique has allowed to vary the grain size from 300 to 170 nm. Grain growth and densification activation energy have been calculated to show the impact of pressure during sintering thermal treatment.

9:30 AM

(ICACC-S8-023-2023) Applications and developments in the field of FAST/SPS-Sintering, taking the development of graded ceramic solid state electrolyte as an example

J. W. Huber*[1]

1. Dr. Fritsch Sondermaschinen GmbH, FAST/SPS Sintering Technology, Germany

Dr. Fritsch and the Fraunhofer institute IPA in Stuttgart, Germany, are partners in a research project for the development of sintering techniques for the development of graded ceramic solid state electrolytes. Ceramic powders are usually hot pressed. The disadvantages of traditional hot-pressing are the long sinter cycle times of several hours, the energy intensive operation of the hot presses and the negative effects of the long exposure to heat on the material properties such as grain growth which results in poor homogeneity and lower density. FAST/SPS reduces such issues to a minimum by reducing the sinter cycle time thanks to high heating rates and comparably short heat exposure. At the same time, higher densities achieved by the FAST/SPS technique are improving the conductivity of ions. The requirement for connecting graded layers is another reason for preferring FAST/SPS over other sintering techniques. The market for advanced batteries is supposed to grow annually in average by 8,53% (CAGR). This growth rate indicates that any technology used in R&D must be suitable for upscaling. The development of a FAST/SPS sinter press with a sinter and seperate cooling chamber, allowing semi-continous production, is an important step towards cost-effective automated mass production.

9:50 AM

(ICACC-S8-024-2023) Synthesis and Characterization of High Entropy Nitrides

S. Das*[1]; A. Eskandariyun[1]; M. Sozal[1]; V. Drozd[2]; A. Durygin[2]; M. Cinibulk[3]; Z. Cheng[1]

1. Florida International University, Mechanical and Materials Engineering, USA
2. Florida International University, Center for the Study of Matter at Extreme Conditions (CeSMEC), USA
3. Air Force Research Lab, USA

High entropy nitrides (HEN) are single-phase nitrides with 5 or more metal elements at the cation site. Mixing of multiple elements at the cation sublattice increases the configuration entropy of the system and might offer enhanced physical or chemical properties compared to their constituent binary nitrides. This study aims to synthesize bulk HEN by reaction flash sintering (RFS) method. Commercial binary metal nitride powders were mixed and consolidated into single-phase HEN using a homemade setup. A constant DC electrical field of ~40 V/cm and pressure of ~15 MPa at room temperature triggered RFS without pre-heating, and the entire process finished in ~250 s. The products' phase composition is characterized by XRD, while the microstructure, as well as oxidation and mechanical properties are characterized using different techniques such as SEM/EDS, TGA, and indentation to understand the high entropy effect on products' microstructure and properties.

10:30 AM

(ICACC-S8-025-2023) Dynamic sinter forging for preparation of high-performance SiC whisker reinforced Al_2O_3 composite (Invited)

G. Shao*[1]; H. He[1]; H. Wang[1]; R. Zhang[2]; L. An[3]

1. Zhengzhou University, School of Materials Science and Engineering, China
2. Zhengzhou University of Aeronautics, Henan Key Laboratory of Aeronautical Material and Application Technology, China
3. Dongguan University of Technology, China

In this talk, we'll present a novel process, dynamic sinter forging (DSF), for the preparation of high-performance ceramic composites. Compared with samples made by conventional sinter forging and hot oscillatory pressing, the SiC whisker reinforced Al_2O_3 composite prepared by DSF at the same temperature exhibited a higher density and significantly improved mechanical properties. The improvements in densification and performance are attributed to simultaneous enhanced shear deformation at both macro- and micro-scales, resulting from the combination of die-free configuration and oscillatory pressure of DSF. The current results shed light on a powerful technique for preparing ceramic composites, which is likely applicable to other systems.

11:00 AM

(ICACC-S8-026-2023) Correlation with the Microstructure and Synergistic Physiochemical Etching Resistance of Nanocomposites under Fluorine-Containing Plasma

H. Ma*[1]; S. Hong[2]; H. Kim[1]; J. Lee[1]; M. Kim[1]; H. Lee[2]; Y. Park[1]

1. Korea Institute of Materials Science, Republic of Korea
2. Korea Research Institute of Standards and Science, Republic of Korea

In the semiconductor fabrication industry, high-power plasma is indispensable to obtain a high aspect ratio of chips. For applications to ceramic components including the dielectric window and ring in the semiconductor etching chamber, the Y2O3 ceramics have attracted interest recently based on excellent erosion resistance. When a high bias voltage is applied in a plasma environment containing fluorine gas, both chemical etching and ion bombardment act simultaneously on the ceramic components. During this etching process, severe erosion and particles generated on the ceramic surface can have effects on overall equipment effectiveness. Herein, we report the outstanding plasma etching resistance of Y2O3–MgO nanocomposite ceramics under a CF4/Ar/O2 gas atmosphere; the erosion depth of this material is 40–79% of that of the reference materials, Y2O3 ceramics. In a robust approach involving effective control of the microstructure with different initial particles and sintering conditions, it is possible to understand the relationship between etching behavior and microstructure evolution of the nanocomposite ceramic. The results indicate that the nanocomposite with fine and homogeneous domain distribution can decrease particle generation and ameliorate its life cycle.

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

New Concepts and Emerging Technologies for Enhanced Product Performance I

Room: Ballroom 1-2 (South Tower)
Session Chair: Chisung Ahn, Korea Institute of Industrial Technology

9:00 AM

(ICACC-S11-001-2023) Machine Learning approach in designing catalytic alloy nanoparticle (Invited)

Y. Kim*[1]

1. Kookmin University, School of Advanced Materials Engineering, Republic of Korea

Searching for an optimal component and composition of multimetallic alloy catalysts, comprising two or more elements, is one of the key issues in catalysis research. Due to the exhaustive data requirement of conventional machine learning (ML) models and the high cost of experimental trials, current approaches rely mainly on the combination of density functional theory (DFT) and ML techniques. In this study, a significant step was taken toward overcoming limitations by the interplay of experiment and active learning that can effectively search for an optimal component and composition of multi-metallic alloy catalysts. The active learning model suggests process parameters that are likely to yield high catalytic performance, and the model is iteratively updated using fabricated solid-solution nanoparticles by the carbothermal shock (CTS) method by examining their electro-catalytic performance for the hydrogen evolution reaction (HER). An optimal metal precursor composition of Pt0.65Ru0.30Ni0.05 is located, which exhibits an HER overpotential of 54.2 mV; this HER potential is superior to that of a pure Pt catalyst (68.2 mV). This result indicates the success of the constructed model by only utilizing the precursor mixture composition as input data, thereby improving the overpotential by searching for an optimal component and composition inside the system.

9:30 AM

(ICACC-S11-002-2023) Material design by combining AI and computational materials science (Invited)

D. Lee*[1]

1. Pohang University of Science and Technology(POSTECH), Materials Science and Engineering, Republic of Korea

The recent development of computer technology and efficient computing methods have brought innovation to material research by using computers for understanding and predicting materials behaviors. In computational researches, materials systems are simulated based on theoretical model and the change in materials behavior are predicted by the change in thermodynamic and kinetic variables. However, it is difficult to include all the variables for the materials processing, so various attempts are made to overcome these limitations using artificial intelligence (AI). AI is a technique that finds multidimensional nonlinear relationships hidden in data, and so it is possible to find correlations between various process variables and the performance of materials. Therefore, research and development of materials can be accelerated by in-depth analysis and understanding the effects of composition and process variables on material properties and performance by combining computational materials science and AI. In this study, we will introduce our recent studies on materials research that combines computational materials science and AI techniques.

New Concepts and Emerging Technologies for Enhanced Product Performance II

Room: Ballroom 1-2 (South Tower)
Session Chair: Sungwook Mhin, Korea Institute of Industrial Technology

10:20 AM

(ICACC-S11-003-2023) Simulation Paradigm Shift to the Platform: Materials Square (Invited)

M. Park*[1]

1. Virtual Lab Inc., Republic of Korea

Computer-aided research plays an important role in the entire field of study along with the paradigm of digital transformation. Metallurgy, ceramics, chemistry and biotechnology, as well as the semiconductor industry, have introduced computer simulation to increase yields and discover new materials. Research paradigm should be changed. In few decades, simulation-based research has attracted attention with the development of computational power and the efficient methodologies, called "In Silico Approach". Rather than finding a target by experimenting sequentially, you can experiment with a sample with high probability first via In Silico Approach. Despite these advantages, there are many barriers to introduce simulation to the research, such as expansive equipment, domain knowledge for computational science and complex Linux environment. Materials Square [1] is a cloud-based simulation platform, it provides everything you need for cutting-edge materials/chemical simulation platform including simulation modeling, computational server and analysis, with a cloud- based intuitive user interface. In this presentation, I would like to introduce innovative simulation platform, Materials Square. References [1] https://www.matsq.com

10:50 AM

(ICACC-S11-005-2023) Development of Surface Treatment Process to Prepare High Density Slurry for Ceramic 3D Printing

C. Ahn*[1]

1. Korea Institute of Industrial Technology, Republic of Korea

The ceramic 3D printing process mainly uses slurry as the form of a ceramic-polymer mixture, quality degradation due to shrinkage and deformation may occur during post heat treatment process for degreasing and sintering of the fabricated objects. As one of the methods for minimizing such a trouble, a high dispersion/density slurry preparation process is acting as an important technical factor. However, the existing physical process method using a ball mill or the like takes a long time to form a functional group on a surface of a ceramic powder and may cause deformation of its shape. Therefore, it is required suitable study on surface treatment process of ceramic powder as a basic step for establishing a rapid manufacturing system. In this study, a plasma-based rotational chamber system was suggested to form a uniform hydroxyl (OH-) functional group chemically on the surface of the Al_2O_3 powder within a short period of time and relevant experiments was carried out under each process condition. As an optimized samples, 10 g of ceramic powder was diluted in a 50 mL DI water solvent, and a sedimentation rate over time was observed. An optimum point of plasma process conditions for demonstrating the high dispersion/density slurry was present, and particularly, the process time was confirmed to be within 30 minutes.

11:10 AM

(ICACC-S11-006-2023) Intrinsic self-healing of $RE_2Si_2O_7/RE_2SiO_5$ (RE = Y, Ho, Gd) ceramic matrix composites for environmental barrier coatings

S. T. Nguyen*[1]; A. Okawa[2]; T. Nakayama[2]; T. Takahashi[1]; H. Suematsu[2]; K. Niihara[2]

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

Rare-earth silicates are promising materials for environmental barrier coatings, which are employed to protect SiC/SiC gas-turbine engine's blades under extreme environments. In this study, the composites of $RE_2Si_2O_7$ and RE_2SiO_5 (RE = Y, Ho, Gd) with various disilicate/monosilicate weight ratios were fabricated by pressure-less sintering in air. The effect of annealing at different temperatures (1100 - 1400 °C) and time (5 - 50 h) on the microstructural change and self crack healing ability of the composites were investigated. Some composites, such as $Y_2Si_2O_7/Y_2SiO_5$ exhibit an intrinsic crack healing behavior during high-temperature exposure. The healing mechanism is attributed to the compressive stress caused by thermal expansion in the $RE_2Si_2O_7/RE_2SiO_5$ matrix. Intrinsic self-healing ability that can heal the composite damages without using healing agent (SiC) is a key improvement for environmental barrier coating materials since they can avoid damages caused by excessive internal stress come from SiO_2's volume expansion when the turbine blades exposed to high-temperature at long hours.

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

Test Methods, Codes and Standards, Design Methodology, and Material Qualification

Room: Ballroom 4 (South Tower)
Session Chair: Gyanender Singh, University of Tennessee

8:30 AM

(ICACC-S13-024-2023) Compressive Strength of CMC Tubular Components for High-Temperature Reactors (HTR): Progress on ASTM Draft Standard for Compressive Axial Loading

M. G. Jenkins*[1]; J. E. Gallego[1]

1. Bothell Engineering and Science Technologies, USA

Plans by US DOE are to use advanced materials for the core and the reactor unit components in various advanced, high-temperature reactor concepts. SiC-SiC ceramic matrix composites (CMCs), in 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. 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 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.

8:50 AM

(ICACC-S13-025-2023) Nuclear Applications of SiC-SiC CMCs and Graphite: Design and Construction Rules in ASME BPV Code Section III, Division 5 for Nonmetallic Materials

M. G. Jenkins*[1]; S. T. Gonczy[2]; J. W. Geringer[3]; Y. Katoh[3]

1. Bothell Engineering and Science Technologies, USA
2. Gateway Materials Technology, USA
3. Oak Ridge National Laboratory, USA

US DOE is proposing to use SiC-SiC CMCs and graphite to enhance fuel performance and improve accident tolerance in high-temperature reactors (HTRs) because these materials are tolerant to the relevant irradiation and chemical environments. Because SiC-SiC CMCs and graphite are nonconventional materials, they are of special concern because the mission of the US Nuclear Regulatory Commission (NRC) is to license and regulate the nation's civilian 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 since 2008. For SiC-SiC CMCs to be incorporated into future HTRs, they must be included in ASME BPV Code as acceptable materials. A progress update is provided on the 2023 edition of Design and Construction Rules in ASME BPV Code that specifically address use of graphite and SiC-SiC CMCs in core and core components.

9:10 AM

(ICACC-S13-026-2023) Initial analysis of the ASME Code Rules for Composite Materials for HTR Design Requirements

J. W. Geringer*[1]; J. Podhiny[5]; S. T. Gonczy[2]; M. G. Jenkins[3]; W. Windes[6]; N. Gallego[4]; T. Koyanagi[4]

1. Oak Ridge National Lab, Materials Science and Technology, USA
2. Gateway Materials Technology, USA
3. Bothell Engineering and Science Technologies, USA
4. Oak Ridge National Laboratory, USA
5. Materials Research and Design Inc., USA
6. Idaho National Lab, USA

Ceramic Matrix Composites (CMCs) which include silicon-carbide silicon-carbide and carbon-carbon (C-C) composite materials, for the use of core components for high temperature reactors in the ASME Section III Division 5 code, were introduced due to their light weight, strength in extreme conditions, and in some instances great neutron irradiation resistance. It was recognized that integration with the aerospace industry needs to exist, however due to the nature and secrecy associated with the manufacturing and its uses (especially C-C applications), the integration efforts were not always optimal. The code relies heavily on industry to align itself in the nuclear environment to further improve on what it considers best practice. With the first publication of the CMC code rules under ASME III-5 HHB, it was known that it still needed some technical basis to support and demonstrate the newly developed rules. Focus on this topic will include discussion on optimization areas that have been identified since its first publication in support of the ASME Nonmetallic Design and Materials working group, as well as potential benchmark activities that are being proposed through current industry synergy. This work was supported by the U. S. DOE, Office of NE, under the ART and AFC programs. ORNL is managed by UT-Battelle, LLC under Contract No. DE-AC05-00OR22725 for the U.S. DOE.

9:30 AM

(ICACC-S13-027-2023) Infrared imaging and image-based modeling assessment of SiC/SiC cladding - Novel methods to assess the diffusivity and defects in composites

J. D. Arregui-Mena*[1]; T. Koyanagi[1]; H. Wang[1]; Y. Katoh[1]

1. Oak Ridge National Laboratory, USA

SiC/SiC composites are part of the novel strategies of accident-tolerant fuels that offer new advantages to the conventional materials used for fuel cladding materials. SiC/SiC cladding can potentially provide better safety margin improvements due to their irradiation resistance, low activation, and good thermomechanical properties. Moreover, the architecture of the SiC/SiC cladding can be tailored to withstand different reactor environments. In order to deploy this cladding technology, it is necessary to create new non-destructive techniques and modeling that help evaluate the performance of different microstructures and identify possible defects and irradiation effects. ORNL has adopted infrared imaging, x-ray computed tomography, and image-based simulations to assess the thermal diffusivity of SiC/SiC cladding. This infrared imaging technique employs a heat pulse mapped with an infrared camera to detect defects and derive the diffusivity of the cladding using a front surface flash approach. This research was complemented with x-ray computed tomography experiments and image-based modeling to assess the effectiveness of the experimental diffusivity measurements. These new non-destructive techniques will enable the routine survey of in-service SiC/SiC cladding and help determine the possible damage induced by the reactor environment.

Novel Ceramics and Composites for Nuclear Systems II

Room: Ballroom 4 (South Tower)
Session Chair: Patrick Burr, University of New South Wales

10:20 AM

(ICACC-S13-028-2023) High entropy hydrides for neutron moderators/shielding (Invited)

C. Moore[1]; S. C. Middleburgh*[1]; J. Astbury[2]

1. Bangor University, Nuclear Futures Institute, United Kingdom
2. Tokamak Energy, United Kingdom

Building from the growing body of high entropy alloy research experience and their differentiating properties, high entropy hydrides have been modelled and tested for the first time with the purpose of designing an optimised neutron shield/moderator for nuclear systems. Hydrides are expected to be efficient moderators and shielding materials due to their high concentration of hydrogen (a good moderator) and the inclusion of isotopes that absorb efficiently in the moderated neutron spectrum. The equiatomic $(TiZrNbHfTa)H_{0.4-2.0}$ high entropy alloy hydride has been modelled at the atomic scale and the phase transformations that occur during hydrogen absorption have been simulated. The thermodynamics of vacancy formation, hydrogen accommodation and hydride decomposition have been examined using density functional theory, linking results to experimental investigations. A model predicting the decomposition of the TiZrNbHfTaH system is proposed based on the temperature dependence of configurational and vibrational entropy terms and the hydrogen solution energies of individual interstitials. The neutronic properties and have also been modelled using a range of predictive tools. Microstructural considerations are now being considered to produce a robust material that can be thermally cycled with minimal degradation to its mechanical and neutronic properties. Progress and advances will be presented.

10:50 AM

(ICACC-S13-029-2023) Direct LiT Electrolysis in Molten Lithium (Invited)

C. S. Dandeneau*[1]; D. A. Hitchcock[1]; R. Rajeev[2]; S. Jadhav[2]; K. Brinkman[2]; B. L. Garcia-Diaz[1]

1. Savannah River National Lab, USA
2. Clemson University, Materials Science and Engineering, USA

Liquid metals (e.g., Li and Pb-Li) are attractive for use as plasma facing components and tritium breeding in fusion applications. In reactor designs where the breeding material is also employed as a coolant, nuclear heating is directly deposited inside the breeding material, thereby simplifying blanket configurations. However, tritium inventory in fusion reactors must be kept low for reliable and safe operation. For molten Li blankets, the high solubility of LiT in the metal makes tritium extraction challenging, and current tritium recovery processes require the addition of halide salts and use of expensive equipment with limited lifetimes. Savannah River National Laboratory (SRNL) has developed an alternative electrochemical extraction method involving direct LiT electrolysis in molten Li mixtures. The technique utilizes solid-state Li-ion conducting $Li_7La_3Zr_2O_{12}$ (LLZO) electrolytes with suitable stability in molten Li. LLZO powder was synthesized by both sol-gel and microwave solution combustion synthesis (SCS) methods; energy released in the SCS reactions led to appreciable crystallization of the LLZO prior to calcination. Cell electrochemistry was demonstrated through testing with high-aspect ratio Ga-doped LLZO electrolytes immersed in Li-LiD mixtures at 550 °C. The effects of impurities on cell performance and the stability of LLZO ceramics in Pb-Li mixtures was also evaluated.

11:20 AM

(ICACC-S13-030-2023) Hydrogen desorption kinetics in hafnium hydride

J. P. Pollard*[1]; J. Astbury[2]; F. Giuliani[1]; S. A. Humphry-Baker[1]

1. Imperial College London, Materials, United Kingdom
2. Tokamak Energy Ltd, United Kingdom

Hafnium hydride is a candidate neutron shielding material for compact tokamak fusion reactors as well as an advanced control rod material for fast fission reactors. However, a detailed investigation into the kinetics of hydrogen gas release at high temperature has not yet been undertaken. Here, we use thermogravimetric analysis to measure the H-desorption at high temperature from HfH_{2-x} powders. The mass loss follows a series of regimes that are correlated to the phase diagram using X-ray diffraction measurements and Scanning Electron Microscopy (SEM). SEM images taken from cross sectioned heat-treated powder show a core-shell desorption structure, indicating that the desorption is diffusion limited. A temperature-time desorption map has been produced to predict operating temperatures for Hf-H in a high temperature environment. Comparisons with other nuclear metal hydrides that are candidate moderator materials (e.g. Ti-H, Zr-H and Y-H) indicate significant challenges to HfH_{2-x} in terms of powder consolidation and reactor operation thermal excursions.

11:40 AM

(ICACC-S13-031-2023) Microstructural Optimisation of W_2B_{5-x} via Hot Pressing for High Energy Neutron Shielding

J. Davidson*[1]; J. Astbury[2]; S. A. Humphry-Baker[1]

1. Imperial College London, Materials, United Kingdom
2. Tokamak Energy Ltd, United Kingdom

Neutronics modelling of the W-B system indicates the stoichiometry of W_2B_5 as the optimum for shielding high energy neutrons in compact fusion engineering devices. Synthesis of W_2B_5 to full density is challenging owing to its intrinsically high flow stress at high temperature. We first address this challenge by exploring two different processing routes: (i) via commercial W_2B_5 powders and

(ii) via reaction sintering of pure W and pure B powders. The effect of the hot-pressing conditions (time, temperature, and atmosphere) and the pre-sintering conditions (green body density and impurities) are reported. We show that control of the oxygen content is critical to achieving high density. Second, we address the debate as to whether the common formula W_2B_5 is the most stable stoichiometry, or whether the material's stoichiometry is WB_2. This is important since it has a direct influence on the theoretical density and thus the neutronics performance of the material. We present both direct density measurements via gas pycnometry and indirect measurements via the characterisation of B:W ratio. For the latter we use Laser Induced Breakdown Spectroscopy (LIBS). This data is combined with lattice parameter measurements collected using XRD to determine theoretical density. The two methods are compared and differences discussed in terms of factors including changes in chemistry that occur during sintering.

S15: 7th International Symposium on Additive Manufacturing and 3-D Printing Technologies

Applications of Materials and Components

Room: Coquina Salon H (North Tower)
Session Chair: Soshu Kirihara, Osaka University

8:30 AM

(ICACC-S15-018-2023) Near-net-shape manufacturing of glasses for a wide range of applications (Invited)

J. Schilm*[1]; T. Moritz[1]; D. Wagner[1]; E. Schwarzer[1]; S. Weingarten[1]; J. Abel[1]; A. Mannschatz[1]; A. Mueller-Koehn[1]

1. Fraunhofer Gesellschaft, Institut für Keramische Technologien und Systeme, Germany

The Fraunhofer IKTS is active in the development of near-net-shape manufacturing processes for geometrically complex and functionalised glass components via conventional powder-technological manufacturing routes, such as injection moulding, or via additive processes, such as multi-material jetting (MMJ), fused filament fabrication (FFF) or vat photopolymerization (VPP), which also play an important role for high-performance ceramic components. In the first part, fundamentals and mechanisms of the sintering of glass powder compacts are discussed in close connection with effects leading to defects in sintered amorphous microstructures. The second part of the presentation provides an insight into the technical possibilities for manufacturing components that are individualised on the one hand and suitable for large-scale production on the other, from various glasses that have sharp edges or microstructures without having to use expensive or grinding and environmentally hazardous wet etching processes. Also aspects of multi-material processing by injection moulding and additive jetting technology are adressed. The range of properties of the manufactured sintered glass components extends from electrically conductive/insulating to luminescent to permanently colourfast.

9:00 AM

(ICACC-S15-019-2023) 3D Printed Polishing Tools Towards Precision Engineering: Design, Fabrication and Performance (Invited)

H. Wang*[1]

1. National University of Singapore, Mechanical Engineering, Singapore

3D printing (3DP) technology has been facilitating the manufacturing of unprecedented materials and structures, which is reshaping the work and life of human beings. Functionally-graded lattice structure (FGLS) is one of those most fascinating products being readily fabricated by 3DP. It has advantages such as lightweight, on-demand stiffness, and superior energy absorption. These features qualify FGLS a promising candidate for precision polishing tools. This talk will present a novel design of polishing tools with variable stiffness on their functional surfaces based on 3DP FGLS. The contact and deformation statuses of these tools are investigated by the finite element method. The printing accuracy is evaluated by micro X-ray computed tomography. Polishing experiments show that the designed tools can yield desired polishing footprints on both ductile and brittle materials. The surface finish is improved by up to 97% after polishing. This work extends the application of 3DP to precision engineering industries and provides a promising technique to finish optical components, mould and die, engine blades, etc.

9:30 AM

(ICACC-S15-020-2023) Non-oxide Ceramic Synthesis using Laser-Induced Reaction Bonding (Invited)

J. B. Spicer*[1]; A. B. Peters[1]; D. Zhang[2]; D. Nagle[3]; A. Hernandez[1]; C. Wang[1]; T. Mueller[1]

1. Johns Hopkins University, Materials Science and Engineering, USA
2. Johns Hopkins University Applied Physics Laboratory, USA
3. Johns Hopkins University, Energetics Research Group, USA

This work explores selective laser reaction sintering (SLRS) and laser-induced, reaction bonding methods for production of near net-shape ultrahigh temperature ceramics (UHTCs) such as HfC, ZrC, TiC, HfN, ZrN, and TiN. Group IV transition metal and metal oxide precursor materials were chemically converted and reaction-bonded into layers of UHTCs using single-step selective laser processing in reactive atmospheres. SLRS processing of metal or metal oxide alone produced near-stoichiometric UHTC phases with high yields of carbides and nitrides; however, when either a metal or a metal oxide was used, volumetric changes that occurred during conversion resulted in residual stresses that ultimately affected the mechanical integrity of the ceramic product. To mitigate these conversion-induced stresses, composite metal/metal oxide precursors were formulated to compensate for the volume changes of the individual precursor components. Composite formulations reduced conversion stresses, but interparticle adhesion must occur as well in order to produce robust UHTC layers and this requires surface diffusion. Computational simulations of carbon and nitrogen diffusion in host transition metal lattices were used to interpret experimental results where conversion might have inhibited interparticle adhesion. Results show that this reactive approach may be viable for the additive manufacturing of various UHTC materials.

Multi-Material and Hybrid Printing Techniques

Room: Coquina Salon H (North Tower)
Session Chair: Hao Wang, National University of Singapore

10:20 AM

(ICACC-S15-021-2023) Photonic sintering of ceramics applied to additive manufacturing for a single step process

N. Somers*[1]; A. Monton[1]; M. D. Losego[1]

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

Additive Manufacturing (AM) of ceramics allows to produce functional ceramic parts with geometrical complexity that have utility in fields including electronics, medicine, aerospace, and sustainable energy. Recently, photonic sintering of ceramics has been studied using ultra-rapid heating with intense light. Xenon Flash lamps can produce intense, ultrashort light pulses over a broad spectral band with the potential to supply extremely fast heating rates. Various resesarchers have investigated this photonic technology to sinter ceramic thin films (1) and ceramic bulk samples (2), opening a new route to scalable, fast, efficient and versatile sintering. Here we investigate whether photonic sintering of ceramics can be used to develop a single-step AM process. The idea is to deposit thin ceramic layers by spraying, aerosol jet printing, or material extrusion and then sinter them by flash lamp annealing prior to deposition of the subsequent layer. These steps can be repeated layer by layer to produce a ceramic 3D part without the need of a post-treatment. Up to now, the use of photoactive and melting additives have allowed to induce a liquid phase sintering of Al_2O_3 particles with flash lamp annealing (10 minutes treatment) as well with infrared irradiation (20 minutes treatment). The affected thickness varies between 15 and 25 µm depending on the irradiation parameters and compositions.

10:40 AM

(ICACC-S15-022-2023) Multi-material Additive Manufacturing and Characterization of Lightweight Polymeric Materials

V. Vakharia*[1]; M. Singh[2]; M. C. Halbig[1]

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

Innovations in 3-D printing technology have created a demand for multifunctional composites. A study into the properties of multi-material bi-layered composites manufactured using fused filament fabrication (FFF) was conducted. A multi-material system of ABS (acrylonitrile butadiene styrene) and ABS with chopped carbon fibers was printed with different layering patterns. The elastic modulus of multi-material composites of ABS and ABS with chopped carbon fibers was 10-30% higher than that of standard ABS alone. A similar study of the high-temperature polymer, ULTEM (an amorphous thermoplastic polyetherimide), was performed. Advanced polymers like ULTEM and TCPoly can create robust, lightweight, and efficient structures while providing high-temperature stability and thermal conductivity. These filaments were printed together to create a multi-material part. The high-temperature multi-materials' tensile strength and thermal conductivity were comparable to aluminum and stainless steel, respectively. The microstructure of fractured surfaces and polished cross-sections were imaged. In the high-temperature multi-materials, layers printed closer to the heated bed were of higher quality than those closer to the top of the finished part. These results demonstrate the potential for designing multi-material printed parts that provide multifunctionality without sacrificing mechanical properties.

11:00 AM

(ICACC-S15-023-2023) 3DPrinting, Several technologies in One, a new machine to shape ceramics

R. Svintsitski*[1]; R. Gaignon[1]

1. 3DCERAM SINTO, France

Today, there are many ways to shape materials among which additive manufacturing. Until now, the 3D printing has been identified as interesting in the framework of prototyping. On another hand, stereolithography is known as the best technology to get the best quality after sintering. 3DCS has developed two new printers which will fit the dreaming tools for research centers: New hybrid machines which could combine two different 3D technologies, one will be our stereolithography and the second one would be co-developed with the research center. We put our know-how and our machines at the service of research centers to allow them to integrate the technologies that they themselves have developed into our machines. Our goal is to open our machines to become technological hosts. A Multi Additive Technologies (M.A.T.) printer which would be able to host several technologies in the same frame to combine complementary skills. This new way of conceiving the development of 3D machines opens up perspectives that will be revealed as needs arise. With the new M.A.T. machines, we are creating a favorable ground for a multi-technological deployment.

11:20 AM

(ICACC-S15-024-2023) How to produce a wide range of complex-shaped ceramic materials from selective laser melting of polymers

M. Pelanconi*[1]; P. Colombo[2]; A. Ortona[1]

1. SUPSI, Department of Innovative Technologies, Switzerland
2. University of Padova, Industrial Engineering, Italy

The fabrication of ceramic components by a novel hybrid additive manufacturing process was investigated. Selective laser sintering of polyamide powders was used to 3D print a polymeric preform with controlled relative density, which allows manufacturing geometrically complex parts with small features, such as cellular architectures. Preceramic polymer infiltrations with a wide range of precursors followed by pyrolysis (PIP) were used to convert the preforms into different ceramics, and other PIP cycles were performed to increase the relative density of the parts. A comparison in terms of relative density, mechanical strength and heat exchange performance was performed for the converted ceramics. Then, the final densification was achieved via non-reactive and reactive liquid silicon infiltration (LSI) obtaining complex ceramic components without defects and shape distortion. The crystallization of the previously generated ceramic phase, with associated volume change, allowed to fully infiltrate the part leading to an almost fully dense material. The advantage of this approach is the possibility of manufacturing ceramics directly from the preceramic precursor, without the need of ceramic powders. This is an alternative AM approach to Binder Jetting for the production of complex parts due to its finer resolution.

11:40 AM

(ICACC-S15-025-2023) SiC-SiC Composites Joined by Embedded-Wire CVD, as an Extension of LCVD-based Additive Manufacturing Techniques

M. C. Schaefer*[1]

1. Free Form Fibers, USA

Embedded-Wire CVD (EWCVD) was developed by FFF as an innovative approach to produce high purity, high density CMC structures. This technique builds upon our previous work with Laser-CVD (LCVD), utilizing dimensionality of mass-transport and its large influence on CVD/CVI. Our development of the LCVD process has been analogous to An important application of EWCVD is in the long-sought goal of accomplishing joining (welding) of ceramic and ceramic-based composite components together. FFF has demonstrated homogeneous joining of SiC fiber-reinforced SiC matrix composites without the use of extraneous materials that limit the performance of the joint structure. EWCVD has also been implemented in the development of SiC thin wall composite shells for the replacement of metal-based cladding to contain uranium oxide fuel pellets in nuclear power applications. SiC is currently a candidate for nuclear fuel cladding as either a monolith, a SiC fiber-reinforced SiC matrix (SiC/SiC) composite, or a hybrid of metal and SiC/SiC composite, utilizing our LCVD SiC fibers. EWCVD is also being deployed to address this issue and form a fully dense, hermetic end cap seal. Further extension of the EWCVD technology to other thin shell applications in the aerospace field are also being explored.

S16: Geopolymers, Inorganic Polymers and Sustainable Construction Materials

Mechanical Properties of Geopolymer Composites I

Room: Coquina Salon C (North Tower)
Session Chair: Mohsen Issa, University of Illinois at Chicago

8:30 AM

(ICACC-S16-016-2023) Influence of Nanomaterials on the Structure and Performance of Metakaolin-Based Geopolymers – State-of-the-art and Lessons Learned (Invited)

A. Akono*[1]

1. Northwestern University, Civil and Environmental Engineering, USA

Geopolymers are x-ray amorphous inorganic polymers that are synthesized by mixing a potassium or sodium silicate solution with synthetic metakaolin. In this talk, we investigate the influence of nanomaterials such as carbon nanofibers, multiwalled carbon nanotubes, graphene nanoplatelets, and boron nitride nanotube on the structure and mechanical properties of geopolymers. To this end, we employ advanced methods such as statistical nanoindentation, microscopic scratch testing, mercury intrusion porosimetry, high-resolution scanning electron microscopy, and transmission electron microscopy. Specifically, we found that carbon-based nanomaterials influence the geopolymerization reaction directly at the molecular scale while preserving the amorphous nature of geopolymer. We observed strengthening of the inner solid skeleton along with a change in pore structure at both the nanometer and micrometer length scales. These molecular modifications lead to an enhancement in mechanical properties such as elasticity, strength, and fracture toughness. Our results pave the way towards novel nanostructured and multifunctional enhanced-performance geopolymer composites. This work was supported by the National Science Foundation under Grant No. CMMI 1928702.

9:00 AM

(ICACC-S16-017-2023) Modeling the compressive strength of metakaolin-based geopolymers (Invited)

M. Muracchioli*[1]; M. D'Agostini[1]; G. Franchin[1]; G. Menardi[2]; P. Colombo[1]

1. University of Padova, Industrial Engineering, Italy
2. University of Padova, Statistical Sciences, Italy

Geopolymers represent an innovative green binder that can compete with ordinary Portland cement as a structural material. In order to enable their adoption, however, there is a need to standardize the manufacturing process and the testing process. As for now, there are studies assessing the influence of some properties on the compressive strength, but they typically use a very limited number of samples and conditions, which makes the results not very representative. Moreover, studies carried out by different research groups are very difficult, if not impossible, to compare, considering the variability in experimental conditions. There is a lack of extensive and comprehensive statistical studies, like the one we carried out, that simultaneously take into consideration both chemical (i.e. composition) and processing aspects (i.e. aging-curing condition and curing temperature). With this work, we aimed at addressing this issue and at proposing a predictive model that could inform the geopolymer production.

9:30 AM

(ICACC-S16-018-2023) The mechanical efficiency of distinct geopolymer composites reinforced with aggregates and fibers (Invited)

A. C. Trindade*[1]; W. M. Kriven[2]

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

Geopolymers (GP) correspond to brittle inorganic polymeric materials considered to replace cement-based and high-tech ceramics by using simpler manufacturing processes, in a more sustainable and long-term manner. Several formulations have been proposed for a range of precursor's types and alkali solutions. Still, their fragile nature arises as one of the most challenging aspects. For this reason, the aim of this study is to present suitable reinforcements to enhance the material reliability through a proper tailoring of their ductility and cracking patterns. For this, a potassium-based GP was used as main binder and reinforced with different contents of particles, as well as 20-mm long steel, basalt and glass fibers. Their mechanical efficiency was verified through compression, flexural, splitting tensile, toughness, and shear testing. Digital Image Correlation (DIC) and scanning electron microscopy (SEM) techniques were used to further investigate the cracking mechanisms. In general, both particulate reinforcements enhanced the material strength, without affecting its brittle nature. The use of steel fibers resulted in the highest responses, up to 87 MPa in compression and 12 MPa in shear strength. The basalt fibers, despite showing slightly reduced values, were able to reach a multiple cracking performance, while the glass fibers did not improve the material properties.

Mechanical Properties of Geopolymer Composites II

Room: Coquina Salon C (North Tower)
Session Chairs: Waltraud Kriven, University of Illinois at Urbana-Champaign; Ana Carolina Trindade, University of Illinois at Urbana-Champaign

10:20 AM

(ICACC-S16-019-2023) On the effect of adding reinforcements to prevent brittle geopolymer failure: A study on composition, reinforcement type, specimen scale and geometry

A. C. Trindade*[1]; W. M. Kriven[2]

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

Geopolymers (GP) are inorganic polymeric materials that present intermediate features to those of cements and ceramics, with clear benefits in comparison to both groups. Regarding cements, they can reach their ultimate mechanical capacity in much shorter curing times, reducing temperature exposure and long-term degradation issues, due to a more stable microstructure. Yet, single phase GP are fragile materials that require the inclusion of reinforcements for their safer use in the face of high efficiency demands, such as earthquakes and such. They can be reinforced with particles and fibers, resulting in variable and not comparable effects to other composite materials. This variability prevents the standardizations necessary for the wide dissemination of the technology. Therefore, the goal of this study was to present a detailed investigation on the use of different reinforcements in Na and K-based GP mixes. The specimen geometries and sizes were compared after compressive testing, coupled with image acquisition, allowing further evaluation of the role of each variable on its deformation. In general, single phase GP demonstrated a brittle behavior with spalling effects, while the inclusion of aggregates and fibers improved their strength by keeping the specimen parts in place until ultimate failure.

10:40 AM

(ICACC-S16-020-2023) Metakaolin-based geopolymer matrix design for higher flexural strength (Invited)

R. A. Sa Ribeiro*[1]; M. G. Sá Ribeiro[2]; D. Samuel[3]; A. Ozer[3]; W. M. Kriven[4]

1. INPA-National Institute for Amazonian Research, Green Building and Engineering Laboratory, Brazil
2. Bionorte Network, Biodiversity and Biotechnology, Brazil
3. University of Illinois at Urbana-Champaign, Materials Science and Engineering, USA
4. University of Illinois at Urbana-Champaign, USA

The geopolymer matrix plays an important role in the design of high strength composites. The matrix design phase is aimed at meeting requirements in fundamental engineering properties such as flexural strength. Design variables such as types of ingredients, mix proportions, curing regime are optimized to achieve required performances. Much work has been done on laboratory-made waterglass to activate metakaolin-based geopolymers (MKGP). This study focused on commercial waterglasses to activate MKGP for higher flexural strength in sustainable construction applications. The following geopolymer matrix preliminary design for higher flexure strength was devised: one type of commercial metakaolin; six commercial waterglass formulations; a single low energy geopolymer processing. PQ Sodium Silicate D with 13 M of water and Metamax MKGP resulted in the highest strength matrix, comparable to PQ Sodium Silicate MetsoBeads 2048 with 13 M of water.

11:00 AM

(ICACC-S16-021-2023) Optimization of Workability and Strength of Metakaolin-based Geopolymer Concrete Mixture: An Experimental Study of Railroad GPC Crossties Prestressed (Invited)

G. K. Al-Chaar[2]; M. A. Issa*[1]; M. Kassem[1]; A. Saroufim[1]; M. Mahdi[1]

1. University of Illinois at Chicago, Civil, Materials, and Environmental Engineering, USA
2. US Army Corps of Engineers, Research and Development Center, USA

The usage of concrete ties is expanding in North America today as they have become a cost-effective replacement for the traditional wood ties used in industry, outperforming their rivals in terms of durability and capacity. Prestressed concrete crossties are considered a more sustainable and durable substitute for conventional wooden ties in current railroad construction projects. Moreover, using fiber-reinforced polymer (FRP) composites as prestressing material has usually been considered where it is an environmentally friendly material with better corrosion resistance and freeze and thaw cycles than conventional steel reinforcement, attracting attention to the use of this material in special structures. Geopolymer concrete is a substitute for conventional concrete, reducing the CO_2 emission caused by the cement industry. Studies show that geopolymers have excellent physical properties, high early strength, and low shrinkage. Geopolymers are majorly produced from metakaolin, fly ash, kaolin, and slag. Metakaolin-based Geopolymers have remarkable strength, excellent mechanical properties, and high resistance against acid and alkali corrosion.

11:30 AM

(ICACC-S16-022-2023) Fatigue Response of Metakaolin-Based Geopolymer (Invited)

A. Akono*[1]

1. Northwestern University, Civil and Environmental Engineering, USA

Fatigue is the propagation of fractures in a material under an applied repetitive loading at loads well below the catastrophic level. Fatigue failure occurs in three stages: crack nucleation or regime I, crack propagation or regime II, and catastrophic fracture or regime III. Our research objective is to understand the intrinsic fracture response of metakaolin-based geopolymer. To this end, we employ depth-based sensing methods. First, we rely on cyclic indentation, where a load is periodically applied using a Berkovich indenter. A decrease in the indentation hardness is observed as a function of the number of loading cycles, maximum vertical load, and load increment. Compared to homogenous materials such as glasses and polymers, a higher decrease in indentation hardness is observed in metakaolin-based geopolymers. Using fatigue mechanics modeling, we then calculate the cycles to failures as a function of the maximum applied load. In future studies, we will apply Paris fatigue's law to characterize the fatigue crack propagation law of geopolymers. These studies are important to understand the durability of geopolymer composites. This research was supported by the National Science Foundation under Grant No. DMR 1928702.

S17: Advanced Ceramic Materials and Processing for Photonics and Energy

Multi-functional Materials III

Room: Coquina Salon G (North Tower)
Session Chairs: Fiorenzo Vetrone, Institut National de la Recherche Scientifique, Université du Québec; Farid Akhtar, Lulea University of Technology

8:30 AM

(ICACC-S17-022-2023) Structured porous ammonia carriers for seasonal energy storage (Invited)

F. Akhtar*[1]

1. Lulea University of Technology, Division of Materials Science, Sweden

The objective of the research is to establish safe storage of seasonal energy in form of green ammonia using structured sorbents. We will present the structuring of porous sorbents and alkaline earth metal halides (AEMHs) as ammonia carriers with superior sorption kinetics and structural stability. AEMHs are structured using carbon materials to overcome issues of melting and volume swing during sorption. We show that the incorporation of 20 wt. % carbon materials into (AEMH) $MgCl_2$, the melting spread of the $MgCl_2$ is prevented during the ammonia sorption to reduce the structural risk of using alkaline earth metal halides (AEMHs) as ammonia carriers. Moreover, the ammonia kinetics of the 20 wt.% graphene nanoplatelets aggregates (GNA) -80 wt.% $MgCl_2$ composite shows an increase of 83% and 73% in ammonia sorption and desorption, respectively. The volume expansion and shrinkage of the AEMHs as ammonia carriers are as large as 400% and can be countermined using novel design and fabrication of porous $SrCl_2$ porous structures, scaffolded by reduced graphene oxides (rGO) networks. The porous $SrCl_2$ structure maintains the macro- and micro-structure accommodating the volume swing after 20 ammonia absorption–desorption cycles. Moreover, the porous 80 wt% $SrCl_2$-rGO composite possesses rapid ammonia absorption–desorption kinetics, 1.4 times faster in absorption and 5.4 times faster in desorption compared with pure $SrCl_2$.

9:00 AM

(ICACC-S17-023-2023) Biomimetic Advanced materials for Photonics and Energy applications (Invited)

V. M. Castano*[1]

1. Universidad Nacional Autonoma de Mexico, Mexico

A review of how Nature produces materiasl, in particular ceramics, will be offered. Several examples of Nature.inspired materials will be discussed, including solar cells, optical switches, energy conversion etc.

9:30 AM

(ICACC-S17-024-2023) Design of photoactive inorganic nanosystems for energy and environmental applications (Invited)

E. Moretti*[1]; L. Liccardo[1]; M. Bordin[1]; A. Vomiero[2]

1. Ca' Foscari University of Venice, Department of Molecular Sciences and Nanosystems, Italy
2. Lulea University of Technology, Engineering Sciences & Mathematics, Sweden

In the broad scenario of multi-component nanosystems, oxide-based materials are of primary importance for many different applications of current interest, spanning from catalysis to photovoltaics, water remediation and energy conversion and storage. Such a wide range of applications can be tailored by means of suitable synthetic methodologies. In particular, tuning the morphological features of a material has emerged as an important strategy to improve its performance, and there has been extensive research to develop highly active oxide-based systems rationally designed with a controlled shape and an ordered porosity at the nano/microscale for energy and environmental applications. This talk will focus on the importance of tuning the morphological features of a catalyst as a strategy to improve the photocatalytic activity, focusing on how rationally designing inorganic materials at the nanoscale can lead to morphologies and structures suitable to enhance the catalytic performance of industrially and environmentally important processes. The talk will discuss some applications that can be addressed by multi-component systems, synthesized via the bottom–up approach, highlighting their structure-reactivity relationship. H_2 production and purification, and drugs photodegradation will be presented as successful cases history.

10:20 AM

(ICACC-S17-025-2023) Rare Earth Doped Nanoparticles: Design, Architecture and Applications (Invited)

F. Vetrone*[1]

1. INRS, Université du Québec, Centre Énergie, Matériaux et Télécommunications, Canada

In the last decade, the field of rare earth doped nanoparticles has vastly matured, progressing from the basic understanding of the photophysical properties governing their nanoscale luminescence, to their use in the study of a number of fundamental properties and ultimately to a plethora 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. Therefore, with a single NIR excitation wavelength, it is possible to observe anti-Stokes emission, known as upconversion, or single photon (Stokes) NIR emission. Here, we present our work on the synthesis and surface functionalization of various NIR excited (and emitting) core/shell rare earth doped nanostructures/nanoplatforms and demonstrate how their various emissions could be harnessed for applications in biology and nanomedicine. Moreover, we investigate how these functional nanoparticles could be rendered even more versatile through rational combination with other NIR excited optical nanostructures.

10:50 AM

(ICACC-S17-026-2023) Towards tunable mid-infrared detection for advanced optical imaging with epitaxial graphene on silicon carbide on silicon (Invited)

F. Iacopi*[1]

1. University of Technology Sydney, Faculty of Engineering & IT, Australia

We have pioneered a method to obtain large-scale, consistent epitaxial growth of graphene on cubic silicon carbide on silicon pseudo-substrates using a solid-source, catalytic alloy approach. This platform allows to fabricate any complex graphene-coated silicon carbide 3D nanostructures selectively at the wafer-scale with a comparable electrical conductivity to that of graphene on hexagonal SiC wafers. This epitaxial graphene on 3C-SiC platform is of particular interest for integrated optics and nanophotonics. Thanks to the hybridization of surface plasmons in the graphene with the surface phonons supported by the carbide, graphene-coated SiC nanostructures can lead to low loss MIR response with high confinement. We have recently demonstrated an enhanced MIR absorption using graphene/SiC nanowires and indicated the capability for strong field confinement within a nm-thin oxide spacer between the graphene and the core SiC of the nanowire. Based on both analytical methods and numerical simulations, we have also indicated that, correspondingly, the light-matter interaction in the MIR can also be strongly enhanced by using a 3-layer nanoparticle made of a polar core, a low refractive index spacer and a graphene shell. We will illustrate an example of metasurface as an all-optical spatial filter for phase -contrast imaging.

S18: Ultra-High Temperature Ceramics

Processing-Microstructure-Property Relationship II

Room: Coquina Salon A (North Tower)
Session Chair: Scott McCormack, University of California, Davis

8:30 AM

(ICACC-S18-024-2023) Uncertainty quantification and processing optimization for UHTC manufacturing through an ICME framework (Invited)

R. Swanson[1]; A. Hilmas[2]; H. Babaee[4]; W. Xiong[4]; W. Fahrenholtz[3]; S. J. McCormack*[1]

1. University of California, Davis, Materials Science and Engineering, USA
2. Air Force Research Lab, USA
3. Missouri University of Science & Technology, Dept. of Materials Science and Engineering, USA
4. University of Pittsburgh, Mechanical and Materials Science, USA

Standard ultra-high temperature ceramic (UHTC) manufacturing results in components with large differences in properties due to variability in microstructural "critical flaw" distributions. Critical flaws can be any irregularity in a component, such as a secondary phase, inclusion, cracks, pores etc. This is problematic when designing reproducible UHTC components. The goal of this project is to build a probabilistic characterization for processing-structure-properties (PSP) parameters and link them at each stage of UHTC processing in a way that allows for uncertainty propagation. This methodology has not been performed in the past due to the complex interrelations of UHTC PSP parameters that need to be deconvolved. Thus, multi-fidelity PSP database development and effective integrated computational materials engineering (ICME) combined with statistical modeling is key to minimize uncertainty during UHTC manufacturing. ZrB_2 will be used as a case study, detailing the construction of a PSP database from literature data and how high-fidelity 3D microstructural data will be used to reduce uncertainty along with other types of data.

9:00 AM

(ICACC-S18-025-2023) Effect of mechanical activation on carbothermal synthesis and densification of ZrC

N. Obradovic*[1]; S. Filipovic[1]; L. Feng[2]; M. Mirkovic[3]; W. Fahrenholtz[4]

1. Institute of technical sciences of SASA, Materials, Serbia
2. Missouri University of Science & Technology, USA
3. University of Belgrade, "Vinca" Institute of Nuclear Sciences-National Institute of the Republic of Serbia, Materials, Serbia
4. Missouri University of Science & Technology, Dept. of Materials Science and Engineering, USA

Mixtures of ZrO_2 and C were prepared by high-energy ball milling. Powders were milled for times from 0 to 120 minutes in air atmosphere. As milling time increased, surface area of powders increased, indicating significant particle size reduction. Milled powders were densified by spark plasma sintering at 2000 °C. Unmilled powders did not reach full density. Milled powders reached full density, but ZrO_2 impurities were found for specimens prepared from powders milled for 60 and 120 minutes. Microstructure analysis showed that grain size was less than 2 microns for powder milled for 15 minutes. Based on densification data and impurities level, milling time of 15 minutes appears to give the best balance of particle size reduction to promote densification while minimizing impurities level.

9:20 AM

(ICACC-S18-026-2023) Gel casting elaboration process for porous alumina ceramics with complex shapes

Y. Belrhiti*[1]; P. Kerth[2]; M. McGilvray[2]; L. J. Vandeperre[1]

1. Imperial College London, Materials, United Kingdom
2. University of Oxford, Engineering science, United Kingdom

Thanks to their excellent thermomechanical properties while moving to high porosity, porous Al_2O_3 are good candidates for transpiration cooling. This technology is used between the hypersonic flight component and the hot freestream flow to reduce the heat flux to the material in a hostile environment. Gelcasting is the elaboration process selected to manufacture porous Al_2O_3 ceramics with complex shapes and homogeneous properties. It allows obtaining materials with relative ease and high green mechanical properties, however, the drying step without damage or crack apparition remains challenging. Indeed, porous materials with low loading powder and complex shapes are particularly difficult to properly dry without internal stresses. Decreasing solid loading in the porous structure of the gel cast part induces a higher water potential and the low solid particle packing is equivalent to low resistance to internal moisture diffusing to the surface of the gel cast part. Besides, the ceramic final shape complexity may present a nonuniform and differential drying, so mechanical stress apparition is caused by non-uniform shrinkage. For this reason, the drying step is optimized to avoid cracking before sintering.

9:40 AM

(ICACC-S18-027-2023) Thermal and Electrical Properties of $(Ti,Cr)B_2$ Ceramics

S. M. Smith*[1]; L. Feng[1]; W. Fahrenholtz[1]; L. Silvestroni[2]

1. Missouri University of Science & Technology, Dept. of Materials Science and Engineering, USA
2. CNR, ISTEC, Italy

Chromium additions change the densification behavior, grain size, Vickers hardness, and flexural strength of $(Ti,Cr)B_2$ ceramics. Hardness and strength of the $(Ti,Cr)B_2$ ceramics were highest for a Cr content of 10 at%. The goals of the present study were to determine if the effect of Cr solid solution extends to the thermal and electrical properties of $(Ti,Cr)B_2$ ceramics and whether Cr content affects the anisotropy in the coefficient of thermal expansion (CTE). $(Ti,Cr)B_2$ ceramics containing varying amounts of Cr solid solution were synthesized by boro/carbothermal reduction. Powders were consolidated by spark plasma sintering. The CTE was determined for the principle crystallographic directions by variable temperature X-ray diffraction. Thermal diffusivity of sintered samples was measured by the laser flash method and was used to calculate thermal conductivity. The electrical resistivity was measured using the 4-point linear probe method. The discussion will focus on identifying any systematic changes in properties that occur as the Cr content of the $(Ti,Cr)B_2$ ceramics changes.

Response in Extreme Environments II

Room: Coquina Salon A (North Tower)
Session Chair: Elizabeth Opila, University of Virginia

10:20 AM

(ICACC-S18-028-2023) Oxidation mechanisms of developmental refractory materials (Invited)

C. Stephens[1]; L. Backman[2]; C. Brandenburg[1]; E. J. Opila*[1]

1. University of Virginia, Materials Science and Engineering, USA
2. U.S. Naval Research Laboratory, Spacecraft Engineering Division, USA

Materials for long-term application in high temperature oxidizing environments have relied on formation of protective Cr_2O_3, Al_2O_3, and SiO_2 scales. These materials are limited to use temperatures of about 1500°C. A need exists for refractory materials for use in oxidizing environments at temperatures > 1500°C. Refractory alloys and ultra-high temperature ceramics have been proposed for these use conditions. While much effort has been expended to develop these materials, a lack of both fundamental understanding of their oxidation mechanisms and methods to improve their oxidation resistance exists. Furthermore, the advantages and disadvantages of utilizing refractory metals, alloys, or carbides have not been completely elucidated. In this study, refractory metals and their corresponding carbides were oxidized under the same conditions. Additionally, oxidation of multicomponent alloys and carbides containing, Ti, Zr, Hf, Nb, and Ta were studied. Short term oxidation studies (2 minutes to 20 hours) were conducted at temperatures from 900 to 1800°C in 1% O_2/balance argon using resistive heating or thermogravimetric analysis. Weight change, oxide thickness, and material consumption rates with microstructural characterization were used to elucidate oxidation mechanisms including oxygen dissolution in alloys, CO(g) production from carbides, and complex oxide formation from multicomponent materials.

10:50 AM

(ICACC-S18-029-2023) Study of Ceramic Composites High-Temperature Response in Aerospace High-Enthalpy Flows (Invited)

S. Mungiguerra*[1]; A. Cecere[1]; R. Savino[1]

1. University of Naples Federico II, Department of Industrial Engineering, Italy

Interest for new-generation reusable space vehicles is spreading worldwide, calling for novel materials able to survive the extreme conditions of hypersonic atmospheric re-entry and rocket propulsion. Among the most promising candidates are the Ultra-High-Temperature Ceramics (UHTC), transition metal borides and carbides, which excel for their high-temperature properties, e.g. oxidation and ablation resistance. Research is increasingly oriented towards the introduction of secondary phases, such as silicon carbide and carbon fibers reinforcement (Ultra-High-Temperature Ceramic Matrix Composites, UHTCMC), to improve the materials performance. The European H2020 C^3HARME research project was aimed at development and characterization of new UHTCMCs for near-zero ablation TPS and near-zero erosion rocket nozzles. The University of Naples Federico II, having long-term expertise in aero-thermo-dynamics and propulsion, was in charge for requirements definition, prototypes and test condition design, and carried out extensive test campaigns to characterize the thermal response of the samples in an arc-jet plasma wind tunnel and a lab-scale hybrid rocket engine. Numerical computations of fluid-dynamic flowfields and materials thermal behavior complemented the test activities. The results supported development and testing of full-scale TPS assemblies and a large-size solid rocket nozzle.

11:20 AM

(ICACC-S18-030-2023) Oxidation resistance of polymer-derived (Hf,Ta)C/SiC ceramic monoliths

N. Petry*[1]; N. Thor[2]; J. Bernauer[3]; Q. Wen[4]; A. S. Ulrich[1]; E. Ionescu[5]; H. Kleebe[3]; M. C. Galetz[1]; M. Lepple[6]

1. DECHEMA-Forschungsinstitut, Materials and Corrosion, Germany
2. Technical University Darmstadt, Institute of Applied Geosciences, Germany
3. Technical University Darmstadt, Institute of Materials Science, Germany
4. Central South University, Powder Metallurgy Research Institute, China
5. Fraunhofer Research Institution for Materials Recycling and Resource Strategies IWKS, Germany
6. Justus-Liebig-University Giessen, Institute of Inorganic and Analytical Chemistry, Germany

Polymer-derived UHTC materials have received increasing attention in the last few years, due to their versatile processability. The polymeric precursors are liquid or can be dissolved in organic solvents, which allows the usage of common polymer deposition techniques namely dip, spray or spin coating. Applications of such coatings can be thermal (TBCs) or environmental barrier coatings (EBCs). Typically, UHTC materials show poor oxidation resistance in the temperature range from 400 °C to 1600 °C, which can be improved by adding a secondary SiO_2-forming phase. Using Si-based polymeric precursors modified with metalorganic compounds enables the production of polymer-derived ceramic nanocomposites (PDC-NCs) consisting of UHTC phase finely distributed in a silica-forming matrix such as SiC. In this study two PDC-NCs were synthesized, namely $Si(Hf_{0.7}Ta_{0.3})C$ and $Si(Hf_{0.2}Ta_{0.8})C$, to examine the influence of the chemical composition on the oxidation resistance, which is a crucial property for the application as an EBC/TBC material. Dense monolithic bulk samples were prepared by SPS leading to a microstructure of $(Hf_xTa_{1-x})C$ dispersed within a SiC matrix. TGA was used to investigate the oxidation kinetics at 1200 °C, 1400 °C, and 1600 °C for 50 h and 100 h. Extensive microstructural investigations using various methods such as EPMA and TEM explain the significantly enhanced oxidation resistance compared to pure SiC.

11:40 AM

(ICACC-S18-031-2023) Effect of HfO_2 coatings on the oxidation behavior of ZrB_2

J. E. Förster*[1]; R. Naraparaju[2]; W. Fahrenholtz[3]; G. Hilmas[3]

1. DLR - German Aerospace Center, Institute of Materials Research, Germany
2. DLR - German Aerospace Center, Materials Research, Germany
3. Missouri University of Science & Technology, Dept. of Materials Science and Engineering, USA

Zirconium diboride (ZrB_2) is a highly investigated material from the class of ultra-high temperature ceramics (UHTCs) due to its rare combination of a high melting point, low density, and high electrical/thermal conductivity. Hence, ZrB_2 has a high potential for the space- and aerospace industry as thermal protective shields (TPS) or leading edges of hypersonic vehicles. Unfortunately, the low oxidation resistance in oxidative atmospheres above 1400°C limits sustained usability and is a drawback for such kind of applications, forming unprotective zirconium oxide (ZrO_2) and boria (B_2O_3). The formation of a dense mixed oxide scale as a result of oxidation on top of ZrB_2 would provide a solution to improve its oxidation behavior. A potential candidate to induce the densification of oxide scale is the application of hafnium oxide (HfO_2), which forms a solid solution with ZrO_2 at high temperatures as a top coat for ZrB_2. Thin HfO_2 ceramic coatings were deposited by means of magnetron sputtering on top of ZrB_2 and a subsequent annealing treatment (vacuum) at 1800°C was carried out to improve the adhesion of HfO_2 with ZrB_2. High-temperature oxidation studies were performed at 1500°C in a tube furnace under constant air flow (0.4 l/min) for 1 and 4h exposure times. After annealing, the HfO_2 coatings were found to be intact. The influence of the annealing as well as the results of the oxidation experiments will be discussed.

S19: Molecular-level Processing and Chemical Engineering of Functional Materials

Polymer-Derived Ceramics V

Room: Ballroom 3 (South Tower)
Session Chair: Sven Barth, Goethe University Frankfurt

8:30 AM

(ICACC-S19-024-2023) Polycarbosilanes: A Versatile Bridge between Polymer Chemistry, Soft Matter Processing, and High-Temperature Ceramics (Invited)

M. B. Dickerson*[1]; K. Martin[1]; J. Bowen[1]

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

Polycarbosilanes are specialized polymers containing silicon and carbon in their backbone and are critically important to the modern aerospace industry as ceramic precursors. The importance of polycarbosilanes arises not only from their role as preceramic polymers but in the processability of these macromolecules. For example, polycarbosilanes can be spun into fibers and heat treated to create high-performance SiC fibers. In addition to fiber spinning, the unique properties of polycarbosilanes lend these polymers to new avenues of research into structure-property relationships in polymer chemistry, hybrid material fabrication, additive manufacturing, and resulting polymer-derived ceramics. In this presentation, we will discuss our recent studies into the synthesis of polycarbosilane-nanoparticle hybrid materials (preceramic polymer grafted nanoparticles(PGNPs)). Specifically, we will discuss the impact of polycarbosilane chemistry on the rheology and curing of these PGNPs as well as the composition of the resulting ceramic materials. Beyond hybrid particles, the production of hierarchically structured ceramics via the additive manufacturing of polycarbosilanes with structure directing additives will be presented.

9:00 AM

(ICACC-S19-025-2023) Quantum-Chemical Simulation of Early Stages in Pyrolysis of SiCON Polymers (Invited)

P. Kroll*[1]

1. University of Texas, Arlington, USA

Processing polymer resins into ceramics provides a tremendous variety of different materials through tailoring of functional groups and chemical composition. Thus, the polymer-to-ceramic route is ideal for manufacturing techniques that require morphology control, high yield, and scalability to produce 3D-structured objects. At the beginning of the thermal conversion of polysilanes, polycarbosilanes, polysilazanes, and polysiloxanes, fundamental reactions occur during cross-linking and in the early stages of pyrolysis. Many of these reactions have been conjectured based on the outcome, while details and mechanisms remained unresolved. Molecular Dynamics simulations based on quantum-chemical approaches, therefore, have the potential to deliver new insight into reaction paths and products. Here we present atomistic details obtained from quantum-chemical (ab-initio) Molecular Dynamic Simulations for cross-linking and pyrolysis reactions of several silicon-based polymers. We observe intra-chain and inter-chain coupling, cross-linking, and elimination reactions. In particular, we analyze Kumada and Kumada-type reactions that yield incorporation of carbon into the polymer backbone.

9:30 AM

(ICACC-S19-026-2023) Machine Learning in Materials Design and Knowledge Discovery (Invited)

O. N. Oliveira*[1]; M. Ferreira de Oliveira[2]

1. University of Sao Paulo, Sao Carlos Physics Institute, Brazil
2. University of Sao Paulo, Brazil

The use of machine learning is becoming ubiquitous in most areas of science and technology, and materials sciences is no exception. In this lecture, we shall offer a broad perspective on recent progress in applying machine learning to the design and discovery of materials, including for reaching optimized properties. This progress has been facilitated by extensive databases of materials and their properties currently available, compiled through high-throughput experiments and theoretical simulations. The discovery of materials has also been linked with knowledge discovery, a relevant subject in computer science for decades, relying mostly on information retrieval, text mining and natural language processing (NLP). Recent advances in deep learning architectures for NLP tasks introduce novel possibilities for knowledge discovery from mining the scientific literature. Examples will be provided of semi-automated surveys of the literature, in which materials properties and applications can be correlated from the analysis of tens of thousands of papers. Another frequent use of machine learning in materials sciences is on data analysis for sensors and biosensors, which supports enhanced clinical diagnosis and environment monitoring. We shall illustrate how sensing data can now be combined with other types of information, e.g. text and image, within a paradigm exploiting the capabilities of knowledge discovery.

Innovative Chemical Approaches & Processing Methods

Room: Ballroom 3 (South Tower)
Session Chair: Thomas Fischer, University of Cologne

10:20 AM

(ICACC-S19-027-2023) Preparation of functional nanosheets via controlled interlayer surface modification and subsequent exfoliation

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2. Waseda University, Kagami Memorial Research Institute for Materials Science and Technolog, Japan
3. Waseda University, Department of Applied Chemistry, Japan

Inorganic nanosheets have attracted enormous attention in recent years, and they are typically prepared from layered materials via exfoliation. We have been utilizing potassium hexaniobate, which has two different interlayers with different reactivities for ion exchange reactions, highly reactive interlayer I and interlayer II with low reactivity, appear alternately in its stacking direction. Since expansion of interlayer space by ion-exchange reactions with organoammonium ions is required for grafting reactions in interlayers, regioselective surface modification can be achieved for preparing functional nanosheets. For the first functionalization, polymer chains containing acid-degradable crosslinkers were grown at interlayer I. Then the resulting product was exfoliated at interlayer II to form double-layered nanosheets. Upon dispersion of the double-layered nanosheets in an acidic solution (pH = 4), the thickness of the resulting nanosheets decreased, indicating the conversion of double-layered nanosheets into single-layered nanosheets. For the second functionalization, after surface modification at interlayer I, grafting reaction at inter layer II was achieved by using another type of surface modifying reagent. Exfoliation at both interlayer I and interlayer II led to the formation of Janus nanosheets, which can stabilize an o/w emulsion.

10:40 AM

(ICACC-S19-028-2023) Charged Particle-Based Direct-Write Techniques for the Growth of Inorganic Nanostructures (Invited)

S. Barth*[1]; F. Jungwirth[1]; F. Porrati[1]; M. Huth[1]

1. Goethe University Frankfurt, Germany

Focused electron beam deposition (FEBID) allows the spatially controlled formation of nanoparticles, thin film patches, nanowires and free-standing complex nanoarchitectures on a wide range of substrate materials. Moreover, the method enables a growth on structured surfaces and the formation of predefined arrays by this maskless direct-write approach. However, the purity of the as-grown material is typically limited. Therefore, the method requires precursor development to provide chemical impetus driving the methods applicability. This contribution will shortly summarize the state-of the art of precursors used in this approach, which has been described in a recent review. Specific examples of precursors will be discussed that enable for the direct deposition of binary deposits. The knowledge gained is applied to the synthesis procedures for metal silicide and alloy formation as well as functional metal carbides. Moreover, specific examples of superconducting nanostructures derived by focused ion beam deposition (FIBID) will be presented. In this context, the differences and benefits of FEBID and FIBID will be highlighted and changes in physical properties depending on the deposition technique will be highlighted. Acknowledgment: S.B. acknowledges generous financial support from the Deutsche Forschungsgemeinschaft (DFG, German Research Foundation) projects 413940754 and 413942347.

11:10 AM

(ICACC-S19-029-2023) Chemically Engineered Nanocarriers for Tumor Specific Localization and Drug delivery

S. Ilyas*[1]; A. Szymura[1]; A. Renner[1]; E. Sahnoun[2]; P. Habib[3]; F. Mottaghy[2]; S. Mathur[1]

1. University of Cologne, Institute of Inorganic Chemistry, Germany
2. University Hospital Aachen, Department of Nuclear Medicine, Germany
3. University Hospital Aachen, Department of Neurology, Germany

Low solubility of the molecular drugs fetches poor efficacy and systemic toxicity. Transporting radionuclides and/or encapsulating the drug molecules via nanocarriers promise long blood circulation time. Fusion of molecules on the surface of mesoporous carriers facilitate overcoming intrinsic physiological barriers of clearance through innate immune system and ensure safe delivery of therapeutics to tumor. We developed several nanocarriers for the encapsulation of molecular drugs and radionuclides beside complementary functionalities through covalent functionalization strategies. Stimuli responsive polymers on carrier particles assisted drug storage and controlled release. Surface ligands were quantified using electrokinetic sonic amplitude based approach. Data confirmed the preferential accumulation of carriers into tumor without effecting the healthy organs. The approach was extended to chemically engineered nanoassembly (CENs) by co-conjugating anticancer drugs and radionuclides which showed enhanced therapeutic effect to tumor cells. The minimum off-target and effective ligand-driven transport of therapeutics to tumor illustrate the potential of our site-specific approach.

11:30 AM

(ICACC-S19-030-2023) Silicon carbide and tantalum carbide nanocomposite coatings of by laser chemical vapor deposition

H. Katsui*[1]; K. Shimoda[2]; M. Hotta[1]

1. National Institute of Advanced Industrial Science and Technology (AIST), Multi-Material Research Institute, Japan
2. National Institute for Materials Science (NIMS), Research Center for Structural Materials, Japan

Ceramic coatings with high erosion and wear resistance are promising for prolonging lifetime of mechanical seals and sliding components used in harsh water environments. To enhance the properties,

durability, and compatibility of coating layers, tuning mechanical properties of the coating layers are of importance; generally, the ceramic layers with high hardness and low young's modulus are effective for the improvement of wear resistance and compatibility with substrates (components) of dissimilar materials. Chemical vapor deposition (CVD) techniques utilizing laser apparatus enable us to deposit layers with good controllability of compositions with nanocomposite structures at high deposition rates. In this study, the deposition of composite layers comprising SiC and TaC on silicon nitride substrates was demonstrated by laser CVD using hydridocarbosilane and alkylamido compounds as precursors. Effects of deposition parameters on phase compositions, microstructure, chemical compositions, and mechanical properties are examined. SiC–TaC composite layers, in which each phase is finely mixed in the nano scale, with various compositions were formed; the values of Vickers hardness and young's modulus evaluated by nanoindentation tests in the composite layers with 72 mol.% SiC were 24 GPa and 240 GPa, respectively.

11:50 AM

(ICACC-S19-031-2023) Compositional and Structural Disorder as Enablers for the Circular Economy

E. Ionescu*[2]; T. Dharma Teja[1]; R. Yan[1]; X. Xiao[1]; M. Widenmeyer[1];
S. Klemenz[2]; J. Gassmann[2]; G. Homm[2]; A. Weidenkaff[1]

1. Technical University Darmstadt, Materials Science, Germany
2. Fraunhofer IWKS, Germany

Circular Economy (CE) represents a concept concerning the production and consumption of goods which basically should reduce the amount of waste to a minimum. There is an obvious contradiction between the need for a CE and the tremendously increasing amount and complexity of consumer goods, their planned obsolescence, and the massive amount of waste due to the take-make-waste peculiarity of their production/consumption. CE has a powerful enemy, i.e. the second law of thermodynamics, which actually prevents that closing the production loops leads to full resource efficiency. Thus, minimizing residues and losses should be considered as important as and in addition to closing the material loops. The consequence is the stringent need to minimize the entropy increase across the various added value chains. However, we will show in the present talk that entropy increase may not always be seen as acting against CE. Firstly, we will discuss the concept of structural and compositional disorder. Secondly, we will elaborate on the compositional complexity in materials as an effective way to consider highly heterogeneous waste streams as valuable secondary raw materials. And thirdly, we will perform a prospective discussion on preparative concepts and strategies to develop materials with maximized performance and high tolerancy for admixtures & impurities.

Emerging Materials and Sustainable Manufacturing Technologies in a Global Landscape: Symposium in Honor of Dr. Tatsuki Ohji

Tatsuki Ohji Honorary Symposium IV

Room: Coquina Salon D (North Tower)
Session Chairs: Dileep Singh, Argonne National Lab; Paolo Colombo, University of Padova

1:30 PM

(ICACC-HS-021-2023) Advanced ceramics for green hydrogen technology (Invited)

A. Michaelis*[1]

1. Fraunhofer IKTS, Germany

Ceramic materials and components play a key role in the production, transport, and application of green hydrogen. For large scale green hydrogen production electrolysis has to be used. We present new results for ceramic based steam electrolysis systems: SOE (solid oxide electrolysis). SOE offers two essential unique advantages: 30 % higher efficiency for power to green hydrogen conversion compared to competing technologies (e.g. alkaline and PEM electrolysis) with the use of waste heat. Co-electrolysis capability, i.e. both water steam and CO_2 can be simultaneously reduced to form syngas (H_2 + CO). With this, CO_2 can be actively removed from the environment. The produced syngas can be used for PtX products like e-fuel, higher alcohols, waxes, etc. For this, we present a fully integrated co-electrolysis Fischer Tropsch System. Moreover, the combination of SOE with the Haber Bosch process allows for green ammonia NH_3 production. As a hydrogen carrier Ammonia can contribute to the transport issue in a future hydrogen economy. We also present new ceramic gas separation membranes which allow to separate hydrogen from natural gas (NG). This enables to transport hydrogen in conventional NG pipelines.

2:00 PM

(ICACC-HS-022-2023) Advanced Strategies of Reclaiming and Reusing of Discarded Graphite from Spent Lithium-Ion Batteries (Invited)

R. Riedel*[1]

1. TU Darmstadt, Materials Science, Germany

The lithium-ion batteries (LiBs) recycling market is fueled by the tight availability supply of raw materials utilized in LiBs manufacturing, geographical resource restrictions, and strong demand in the end-use market. Graphite negative electrodes are unbeaten hitherto in LiBs due to their unique chemical and physical properties. The proven global reserves of natural graphite were around 71 million tons in 2014, but the demand is steadily growing at a rate of 250.000 tons per year, with approximately 72 % of graphite going into Li-ion batteries between 2016 and 2025. Herein we present a facile and efficient process of an effective and up-scalable reclamation of graphite from end-of-life LiBs. In order to broaden the application field of the recycled graphite, its functionality as stabilizing matrix for silicon nanoparticles is also studied. Silicon-based anodes are notoriously suffering from the drawback of pulverization due to the huge volume expansion (ca. 300%) with lithium alloying reactions. The successful mitigation of the severe capacity degradation caused by Si pulverization by tuning the Si:regenerated graphite ratio and the thickness of the carbon layer is reported and discussed.

2:30 PM

(ICACC-HS-023-2023) Band-gap Engineering of Perovskites for Photovoltaics (Invited)

M. Braun[1]; M. J. Hoffmann*[1]

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

Methylammonium lead halide perovskites are one of the emerging photovoltaic materials with very high efficiencies in thin film solar cells. While prospective cheap and easy processing could be advantageous, fundamental problems like their use of toxic materials and degradation issues motivate to seek for new efficient, non-toxic and stable absorbers solar cells. Solar cells based on perovskite oxide absorbers achieved recently up to 8 % power conversion efficiency and promise at the same time intrinsic long-term stability. Current studies deal with very few suitable transition metal oxides even though the field offers a wide range of material combinations, but it is challenging to find narrow band-gap semiconductors among metal oxides. The present study investigates the influence of substituting the B-site cation on the properties of ABO_3-perovskites. We will show that fundamental requirements for absorbers such as band gap in the visible light, higher absorption coefficient and sufficient charge carrier mobility can be achieved. It is demonstrated that the band-gap can be modified systematically by introducing Cu^{2+} as well as penta- or hexavalent transition metals on the B-site. Semiconducting properties due to these substitutions are analysed by preparing materials via solid state reactions to process thin films grown with several hundred nanometres thickness onto transparent conductive oxides by pulsed laser deposition.

3:20 PM

(ICACC-HS-024-2023) Size Effects on Transport, Thermodynamics and Energy Storage (Invited)

P. Balaya*[1]

1. National University of Singapore, Department of Mechanical Engineering, College of Design and Engineering, Singapore

Nanostructured materials have triggered a great excitement in the area of energy applications due to both fundamental interest as well as technological impact. Nanosize reduction leads to a variety of unexpected exciting phenomena due to enhanced surface-to-volume ratio and reduced diffusion length for ions/electrons. We will consider some of those anomalous phenomena restricting our discussions to the nano-size effects on (a) transport, (b) thermodynamics and (c) storage behavior with a few examples to illustrate material challenges for developing advanced energy storage devices. (a) Mesoscopic electrical conduction occurs due to overlap of space charges at reduced interfacial spacings. Unlike microcrystalline $SrTiO_3$, having both bulk as well as semi-infinite interfacial contributions to the electrical conduction, nanocrystalline $SrTiO_3$ exhibits only interfacial conduction. (b) Size reduction of materials affects thermodynamic properties and hence their energetics due to excess surface contributions causing stabilization of meta-stable phases at nano-size. Increase in cell voltage due to nanosizing or amorphization will be highlighted. (c) In the context of storage behaviour, nanocrystalline materials exhibit high capacity as well as high coulombic efficiency (reversible storage). We will consider a few case studies on lithium and sodium storage.

3:50 PM

(ICACC-HS-025-2023) Studies on Double Perovskite $Pr_{1-x}Ba_{1+x}Co_2O_{6-\delta}$ and Ruddlesden popper $Sm_{2-x}Sr_xNiO_{4-\delta}$ systems for energy storage applications (Invited)

A. S. Bangwal[1]; M. Chauhan[1]; P. Singh*[1]

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

Double Perovskite, $Pr_{1-x}Ba_{1+x}Co_2O_{6-\delta}$ and Ruddlesden popper $Sm_{1-x}La_xSr_{1-x}Ca_xNiO_{4-\delta}$ are investigated for energy storage application. The phase formation and structure were determined by Powder X-ray diffraction. The catalytic performance of the prepared catalysts in alkaline solution was investigated using electrochemical measurements for both oxygen evolution reaction (OER) and oxygen reduction reactions (ORR). The series of double perovskite oxide materials is found to exhibit catalytic activity for both OER and ORR. $Pr_{0.90}Ba_{1.10}Co_2O_{6-\delta}$ showed enhanced OER activity among all the double perovskite catalysts with a specific capacitance of 598.40 F/g and double-layer capacitance of 38.94 mF/cm^2. Power law indicated oxide-ion intercalation pseudo capacitance in the $Pr_{0.90}Ba_{1.10}Co_2O_{6-\delta}$. The system $Pr_{0.95}Ba_{1.10}Co_2O_{6-\delta}$ exhibited potential behavior for ORR. The Cyclic voltammetry curve of the Ruddlesden popper $Sm_{0.90}La_{0.10}Sr_{0.90}Ca_{0.10}NiO_{4-\delta}$ and $Sm_{0.80}La_{0.20}Sr_{0.80}Ca_{0.20}NiO_{4-\delta}$ depicted combination of both pseudo capacitance and electrochemical double layer capacitance. $Sm_{0.90}La_{0.10}Sr_{0.90}Ca_{0.10}NiO_{4-\delta}$ is found to satisfy the condition of general power law for pseudo capacitance. This material also has a highest value of specific capacitance (910.20 F/g) and electrochemical double layer capacitance (238.25 mF/cm^2).

4:20 PM

(ICACC-HS-026-2023) Energy recovery from fluctuating heat sources using thermoelectric cycling (Invited)

T. Nakayama*[1]; B. Amila[1]; N. Trung[1]; H. Suematsu[2]; T. Goto[1]; K. Niihara[1]

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

The purpose of this research is to investigate the power generation and figure of merit by fabricating and controlling the composition of ferroelectric ceramics that contributes to the energy conversion of fluctuating heat sources. We focused on (1-x)$Pb(Mg_{1/3}Nb_{2/3})O_{3-x}PbTiO_3$ (PMN-PT) as a candidate material from preliminary experiments. PMN-PT with controlled composition was fabricated by pressureless sintering method. As a result, it was confirmed that the sintered body had a perovskite structure. In the power generation evaluation and performance index evaluation, the highest power generation was observed in PMN-27PT, which is the MPB phase. From composition control, soft materials have a good effect on power generation due to their low coercive electric field, while hard materials have an adverse effect on power generation due to electric field application loss due to high coercive electric fields and internal resistance decrease near the Curie temperature. confirmed things. Furthermore, the power generation and the peak temperature of the figure of merit coincided, and the relationship between each was shown. This knowledge is expected to be applied to exhaust heat regeneration from fluctuating heat sources.

FS4: Ceramic/Carbon Reinforced Polymers

Mechanical Behavior of Composite Materials

Room: Flagler A (South Tower)
Session Chair: Asami Nakai, Gifu University

1:30 PM

(ICACC-FS4-001-2023) Experimental observation and modeling of resin pocket cracking in CFRP laminates with ply discontinuity (Invited)

M. Fikry[1]; V. Vinogradov[2]; S. Ogihara*[1]

1. Tokyo University of Science, Department of Mechanical Engineering, Japan
2. Newcasle University, School of Engineering, United Kingdom

Experimental results revealed that several cracks may occur in a resin pocket before delamination develops in a unidirectional CFRP laminate with discontinuous plies. Instead of occurring at the interfaces with the discontinuous plies, the observed cracks form inside the resin pocket and curve towards the center of the pocket. A simple variational stress analysis based on the principle of minimum complementary energy is then developed to study this counterintuitive cracking behavior. It allowed us to estimate the stress field in a laminate with discontinuous plies, with and without cracks in the resin region. The observed cracking pattern is explained by assuming that there are increased residual stresses present as a result of the resin's chemical shrinkage during curing in the pocket's constricted environment. It is demonstrated that the maximum energy release rate principle works best for determining crack locations, while the curved crack path can be governed by the distribution of the maximum principle stress. The predicted cracking sequence and crack curvatures accurately reflect experimental observations.

2:00 PM

(ICACC-FS4-002-2023) High-resolution in situ characterization of microscopic failure mechanisms in CFRP laminates under mode II loading (Invited)

S. Oshima*[1]; M. Hojo[2]

1. Tokyo Metropolitan University, Department of Aeronautics and Astronautics, Japan
2. Kyoto University, Department of Mechanical Engineering and Science, Japan

Microscopic failure mechanisms in carbon fiber reinforced polymer (CFRP) laminates under mode II loading were characterized in situ by advanced inspection techniques. The observations by high-resolution optical microscopy sequentially captured the formation of the damage zone, which is composed of the plastic zone, fiber/matrix debonding, and microcracks. In addition to the optical microscopy observation, the failure process inside the CFRP laminates was studied using synchrotron radiation X-ray computed tomography (SR X-CT). The SR X-CT observation revealed that the interfacial debonding between the carbon fiber/epoxy matrix interface occurred first followed by the formation of microcracks (hackles) in the matrix resin. The microscopic failure process was correlated with the macroscopic behavior of CFRP laminates during mode II fracture toughness tests.

2:30 PM

(ICACC-FS4-003-2023) Progressive damage observation and numerical simulation for thin-ply CFRP laminates with a hole under compressive loading

Y. Fujisawa*[1]; K. Ito[1]; T. Mikami[1]; T. Ogasawara[2]; K. Aoki[3]; S. Uchiyama[3]; S. Sugimoto[5]; T. Yokozeki[4]

1. Tokyo University of Agriculture and Technology, Institute of Engineering, Japan
2. Tokyo University of Agriculture and Technology, Japan
3. SUBARU Corporation, Japan
4. The University of Tokyo, Japan
5. Japan Aerospace Exploration Agency, Japan

Carbon fibre reinforced plastic (CFRP) laminates are attractive structural materials in aerospace structures because of high specific stiffness and strength. In manufacturing aerospace structures, it is important to understand the damage mechanisms of CFRP laminates with an open hole under compression. Therefore, this study focused on open hole compression (OHC) strength of thin-ply CFRP laminates. For experimental approach, X-ray computed tomography (CT) of the failure behaviour around a hole was conducted during the OHC tests. The decrease of ply thickness affected the increase of OHC strength and the suppression of damage propagation such as delamination. For numerical approach, numerical analyses incorporating damage propagation model were carried out to understand the damage propagation around a hole. It was demonstrated that the numerical analyses can predict OHC strength depending on ply thickness.

2:50 PM

(ICACC-FS4-004-2023) Numerical Study on the Effect of Process-induced Fiber Waviness on Mechanical Properties of Composite Laminates

T. Nishioka*[1]; R. Higuchi[1]; T. Yokozeki[1]

1. University of Tokyo, Japan

Recently, Carbon Fiber Reinforced Thermoplastics (CFRTPs) have been applied to structural components of aircraft owing to their short manufacturing time and recyclability. One of the serious problems in CFRTPs is the process-induced fiber waviness that happens in the cooling process from the higher manufacturing temperatures than that of thermoset composites. This waviness deteriorates the damage tolerance of the structure, particularly in longitudinal compressive loading. Although many studies were conducted to examine the effect of waviness on the mechanical properties of composites, to the best of the authors' knowledge, most of them focused only on unidirectional ply. The purpose of this study is to numerically investigate the effects of fiber waviness on mechanical properties in composite laminate structures. In unidirectional composites with waviness, transverse cracks can propagate along fiber waviness. In order to model such curved cracks depending on the waviness geometry, eXtended Finite Element Method (XFEM), which can model the cracks separately from elements, is introduced in this study. The proposed model is validated via the comparison of numerical and experimental results in the unidirectional ply. Then, the effect of waviness on the laminate strength is examined by the proposed scheme.

Composites for SDGs

Room: Flagler A (South Tower)
Session Chair: Satoshi Kobayashi, Tokyo Metropolitan University

3:30 PM

(ICACC-FS4-005-2023) Circular Economy of Carbon-Fiber Reinforced Polymer Composites (Invited)

E. Ionescu*[1]; W. Benner[1]; M. Vogelgesang[1]; C. Li[1]; A. Gassmann[1]; G. Homm[1]; C. Deubel[2]

1. Fraunhofer IWKS, Germany
2. SKZ - das Kunststoff-Zentrum, Germany

The highly dynamic growth of the global market for Carbon-Fiber-Reinforced Polymers (CFRPs) and the lack of appropriate concepts for a circular economy of end-of-life CFRPs have been currently in the focus of the academic community as well as industry. In the present talk, common efforts performed at Fraunhofer IWKS and SKZ related to the development of appropriate recycling strategies for end-of-life CFRPs in order to recover the carbon fibers and consider them for the preparation of novel carbon-fiber reinforced polymer parts will be introduced and critically discussed. Additionally, research performed at Fraunhofer IWKS which addresses strategies towards circular economy of polymers will be highlighted. Within this context, a holistic, entropy-based assessement model which evaluates best recycling paths (mechanical recycling, solvolysis, pyrolysis and gasification etc.) and combinations thereof for specific highly heterogeneous waste plastic flows will be presented.

4:00 PM

(ICACC-FS4-006-2023) Effect of cooling conditions during c-FRTP molding on the crystallinity and mechanical properties

A. Nakai*[1]

1. Gifu University, Japan

In the purpose of obtaining knowledge about the cooling conditions of the material when the pultrusion speed is improved in the pultrusion molding using c-FRTP (continuous fiber reinforced thermoplastic), the effects of the cooling speed on the crystallinity of the resin and the mechanical properties of the molded product are investigated. As a result, it became clear that the crystallinity can be controlled by controlling the cooling temperature. Subsequently, the effects of crystallinity on the fiber / resin interface characteristics, onset and progress of micro fractures, and the mechanical properties of the molded product were clarified. The elastic modulus increased as the cooling rate was slower (higher crystallinity), and the bending strength increased as the cooling rate was slower (lower crystallinity).

4:20 PM

(ICACC-FS4-007-2023) Design of novel Cellulose Acetate blends by adding lignin and cellulose as reinforcement

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

Humans utilize vast quantities of plastic each year, manufactured from several different industrial sectors. Most of these plastics are petroleum-based polymers and are not biodegradable, which causes significant environmental problems. In addition, there is the need for alternate and sustainable resources for the future due to the diminishing supply and high cost of fossil fuels. As a result, a lot of research is being done to create biodegradable plastics or bioplastics from sustainable resources for a variety of applications. Cellulose Acetate (CA) is an example biopolymer with important attributes like biodegradability, renewable resource sourcing, and non-toxicity. However, bioplastics have only found limited applications because of their weak mechanical strength. Hence numerous research is being done in the development and application of reinforcement materials in bioplastics to develop bioplastic composites with improved mechanical properties. This study will focus on synthesis and characterization of cellulose (in bulk and nano form) and lignin (in solution and particle form) as reinforcement materials in the Cellulose Acetate matrix.

S1: Mechanical Behavior and Performance of Ceramics & Composites

Processing-Microstructure-Mechanical Properties Correlation I

Room: Ballroom 5 (South Tower)
Session Chairs: Marina Ruggles-Wrenn, Air Force Institute of Technology; Matthew Appleby, NASA Glenn Research Center

1:30 PM

(ICACC-S1-035-2023) Microstructure and high-temperature mechanical properties of WC-Ni cemented carbides sintered from novel precursor powders

P. H. Gruber*[1]; S. Naim Katea[3]; G. Westin[2]; F. Akhtar[1]

1. Luleå University of Technology, Materials Science - Engineering materials, Sweden
2. Uppsala University, Sweden
3. Höganäs AB, Sweden

Increasing the sintering activity of WC-Ni cemented carbide precursor powders can provide greater microstructural control during consolidation and has the potential to reduce the amount of required binder phase. By applying a novel solution-based powder coating technique, WC powder was coated with 2, 6, and 14 vol. % metallic Ni in the form of nanometer-sized dots. SEM micrographs showed that the Ni dots were evenly covering a relatively large amount of the carbide's surface. The Ni-coated WC powders were then consolidated using spark plasma sintering (SPS) at 1350°C and 50MPa. The sintered specimen exhibited high relative densities over 97%, even binder phase distributions, even for 2 vol. % Ni, and sub-micrometer-sized WC grains. This indicates a high sinter activity and little to no grain growth. Hardness and toughness at temperatures from 20°C to 700°C were studied utilizing 10kg Vickers indentations. The results indicate suitability for tooling applications and are on par with conventional WC-Co cemented carbides and more elaborate or costly WC-Ni processing routes. SEM, EDS, XRD, and EBSD were used to characterize the microstructure before and after high-temperature testing. The numerous interfaces between Ni and WC as well as the small size of the Ni-dots yielded a high sinter activity while ensuring control over grain growth during SPS.

1:50 PM

(ICACC-S1-036-2023) Pillar Compression Study of Binderless Tungsten Carbide Consolidated by Flash Sintering and Spark Plasma Sintering

I. Mazo*[1]; A. Molinari[1]; J. Molina-Aldareguia[2]; V. M. Sglavo[1]

1. University of Trento, Industrial Engineering, Italy
2. IMdea Materials, Spain

Binderless tungsten carbide (BTC) is a material with outstanding mechanical properties, although its widespread use is limited by the lack of suitable processing route which guarantees the right combination of hardness and toughness. This study aims at unravelling the effect of fast and ultrafast field-assisted sintering processes, namely spark plasma sintering (SPS) and flash sintering (FS), on the room-temperature plasticity of polycrystalline BTC materials. Micropillars, 3 μm in diameter, were prepared by focused ion beam (FIB) to study the small-scale deformation. An electron-transparent lamella was milled out of the pillars plastically deformed at different strain levels to carry out Transmission Kikuchi diffraction (TKD) and HRTEM analyses. Pillars prepared from the flash sintered material characterized by biphasic microstructure (WC/W_2C) reach an outstanding plastic strain of ε=8%. Plasticity in polycrystalline BTC pillars occurs at a similar stress level of grain boundary decohesion (intergranular crack propagation). Thus, the introduction of highly defective W_2C surrounding WC grains by flash sintering promotes an intrinsic toughening mechanism which grants stable crack propagation up to higher strain levels before pillar failure.

2:10 PM

(ICACC-S1-037-2023) Effect of the Manufacturing Conditions on the Local Mechanical Behavior of Cast Tungsten Carbide

M. Ciurans Oset*[1]; D. Jarzabek[2]; J. Mouzon[1]; F. Akhtar[1]

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2. Institute of Fundamental Technological Research, Polish Academy of Sciences, Department of Mechanics of Materials (ZMM), Poland

Cast tungsten carbide (CTC) results from the eutectoid decomposition of a melt containing W-3.9 wt.% C and consists of a biphasic structure of WC lamellae in a W_2C matrix. In this study, the local mechanical behavior of multiple spherical CTC and crushed CTC powders was thoroughly investigated. The carbon content of all powders was in the range of 3.8-4.1 wt.%. Vickers microindentation testing of individual particles was performed under 0.25 N. Spherical CTC exhibited significantly higher hardness than crushed CTC (28.3 ± 1.8 GPa and 24.2 ± 1.6 GPa, respectively). Furthermore, among all spherical powders investigated, plasma-atomized CTC exhibited the highest hardness (31.4 ± 1.6 GPa), together with the highest crack formation sensitivity. Compressive strength and fracture toughness of individual particles were evaluated from micropillar compression tests. Compressive strengths of 13.8 ± 1.8 GPa and 10.9 ± 2.1 GPa were obtained for as-produced plasma-atomized CTC and crushed CTC, respectively. Both materials evolved from purely elastic to elastoplastic behavior upon heat treatment, with respective compressive strengths decreasing to 10.7 ± 1.8 GPa and 7.1 ± 1.8 GPa. XRD was used to evaluate the fineness of the microstructure and the residual stresses of each powder, to ultimately define the interplay between manufacturing conditions, microstructure, and mechanical properties.

2:30 PM

(ICACC-S1-038-2023) On the development of multifunctional ceramic composites – Fracture resistance

C. Muñoz Ferreiro*[1]; H. Reveron[2]; J. Chevalier[2]; A. Rodríguez Morales[1]; R. Poyato[3]; Á. Gallardo López[1]

1. Universidad de Sevilla, Física de la Materia Condensada, Spain
2. Univ Lyon, MATEIS UMR5510, Insa de Lyon, Ceramics and Composites Group, France
3. Instituto de Ciencia de Materiales de Sevilla (ICMS), Spain

The development of new materials is a key factor in the progress of the environment, medicine, space, or energy industries. To this end, composite materials are being developed to meet the multifunctional properties required in these fields. In particular, ceramics are of great importance because of their chemical inertness and resistance to corrosion and high temperatures, which make them suitable as matrices for the development of new composites. However, we still need to better understand the failure mechanisms of ceramic matrix composites because for long-term-use applications, their mechanical performance cannot be compromised. In this work, we developed 3 mol% yttria tetragonal stabilized zirconia (3Y-TZP) reinforced with two-dimensional nanomaterials such as graphene. Whereas the secondary phase would provide new functionalities to the composite (e.g. thermal or electrical conductivity), its introduction should not be in detriment of the mechanical behavior. In this communication, we will deeply analyze the correlation between the processing method, the secondary phase features (amount, size, crystallinity and distribution) and the behavior of 3Y-TZP ceramics containing 2D-nanomaterials (< 5 vol.%). In particular, the crack resistance propagation will be discussed based on R-curve analyzes coupled to SEM microstructural characterization.

3:10 PM

(ICACC-S1-039-2023) Fabrication of Al_2O_3-Y_2O_3-ZrO_2 composites using Spark Plasma Sintering

M. Vakhshouri*[1]; A. Najafzadehkhoee[2]; A. Talimian[1]; Á. Gallardo-López[3]; R. Poyato Galan[3]; F. Gutiérrez-Mora[3]; A. Prnova[2]; D. Galusek[1]

1. FunGlass, Alexander Dubček University of Trenčín, Slovakia
2. Joint Glass Centre of the IIC SAS, TNUAD, and FChPT STU, FunGlass, Slovakia
3. Universidad de Sevilla, Dpto. de Física de la Materia Condensada, Spain

Owing to their excellent mechanical properties, Al_2O_3/Y_2O_3 ceramics with eutectic microstructure are a promising candidate for structural application from room temperature to high temperature. Although adding zirconia particles can improve the fracture toughness of ceramics, the mechanism for the increase in fracture toughness of eutectic $Al_2O_3/Y_2O_3/ZrO_2$ ceramics is equivocal. In this study, Pechini's method was utilised to synthesise $Al_2O_3/Y_2O_3/ZrO_2$ composite powders with various amounts of ZrO_2 up to 10 mol%. Samples were densified using Spark Plasma Sintering. The phase composition and the homogeneity of phases were analysed using X-ray Diffraction and Electron Microprobe Analysis. The sintered samples were characterised in terms of densification shrinkage and sinterability, microstructural features, Vickers' hardness, and fracture toughness. μ-Raman analyses were employed to determine the phase transformation of zirconia at the vicinity of cracks generated by Vickers' indentation. The contribution of zirconia phase transformation to the increase in fracture toughness of Al_2O_3/Y_2O_3 was critically discussed.

3:30 PM

(ICACC-S1-040-2023) Tailoring Structural Properties of Reaction Bonded Diamond – Silicon Carbide Composites via Microstructural Parameters

S. McAnany*[1]; S. Salamone[1]; G. Evans[1]

1. Coherent, USA

Reaction bonded diamond – silicon carbide composites are a novel material that is used for high wear resistance or high mechanical and thermal stability. These composites are formed by infiltrating a diamond and silicon carbide green body with silicon, which reacts with the diamond to create more silicon carbide. The resulting part is a non-porous structure of diamond and silicon carbide particles with some residual silicon. In this system there are several factors that can be modified, such as the ratio of diamond to silicon carbide, particles sizes of each phase, diamond grade (or quality) and the volume fraction of residual silicon. By controlling these parameters, the final structure can be tailored to have a range of properties. In this work, the effect of microstructure (diamond:SiC ratio and diamond grade) on the mechanical and thermal properties of diamond - silicon carbide composites is investigated.

3:50 PM

(ICACC-S1-041-2023) Silicon Nitride Collimator Bench for Copernicus CO2M Spectrometer

N. Louh*[1]

1. Thales Alenia Space, France

This material has very interesting properties for space applications, especially for optical instruments and telescopes since its low CTE combined with its high stiffness are well suited to provide high thermo-elastic stability and sustain launch loads. Thales Alenia Space is the prime contractor for developing a spectrometer in the frame of the Copernicus project CO2M for the European Space Agency. The Copernicus Carbon Dioxide Monitoring mission will be the first to measure how much carbon dioxide coming from human activity is released into the atmosphere. Thales Alenia Space has selected Si_3N_4 as material for the design of a complex innovative structure to ensure that mission objectives are reached. Indeed, for CO2M Spectrometer needs in terms of stability, Si_3N_4 was the perfect fit. This part is the biggest and most complex part ever designed by Thales Alenia Space and the largest part for space application manufactured by FCT Ingenieurkeramik. The goal is to obtain a very stiff and stable structure allowing to reach high precision optical performances for the optical instruments to be mounted on it. The first bench manufactured is used to confirm the process for this large piece and to verify the optics alignment and architecture before starting to operate on the flight models design. This article describes how such a part was designed, manufactured and tested for a high precision performance.

4:10 PM

(ICACC-S1-059-2023) Carbon-fiber-reinforced ceramic-matrix composites (Cf-CMCs) for high temperatures: processing flexibility and tailored microstructures

M. Valle*[1]

1. Petroceramics S.p.A., Italy

Carbon-Fiber-reinforced Ceramic-Matrix Composites (Cf-CMCs) combine lightness, stability under mechanical stresses as well as resistance to high temperatures and oxidation. However, the reduction of processing costs and manufacturing of complex geometries are among the partially unsolved challenges which limit the selection of Cf-CMCs for certain applications. LSI-densified Cf-CMCs match good performance in use, cost-effectiveness and remarkable processing flexibility (i.e. suitability for different typologies of Cf-performs regardless of internal structure, shape and dimension). Furthermore, an accurate selection of materials and processing parameters allows an exceptional control on the composite microstructures, and therefore on its final properties. Recently developed

Cf-CMCs named ISiComp® and Oxycomp® have shown exceptional resistance to mechanical stress even under extreme thermo-oxidative conditions and therefore find now application in the automotive and aerospace sectors. The tailored microstructures of ISiComp® and Oxycomp® lie behind their distinct and peculiar properties in terms of thermo-mechanical behavior in application.

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

Environmental Barrier Coatings I

Room: Flagler C (South Tower)
Session Chairs: Jie Zhang, Institute of Metal Research, Chinese Academy of Sciences; Julin Wan, GE Research

1:30 PM

(ICACC-S2-038-2023) Construction of Dual-Phase Ytterbium Disilicate Coatings with Tailored Performance (Invited)

J. Zhang*[1]; H. Wang[1]; J. Wang[1]

1. Institute of Metal Research, CAS, Advanced Ceramics and Composites, China

Environmental barrier coatings (EBCs) are needed to protect SiC_f/SiC ceramics from high temperature water vapor corrosion and molten deposits degradation in modern gas turbines. Ytterbium disilicate, in recent studies, has been considered as a promising EBC candidate. In the current report, pure phase $Yb_2Si_2O_7$ agglomerated feedstocks with varied particle size distribution was employed for atmospheric plasma spraying (APS). Partially volatilized SiO_2 manifested the formation of fine dual-phase heterostructure as well as coarse $Yb_2Si_2O_7$ and Yb_2SiO_5 grains. The crystallographic orientation, microstructural stability and formation mechanism of the dual-phase were investigated. Moreover, the CMAS attack response drastically improved the overall coating resistance to CMAS infiltration by enhancing the apatite-forming capability, as well as the improved crack propagation resistance of thermal cycling performance of $Yb_2Si_2O_7/Si$ coatings were revealed. The current work aimed at understanding the microstructure-property relationships of ytterbium disilicate coatings, thereby providing useful inspirations on the design of advanced EBCs.

2:00 PM

(ICACC-S2-039-2023) Environmental Barrier Coatings with Oxide-Based Bond Coat for Ceramic Matrix Composites

K. Lee*[1]; R. I. Webster[1]; B. J. Puleo[1]; B. J. Harder[1]; M. J. Presby[1]; W. D. Jennings[1]

1. NASA Glenn Research Center, USA

Environmental barrier coatings (EBCs) have enabled the implementation of SiC/SiC ceramic matrix composites (CMCs) in gas turbines by protecting CMCs from corrosive species. The first EBC-coated CMC component entered service in a commercial gas turbine in 2016. Many EBC challenges remain for continued success of CMC components in the next generation gas turbines. There is a continuous push to develop next generation CMCs with up to 1480°C temperature capability. The upper use temperature of current EBCs is limited by the low melting point of the silicon bond coat (1414°C). The viability of 1480°C CMCs, therefore, is contingent upon the successful development of next generation EBCs with up to1480°C bond coat temperature capability. No metals or metal alloys are viable candidates for next generation EBC bond coat because they do not possess oxidation resistance at such high temperatures. Oxides are logical candidates because of their chemical stability in oxidizing environments and high melting points. This paper will discuss the current status of EBCs with oxide-based bond coat at NASA.

2:20 PM

(ICACC-S2-040-2023) Accelerating the design of multicomponent rare earth silicates for SiC_f/SiC CMC by combinatorial material chip design and high throughput screening

X. Lv*[1]; Y. Lei[1]; J. Zhang[1]; J. Wang[2]

1. institute of metal research, Chinese Academy of Sciences, Advanced Ceramics and Composites Division, China
2. Shenyang National Laboratory for Materials Science, Institute of Metal Research, Chinese Academy of Sciences, High-performance Ceramics Division, China

Rare earth (RE) silicates have attracted considerable attention as advanced environmental barrier coating and interphase materials for SiC_f/SiC CMC. Recent researches have proved that RE silicates with multiple RE elements exhibit significant merits than single ones, and efficient methods are required to accelerate the compositional optimization. In this work, high throughput experimentation is employed to establish a ternary system with the compositional range of 30.8 - 75.7 mol.% SiO_2, 16.6 - 61.7 mol.% Yb_2O_3, and 6.3 - 14.1 mol.% Ho_2O_3 through co-sputtering deposition on one combinatorial material chip. Phase composition and mechanical property of samples with various RE/Si ratio and Yb/Ho ratio are comprehensively investigated. Chemical stability and thermal expansion compatibility between SiC and RE silicates with different compositions are also validated. Optimized materials for the application of environmental barrier coating and interphase are screened respectively according to above trends and data. This work is a case study to establish a composition-property library for RE_2O_3-SiO_2 compounds. It is inspired more complicated multicomponent RE silicates could be prepared and characterized by high throughput experimentation, accelerating the design and screening of promising optimal candidates.

2:40 PM

(ICACC-S2-041-2023) Phase Stability and Sintering Resistance of Mulltiphase Rare Earth Aluminate-Zirconate Materials as Prospective T/EBC Materials

Y. Yu[2]; D. L. Poerschke*[1]

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2. University of Minnesota, Chemical Engineering and Materials Science, USA

Rare-earth (RE) aluminates and zirconates are candidates for next-generation thermal barrier coatings (TBC) with exceptional thermal stability and CMAS resistance. Key questions for the design of these materials include understanding the effects of the RE cation identity on the stability of specific aluminate assemblages, and the attendant effects on the sintering resistance of the material. This study investiaged several classes of aluminate-zirconate materials based on Yb, Y, and Gd with systematic variations in the Al:RE:Zr ratios. Particualry slow densification and grain growth were observed in the samples containing yttrium aluminum garnet (YAG) and gadolinium aluminum garnet (GdAG) at 1400 °C. The garnet plays a crucial role in preventing sintering by impeding diffusion; and this effect subsides when the metastable GdAG transforms to gadolinium aluminum perovskite (GdAP) and alumina at 1500 °C. Faster sintering was observed for materails containing perovskite or monoclinic aluminates. Multi-RE cation design approaches showed that even small additions of a smaller RE cation such as Y or Yb can stabilize the sintering-resistance in the Gd-based system and provided information about the relative stability and RE partitioning between phases. These insights are useful to guide next-generation T/EBC design.

3:20 PM

(ICACC-S2-042-2023) Modeling oxidation kinetics of mullite-silicon composites

J. Wan*[1]

1. GE Research, USA

In order to evaluate the potential of oxide/nonoxide composites for application in engine combustion environments, a first principle analytical model was developed to account for the oxidation kinetics of mullite and silicon composites. Diffusion of oxidants, either oxygen or water vapor, through a bi-continuous network of mullite and silica controls the rate of oxidation, which leads to a parabolic relationship between oxidized layer thickness versus time. Rate constants were calculated over different temperature ranges. At higher temperatures, diffusion of oxygen through mullite matrix controls the oxidation rate. At lower temperatures, oxygen or water diffusion though silica dominates. The model was validated experimentally by oxidation in mullite-Si sintered pellets and air plasma sprayed coatings.

3:40 PM

(ICACC-S2-043-2023) Environmental barrier coating to mitigate ignition-risk in high pressure oxygen-rich environments

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1. Massachusetts Institute of Technology, Aeronautics and Astronautics, USA
2. Massachusetts Institute of Technology, Materials Science and Engineering, USA

Oxygen-rich turbopumps are essential to proposed oxidizer-rich and full-flow staged combustion rocket engines currently under development. The turbine in an oxygen-rich turbopump subjects materials to high-pressure, high-temperature gaseous oxygen environments where there is a risk of metal fires, a catastrophic single-point failure mode. A candidate approach for mitigating the risk of a specific ignition source, particle impact ignition, is to coat metallic components with an inert ceramic environmental barrier coating; however, such coatings are susceptible to delamination under the rapid thermal transients on engine startup and shutdown. Here, we investigate the delamination risk of a novel ductile phase-toughened composite environmental barrier coating under a notional flight cycle for a reusable boost-stage engine where the engine restarts four times. Our results show that hot shock from ignition during take-off gives rise to the largest energy release rate for EBC delamination, of order 100 J/m^2. While such energy release rates will delaminate conventional monolithic ceramic coatings, our calculations show that the present coating is likely sufficiently tough due to crack-bridging to resist delamination.

4:00 PM

(ICACC-S2-044-2023) Creep behavior of an environmental barrier coating under thermal gradient

I. Hamadouche*[1]; T. Archer[1]; T. Vandellos[2]; P. Beauchêne[1]; F. Hild[3]

1. ONERA, France
2. Safran Ceramics, France
3. LMPS ENS Paris Saclay, France

EBCs are currently developed to protect CMCs from extreme environments. It therefore is essential to understand the behavior of the CMC/EBC system under representative thermomechanical loadings. In particular, the creep behavior of EBCs was shown to be one of the lifetime limiting mechanism. The main objective of the test considered herein was to identify the creep behavior of the system in a 4-point bending test under thermal loading. It was based on laser beam inducing 3D thermal gradients. An infrared camera enabled temperature fields to be quantified at the surface of the coating. Under such conditions, the specimen expanded and deflected. Displacement fields are measured using two visible light cameras on both sides of the sample via digital image correlation. The mechanical load was first applied. Then, the thermal loading was applied on the center of the coating surface up to 1250C. The laser was maintained for two hours and then turned off instantaneously. Thermal and kinematic fields were measured for comparison with finite element simulations, using a weighted Finite Element Model Updating method. The identification of thermal boundary conditions showed different results depending on the area of minimization. Mechanical identification allowed the creep behavior of the coating to be quantified. Effects of temperature and stress are key parameters for quantifying the creep behavior of EBCs.

4:20 PM

(ICACC-S2-045-2023) Effect of TEBC on the Performance of Al_2O_3/Al_2O_3 Ceramic Matrix Composites

P. Mechnich*[1]; G. Alkan[1]; F. Flucht[1]

1. DLR - German Aerospace Center, Institute of Materials Research, Germany

State-of-the art oxide/oxide ceramic matrix composites (CMC) based on Al_2O_3 fibers and porous Al_2O_3 matrices and are developed for various applications in harsh environments such as combustion atmospheres. Thermal-chemical degradation of such CMC includes fiber grain growth, matrix densification, fiber/matrix sintering, surface reactions with mineral dusts or molten salts, and hydroxylation/volatilization of Si- or Al-hydroxide species. Additionally, the relatively weak porous oxide matrices make CMC surfaces prone to surface erosion e.g. in high velocity gas flows or mechanical impact/abrasion. Low-thermal conductivity and environmentally stable protective oxide coatings promise effective thermal protection, improved chemical stability, reduced gas permeability, and erosion/abrasion resistance. As typical coating deposition processes are introducing additional thermal loads or surface chemical reactions, coatings may also significantly impact the CMC substrate. The effect of coatings on the overall performance of coated Al_2O_3/Al_2O_3 CMC materials is discussed on the examples of slurry-based, reaction-bonded Al_2O_3 and APS Y_2O_3 coatings.

S3: 20th International Symposium on Solid Oxide Cells (SOC): Materials, Science and Technology

Simulation and Testing

Room: Ponce de Leon (North Tower)
Session Chairs: Henrik Frandsen, Technical University of Denmark; Kevin Huang, University of South Carolina

1:30 PM

(ICACC-S3-036-2023) Stack-Scale Modeling of Solid Oxide Cells (Invited)

O. Babaie Rizvandi*[1]; H. L. Frandsen[2]

1. Technical University of Denmark, Department of Energy Conversion and Storage, Denmark
2. Technical University of Denmark, Department of Energy Conversion and Storage, Denmark

Solid oxide cell (SOC) offers high efficiency and fuel flexibility due to its high operating temperature, which makes it an appealing and promising energy conversion technology. Many studies have been devoted to investigating this technology, and additional studies are being conducted to ensure its widespread use and commercialization. Numerical studies are necessary for SOCs since their experiments are costly and timely and do not reveal much information about the detailed distribution of important quantities such as temperature and current density. Nonetheless, detailed SOC models have a high computational cost owing to interactions of various physics in different layers. Thus, SOC models are challenging even at the cell and repeating unit levels. Therefore, for lower dimensional models, such as 1D and 2D, high-fidelity models that include most of the physics and interactions are used, while simplified models are applied to 3D models. In our studies, we have been using a homogenization approach to model the entire SOC stack. This approach replaces the layered domains of the stack with equivalent domains and solves for the effective modeling variables using effective modeling properties for each physic. With such an approach, SOC stacks can be modeled on typical workstations within a feasible runtime. The model has been applied to various stack-scale studies, including degradation, high-pressure, and ammonia-fueled operations.

2:00 PM

(ICACC-S3-037-2023) Experimental and computational investigations of the multiple impurities effect on the SOFC cathode materials

R. Wang*[1]; L. R. Parent[3]; S. Gopalan[2]; Y. Zhong[1]

1. WPI, Mechanical and Materials Engineering, USA
2. Boston University, Mechanical Engineering, USA
3. University of Connecticut, USA

On purpose of studying the multiple impurities poisoning phenomena in the SOFC cathode systems, two common cathode materials, namely $(La_{0.8}Sr_{0.2})_{0.95}MnO_3$ (LSM) and $(La_{0.6}Sr_{0.4})_{0.95}(Co_{0.2}Fe_{0.8})O_3$ (LSCF) were prepared, sintered and finally annealed at 800, 900, and 1000°C at different impurity-containing atmospheres, respectively. By means of X-Ray Diffraction (XRD), Scanning Electron Microscope (SEM) and Transmission electron microscopy (TEM) technique as well as the CALPHAD (Computer Coupling of Phase Diagrams and Thermochemistry) methodology, the secondary phases under different temperatures, P_{O2}, and partial pressure of impurities were well predicted as well as experimentally verified correspondingly. Comprehensive comparisons among the three candidates under different impurity-containing conditions would also be made to provide guidance on the alternatives of the cathode materials. Finally, the accelerated tests, in some cases, were validated in our work which might also be scrutinized in the future as they may present different degradation mechanisms.

2:20 PM

(ICACC-S3-038-2023) A Machine Learning Framework for Rapid Assessment and Optimization of SOC Electrodes from Low Resolution Data

W. K. Epting[1]; H. W. Abernathy*[1]; Y. Lei[1]; G. Hackett[1]; T. Kalapos[1]

1. US DOE National Energy Technology, USA

In this work, machine learning and computer vision techniques were trained with a large bank of data to produce a reduced order predictor of long-term performance of solid oxide cells (SOCs) in both fuel and electrolysis mode. This approach uses results from high-fidelity predictive modeling of thousands of unique electrode configurations. After training, a neural network-based predictor can rapidly predict lifetime cell performance and can be interrogated to determine the impact of different parameters and to generate recommendations for electrode improvement. A convolutional neural network (CNN) model was then developed to predict microstructural properties from low-resolution microstructural data similar to that from a microscale X-ray CT, with resolution far coarser than the characteristic feature size. The predicted microstructural properties can then be used with the model to generate predictions of lifetime performance and tailored improvement recommendations. Preliminary aggregate error analysis shows sub-1% error in these predictions. This framework allows cell developers and other stakeholders to evaluate their cells rapidly, either during development or as a form of quality control, using more widely accessible and rapid techniques such as microscale X-ray computed tomography.

2:40 PM

(ICACC-S3-039-2023) Insights in the temperature dependence of area specific resistance of SOFC stack

M. Kusnezoff*[1]; S. Megel[1]; N. Trofimenko[1]; S. Rothe[1]; W. Beckert[1]; J. Schoene[1]; S. Hielscher[1]; A. Michaelis[1]

1. Fraunhofer IKTS, Germany

Area specific resistance (ASR) of SOFC stack is a product of cell ASR, contacting losses and operating conditions. Depending on operating temperature range the activation energy for stack ASR can change considerably. The experimental investigations on stack ASR operated in dry and humidified hydrogen show that at operating temperatures above 830°C the CFY stack performance is nearly independent on operating temperature. The detailed investigations and comparison with single cell ASR, activation energy of oxide scale and protective layer conduction show that contributions of different constituents change considerably changing the operating temperature. It was found that especially under dry operating conditions the gas conversion impedance contribution, which increases with temperature rise, compensates the ASR drop coming from cell, what is the main reason for total ASR stagnating behaviour.

3:20 PM

(ICACC-S3-040-2023) Using simulation to study solid oxide cell degradation (Invited)

H. W. Abernathy*[1]; W. K. Epting[2]; Y. Lei[2]; T. Yang[2]; P. Salvador[3]

1. National Energy Technology Laboratory, Thermal Sciences, USA
2. US DOE National Energy Technology, USA
3. Carnegie Mellon University, Materials Science and Engineering, USA

Extending solid oxide cell (SOC) lifetime is a major focus of the US Department of Energy's SOFC Program to help reduce system costs. As long duration experiments are cumbersome, characterization and performance data from selected longer cell tests can be used to inform and calibrate performance degradation models. Subsequent simulations are then used to explore a wider experimental space with respect to operating conditions and electrode microstructure/composition. The goal of the simulations is to analyze all of the simulated data to provide guidance on which cell features to focus research efforts to see the greatest gain in long-term performance. The lessons gained from such simulations can be impacted by multiple factors, including the methods and data used to calibrate the models, the nature of the models used, and the performance metrics chosen for optimization. This talk will provide quantitative examples of how changes in methods or data propagate through the simulation and analysis process and how those changes impact final conclusions.

3:50 PM

(ICACC-S3-041-2023) Ni Migration in Porous Ni-YSZ Electrodes During Electrolysis (Invited)

Q. Zhang[1]; D. Cox[1]; S. Du[2]; A. Chadwick[1]; K. Thornton[2]; S. Barnett[1]; P. Voorhees*[1]

1. Northwestern University, USA
2. University of Michigan, USA

Long-term performance degradation remains a key issue that may limit the use of Solid Oxide Electrolysis Cells (SOECs). Nickel-Yttria stabilized Zirconia (Ni- YSZ) fuel electrodes have been widely used in SOECs, but their performance typically degrades over time. One key degradation mechanism that has been identified is directional Ni migration and disconnection near the electrode/electrolyte interface. A phase field model is developed under the assumption that a gradient in Ni/YSZ interface energy drives Ni migration. The large-scale numerical simulations utilize an experimentally measured 3D Ni-YSZ-pore microstructure as the initial condition and are implemented for two possible Ni transport mechanisms – surface diffusion and capillary driven evaporation/condensation. The results show significant Ni migration away from the electrode/electrolyte interface within the electrode active region, along with a tendency for Ni to become isolated from the surrounding Ni network.

4:20 PM

(ICACC-S3-042-2023) Simulating Ni redistribution in the hydrogen electrodes of solid oxide cells: The effect of initial microstructural properties

Y. Lei*[1]; Y. Lee[1]; W. K. Epting[1]; J. H. Mason[1]; T. Cheng[1]; H. W. Abernathy[1]; G. Hackett[1]; Y. Wen[1]

1. US DOE National Energy Technology, USA

Ni redistribution has historically proven to be an important degradation mechanism in the hydrogen/steam electrodes of solid oxide fuel cells/solid oxide electrolysis cells. Experimental evidence from the literature on the Ni redistribution has been inconsistent regarding the direction of the redistribution at different operating conditions. The recently developed modeling framework in this study shows that some variability in the direction of Ni redistribution may be explained by the competition between two different mechanisms of Ni redistribution, namely $Ni(OH)_2$ diffusion and changes in Ni-YSZ wettability. This work explores the effect of the initial microstructural properties (e.g., porosity, tortuosity factor of pore, and volume fraction ratio between YSZ and Ni) on the Ni redistribution. Changing the pore tortuosity factors or volume fraction ratio between YSZ and Ni can reverse the direction of the Ni redistribution, while the porosity also affects the rate of Ni redistribution. These microstructural properties impact the distribution of steam partial pressure and overpotential in the electrode, which are driving forces of Ni redistribution in the utilized model. These results provide insight on possible mitigation of Ni redistribution through tuning not only the operating conditions, but also the microstructural properties of the electrode.

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(ICACC-S3-043-2023) Reactive Molecular Dynamics Simulations for Microstructural Changes in Ni/YSZ Cermet in a Solid Oxide Fuel Cell Anode

T. Ishikawa*[2]; Q. Chen[1]; Y. Asano[2]; Y. Ootani[2]; N. Ozawa[1]; M. Kubo[2]

1. Tohoku University, New Industry Creation Hatchery Center, Japan
2. Institute for Materials Research, Tohoku University, Japan

Solid oxide fuel cells (SOFCs) have the advantage of high power generation efficiency without noble metal catalysts. However, when the concentration of water vapor in hydrogen fuel increases due to fuel starvation, the activity of Ni/YSZ (Yttria Stabilized Zirconia) cermet at the anode degrades. Some experiments show that the microstructure of Ni/YSZ cermet changes during this degradation due to the splitting and diffusion of Ni particles. Therefore, it is essential to clarify the structural change mechanism under a highly humidified hydrogen environment to develop highly durable anode materials. In this study, we investigated the structural change in Ni particle/YSZ(111) under vacuum and highly humidified hydrogen environments by reactive molecular dynamics simulation. Firstly, we adjusted the Ni and O parameters for simulations. Under a vacuum environment, the contact angle of the Ni particle was approximately 130°, which is close to the experimental value. Moreover, under the highly humidified hydrogen environment, we observed migration of Ni atoms at the Ni/YSZ interface, indicating an initial process of microstructural change such as splitting and diffusion. This study will help to clarify very complicated phenomena of chemical reaction and atom diffusion for highly durable Ni/oxide cermets.

S4: Armor Ceramics - Challenges and New Developments

Recent Progress in Diamond-Ceramic Composites

Room: Coquina Salon B (North Tower)
Session Chair: Jerry LaSalvia, DEVCOM Army Research Laboratory

1:30 PM

(ICACC-S4-001-2023) Microstructure and properties of silicon infiltrated diamond-SiC composites with high diamond content (Invited)

M. Herrmann*[1]; B. Matthey[1]; S. Kunze[1]

1. Fraunhofer IKTS, Germany

Silicon carbide bonded diamond composites are one class of materials with a high potential for a wide range of challenging applications in the industry. The high hardness (> 35-40 GPa) and very high wear resistance meet the requirements for wear components of modern industry. Silicon carbide bonded diamond composites can be produced by pressure less infiltration of diamond preforms with Si. This process is like the production of silicon infiltrated SiC. The materials have a lower diamond content in comparison to HPHT diamond composites. However, by using multimodal particle packing diamond contents of 60-65 % by volume in the composites can be achieved. Beside the high wear resistance, the materials exhibit also excellent thermal conductivity with values of up to 600 W/m*K. Systematic experiments show the correlation of microstructure, diamond distribution and thermal properties. The influence of the microstructure on thermal conductivity and its temperature dependence will be discussed. Beside these results, also some microstructural key features of silicon carbide bonded diamond composites will be presented, which should explain the excellent properties of this class of materials. One feature is the mechanically strong interface between diamond and SiC. The properties of the interface as a function of the composition will be discussed on the bases of micromechanical tests.

2:00 PM

(ICACC-S4-002-2023) Effect of Temperature and Time on Diamond Graphitization in a Reaction Bonded Diamond Silicon Carbide Composite

S. D. Walck*[1]; S. G. Hirsch[1]; A. A. DiGiovanni[1]; J. LaSalvia[1]

1. DEVCOM Army Research Laboratory, USA

Superhard diamond-silicon carbide (SiC) composites for wear and thermal management applications can be synthesized at relatively low processing temperatures, often less than 1600°C, by the reaction-bonding approach. Despite the low synthesis temperatures, it is still possible for graphitic phases to nucleate and grow at the diamond/SiC interface. The presence of a graphitic interlayer can be detrimental to mechanical and physical properties such as hardness and stiffness. This investigation examines the effects of heat-treatment temperature and time on diamond graphitization in a commercial reaction bonded diamond-SiC composite. Characterization of microstructure and diamond graphitization are performed using XRD, SEM, STEM, EELS, and CBED of selected areas. The presence, nature, and growth of the graphitic phase are discussed in the context of the heat-treatment conditions and diamond particle size/morphology. Additionally, thickness of the graphitic interlayer is compared with a growth-based graphitization model.

2:20 PM

(ICACC-S4-003-2023) Effect of Diamond Content on the Densification, Microstructure, and Residual Stress of Hot-Pressed Al_2O_3 - Diamond Particulate Reinforced Composites

T. W. Moore*[1]; K. D. Behler[2]; A. A. DiGiovanni[2]; M. C. Guziewski[2]; J. LaSalvia[3]

1. DEVCOM-Army Research Lab, SURVICE Engineering, USA
2. DEVCOM-Army Research Lab, Ceramics and Transparent Materials Branch, USA
3. U.S. Army Research Laboratory, DEVCOM, USA

The effect of diamond content (20 – 60 vol%) on the densification, microstructure, and residual stress of hot-pressed Al_2O_3 – diamond particulate ceramic composites are reported. Powder mixtures of 30-40 micron TiC-coated diamond particles and submicron Al_2O_3 powder were hot-pressed at temperatures from 1300°C to 1500°C under flowing high-purity Ar for 60 to 180 mins at 48MPa. Heating and cooling rates were initially 50°C/min, then were lowered to 10°C/min to inhibit microcracking due to thermal residual stresses. Final densities were determined by the Archimedes method, while microstructures were determined by examining both fracture surfaces and ion-polished cross sections. Fully dense composites were achieved for diamond loadings up to 50 vol%. For high diamond loadings, observed microcracking indicate significant residual stresses due to differences in thermal expansion coefficients and elastic moduli between the diamond particles and Al_2O_3 matrix, consistent with modeling. Experimental procedures, results, and analysis of residual stresses will be presented.

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(ICACC-S4-004-2023) Effects of Diamond Particle Size and TiC-coating on the Phases and Microstructure of Hot-Pressed SiB_6/Diamond Powder Mixtures

J. LaSalvia*[1]; S. D. Walck[1]; C. Garcia[2]; T. W. Scharf[2]; A. A. DiGiovanni[1]

1. DEVCOM Army Research Laboratory, USA
2. University of North Texas, Department of Materials Science and Engineering, USA

Hot-pressing diamond particulate ceramic composites under low pressure/high temperature conditions presents significant challenges to overcome including densification, graphitization, and possible reactions in order to achieve desired properties and performance. Here, the effects of diamond particle size (30-40 mm vs. 109 mm) and TiC-coating (30-40 mm) on phases and microstructures of hot-pressed SiB_6/Diamond powder mixtures are reported. SiB_6/Diamond powder mixtures with a 25 vol% diamond content were hot-pressed to near full density at 1600°C for 60 mins under flowing high-purity Ar and an applied stress of 48 MPa. XRD was used to initially identify phases, while SEM, STEM, EDS, and EELS were used to characterize microstructures. Microstructures show a marked effect of reactions between the diamond particles and SiB_6 matrix on their formation. Based on observations, an understanding on the effects of diamond particle size and TiC-coating on phase and microstructure formation is presented. Findings and experimental procedures will be discussed.

Advances in the Role of Interfaces on Sintering, Microstructure, and Mechanical Properties

Room: Coquina Salon B (North Tower)
Session Chair: Scott Walck

3:20 PM

(ICACC-S4-005-2023) Controlling Densification and Grain Growth During Sintering via Dopants: Doping Alumina with Carbon (Invited)

L. Cohen[1]; R. Marder[1]; W. D. Kaplan*[1]

1. Technion - Israel Institute of Technology, Dept. of Materials Science and Engineering, Israel

Alumina (α-Al_2O_3) is commonly sintered in air, but some sintering techniques such as spark plasma sintering (SPS) use graphite dies. Alumina sintered in graphite furnaces often has a dark grey or even black color, and there is debate in the literature regarding the actual role of carbon on sintering and grain growth. The solubility limit of carbon in 99.99% pure alumina equilibrated at 1600°C under flowing He in a graphite furnace was measured using a wavelength dispersive spectrometer mounted on a scanning electron microscope. The solubility limit of carbon in alumina was found to be 5300 at. ppm, and it is believed that at low oxygen partial pressures carbon-hydrogen species substitute oxygen, which is charge-compensated by oxygen vacancies. Doping alumina with carbon at concentrations below the solubility limit does not impede densification and reduces grain growth. Doping alumina with carbon above the solubility limit hinders densification during sintering. The mechanism by which carbon influences densification and grain growth will be discussed. The influence of carbon on processing alumina will be compared to that of other dopants such as Mg, Ca, and Si.

3:50 PM

(ICACC-S4-006-2023) Kinetics of multiple complexion transitions at 1800 °C in Eu-doped MgAl2O4 (Invited)

C. Marvel*[1]; C. Riedel[2]; A. Koenig[2]; M. Harmer[2]

1. Louisiana State University, Mechanical and Industrial Engineering, USA
2. Lehigh University, USA

Studies on grain boundary complexions rarely correlate initial microstructures and annealing heating rates to complexion nucleation and propagation kinetics. Here, starting grain size distributions and initial grain boundary complexion types of as-sintered Eu-doped MgAl2O4 samples were characterized, heat treatments with varying heating rates (different ‘dwell’ times prior to reaching annealing temperatures) were completed, grain growth behaviors, particularly the onset of abnormal grain growth, were monitored as proxies for detecting complexion transitions, and grain boundary complexion types were re-determined after complexion transitions were detected. This work is inspired by a computational complexion time-temperature-transformation (TTT) diagram that provokingly suggested abnormal grain growth is delayed at high temperatures, as compared to lower temperatures, because ultrahigh complexion nucleation rates limit incubation times for abnormal grains to develop. Thus, annealing at high temperatures (1800 °C) and short annealing times (1-30 min) were emphasized. Overall, multiple generations of abnormal grain growth were observed following different times during the isothermal anneals, thereby suggesting multiple different grain boundary complexion transitions occurred. Importantly, the faster heating rates produced fewer abnormal grains, confirming the behavior discovered in the TTT diagram.

4:20 PM

(ICACC-S4-007-2023) Absolute Surface Energies of Yttrium Oxide

K. Joshi*[1]; J. Mason[2]; R. Castro[1]

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

Nanoceramics have unique properties compared to bulk materials, primarily because of their large interfacial areas. This gives rise to considerable surface and grain boundary energies that play an important role in thermodynamic stability and the sintering process. During sintering, densification of the nanoceramic occurs by the mass transport phenomena, and the reduction of interfacial energy is the driving force for this process. Experimental thermodynamic data on surface energies, and in particular on free surface energies, is lacking though. Yttrium oxide is selected as a model material because of its wide range of applications and abundant sintering studies in the literature. Faceted nanocrystalline yttrium oxide was synthesized by hydrothermal synthesis, and the morphology of these nanoceramics was studied using electron microscopy. Surface energies of specific planes were determined experimentally by water adsorption calorimetry using an experimental setup that includes a water micro-dosimeter combined with a micro-calorimeter. This characterization technique is based on the thermodynamics of water adsorption on the anhydrous surface of the ceramics. Molecular dynamic simulations were also performed to estimate the free surface energies, and the segregation of dopants to the surface was studied based on their segregation enthalpies.

4:40 PM

(ICACC-S4-008-2023) Grain boundary role on hardness of Gd- doped and undoped nanocrystalline YSZ

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Nanocrystalline transparent ceramics (NNC) are of great interest for armored tactical platforms, sensor array compartments, or other optically transparent window applications. Their remarkable properties arguably come from the extended role that interfaces, particularly grain boundaries, play on these materials. This project aims to fundamentally understand the effects of grain boundary energies (GBE) on the hardness of dense NCC and explore the potential control of grain boundary energy excess via dopant segregation. For this purpose, Yttria-stabilized cubic zirconia (YSZ) was doped with 2, 4, and 8 mol% of Gd2O3. Fully dense nanocrystalline samples were sintered via spark plasma sintering (SPS). Gd-doped samples have shown increased hardness compared to undoped YSZ with the same grain size. In addition, the solubility limit, microstructure, dopant segregation, and crack pattern have been investigated to support a future discussion on the impact of the thermodynamics of interfaces on hardness.

5:00 PM

(ICACC-S4-009-2023) On the Terminal Velocity of Cracks in Glasses and Ceramics with Relevance to Armor Applications

G. D. Quinn*[1]

1. National Institute of Standards and Technology, Materials Measurement Sciences Division, USA

The terminal crack velocity, v_{ct}, of cracks in ceramics and glasses is limited by several factors. It is well known that v_{ct} is limited by elastic wave velocities that control how strain energies move with the fast crack tip, but there is more to it than that. Fast cracks are three-dimensional. V_{ct} is limited by three-dimensional perturbations at the crack tip. The perturbations include mist and hackle as well as local deviations caused by the material's micro- and nanostructure. New illustrations show these effects. Older notions of voids opening up in front of fast-moving cracks, that may be suitable for polymers and metals, are not relevant to ceramics and glasses. Theoretical predictions of v_{ct} by the Mott-Roberts-Wells model of the 1940s match experimental measurements for some key glasses, including fused silica and low-density borosilicates. Their model is essentially the Griffith equation modified by a term for the kinetic energy of the crack. Slower v_{ct} results obtained for many other glasses are due to structural variations that cause crack front perturbations. Similar conclusions apply to ceramics as well.

S6: Advanced Materials and Technologies for Rechargeable Energy Storage

Materials Design, Screening, and Electrode Architectures for Lithium Batteries I

Room: Coquina Salon E (North Tower)
Session Chairs: Dany Carlier, CNRS, University Bordeaux; Olivier Guillon, Forschungszentrum Juelich

1:30 PM

(ICACC-S6-030-2023) Solvation Structure and Ion Transport in Highly Concentrated Li salt/Sulfone Electrolyte Solutions (Invited)

Y. Ugata*[1]; K. Ueno[1]; M. Watanabe[1]; K. Dokko[1]

1. Yokohama National University, Japan

Sulfone-based highly concentrated electrolytes (HCEs) have attracted attention for the improvement of thermal stability and operating voltage of Li-ion batteries. We report here that thermal properties, solvation structures, and transport properties of HCEs composed of $LiBF_4$ and various sulfone solvents, sulfolane (SL), 3-methylsulfolane (MSL), dimethyl sulfone (DMS), ethyl methyl sulfone (EMS), and ethyl isopropyl sulfone (EiPS). $LiBF_4$ formed equimolar crystalline solvates at room temperature with SL and DMS, which are symmetric sulfones. In contrast, $LiBF_4$/asymmetric sulfone (MSL, EMS, and EiPS) mixtures maintain their liquid states at room temperature over wide composition ranges. Raman spectroscopy revealed that network structures of Li^+–sulfone–Li^+ and Li^+–BF_4^-–Li^+ are formed in the HCEs. The self-diffusion coefficients of ions and solvent measured with pulsed-field gradient NMR suggested that Li^+ ion diffuses more rapidly than sulfone and BF_4^- in the HCEs of $[LiBF_4]/[sulfone] > 1/2$ except in the EiPS system, and Li^+ ion hopping conduction evolves through ligand exchange. The cyclic sulfone-based electrolytes (SL and MSL systems) exhibited higher self-diffusion coefficient ratios of Li^+/sulfone (D_{Li}/D_{sol}) and Li^+/BF_4^- (D_{Li}/D_{BF4}) compared to the acyclic sulfone-based electrolytes at high concentrations, suggesting that the cyclic sulfones facilitate Li^+ ion hopping.

2:00 PM

(ICACC-S6-031-2023) Silicon Oxynitrides Derived from Agricultural Waste for Li^+ Batteries

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1. University of Michigan, Macromolecular Science and Engineering, USA
2. University of Michigan, Materials Science and Engineering, USA

Hitherto, comprehensive studies have demonstrated silicon nitrides (SiN_x) a conversion type anode for Li^+ batteries (LIB) that forms Li_3N during the first cycle. SiN_x exhibit high capacities (>2000 mAh/g), high electrical conductivities (~10^{-3} S/cm at room temperature), and low potentials close to Li. However, all current SiN_x anodes studied are thin films or nanoparticles produced via high-cost and not easily scaled methods. In contrast, electrochemical investigation of metal oxynitrides is not as extensive as metal nitrides due to restrictions from conventional ceramic nitridation methods and technical difficulties in determining N:O ratios. SiO_xN_y is typically regarded as a mechanical stabilizer without electrochemical activities. We have reported an environmentally-benign method of synthesizing Si_2N_2O nanocomposites from agricultural waste, e.g., rice hull ash (RHA). When examining the feasibility of using SiN_xO_y as a LIB electrode, it was observed that these Si_2N_2O nanocomposites as anodes could achieve specific capacities of >900 mAh/g on long-term cycling. In an effort to prelithiate the system, we find that heating RHA precursor with Li_2CO_3 provides access to lithium silicon oxynitride (LiSiON) coincidently during carbonitriding. The lithiated components facilitate Li^+ diffusion and consequently improve their performance at high C rates. A number of aspects of these efforts will be presented.

2:20 PM

(ICACC-S6-032-2023) Atom efficient provision of lithium and sodium sulfide (M_2S; M=Li, Na) cathode material by air-stable single molecular sources

V. Brune*[1]; D. Patrun[1]; S. Mathur[2]

1. University of Cologne, Inorganic/Materials Chemistry, Germany
2. University of Cologne, Institute of Inorganic Chemistry, Germany

Large-scale energy storage will play a key role in future sustainable and rechargeable energy storage technologies calling from the enormous increase in demand of world energy consumption. Chalcogenide-based electronic materials, especially Li_2S and Na_2S, are promising future alternative regarding their high theoretical gravimetric energy density as well as the highly earth abundance of sulfur, which is economically priced and non-toxic. Recently, lithium sulfide (Li_2S) has received much attention as an electrochemically active cathode material in lithium-sulfur batteries. The chemical approach from a fully characterized single molecular source, where the metal sulfur bond is already preformed, creates the advantages of softer product formation conditions, avoiding the use hazardous byproducts and enabling an atom efficient pathway to the desired electronic material. Good solubility of synthesized molecular source in various solvents allows simple handling and an innovative preoperational fabrication of carbon embedded 3D fiber matts out of M_2S 1D structures by electrospinning processes. Crystalline Li_2S loaded fiber matts shows capacity retention of 73% over 100 cycles and a capacity of about 400 mAh g^{-1} at 1 C. This single molecular approach enable future alkali metal free batteries with a higher safety level.

2:40 PM

(ICACC-S6-033-2023) Carbon-Coated Electrospun Nickel Vanadate Nanofibers as High-Performance Anode Material for Lithium-Ion Batteries

A. Bhardwaj*[1]; K. K. Halankar[1]; M. Wilhelm[1]; T. Fischer[1]; S. Mathur[1]

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

Transition metal vanadate material has attracted many battery researchers due to its layered type structure enabling facilitated lithium ion insertion/extraction and ability of vanadium to exist in multiple valence states. Nickel vanadates have been receiving much research attention as superior anode candidates for use in lithium-ion batteries with high specific capacity. Here, synthesis of 1-D nanofibers comprised of nickel vanadate ($Ni_3V_2O_8$) nanofibers via electrospinning and subsequent thermal treatment in air atmosphere is reported. In order to improve the electrochemical properties, the $Ni_3V_2O_8$ NFs were coated with a thin layer of conductive carbon via plasma-enhanced chemical vapor deposition (PE-CVD) technique. Materials synthesized thus were characterized extensively for the physicochemical properties and used to evaluate as anode materials for Lithium-ion batteries. The discharge capacity, rate capability and coulombic efficiency of carbon-coated $Ni_3V_2O_8$ NFs were compared with carbon-free nanofibers for several charge-discharge cycles. The results indicate that electrospun nanofibers carbon-coated through PE-CVD technique could offer valuable materials for energy storage devices.

Materials Design, Screening, and Electrode Architectures for Lithium Batteries II

Room: Coquina Salon E (North Tower)
Session Chairs: Valerie Pralong, CNRS ENSICAEN; Yosuke Ugata, Yokohama National University

3:20 PM

(ICACC-S6-034-2023) Formation, sintering, and mechanical properties of a novel $Li_7La_3Zr_{0.5}Nb_{0.5}Ta_{0.5}Hf_{0.5}O_{12}$ high-entropy Li-garnet

Z. Fu*[1]

1. Penn State Harrisburg, USA

We studied the formation, sintering, and mechanical properties of $Li_7La_3Zr_{0.5}Nb_{0.5}Ta_{0.5}Hf_{0.5}O_{12}$ (LLZNTH) high-entropy Li-garnet with the comparison with $Li_{6.75}La_3Zr_{1.75}Ta_{0.25}O_{12}$ (LLZT). The study indicates that the LLZNTH sample has a finer particle size after both calcination and ball mill than LLZT. Both samples can be densified to a relative density up to ~93-94% at a temperature of 1100°C, however, they show significantly different sintering and grain growth behaviors. The LLZNTH sample takes 16 hours to reach the maximum relative density while the LLZT samples only need 12 hours. LLZNTH sample has a significantly lower grain growth parameter due to the sluggish effects of high-entropy compounds so that it maintains fine microstructures (grain size ~10μm) than the LLZT sample (grain size over 100 μm). Due to the fine microstructures, the LLZNTH sample shows both higher flexural strength (84.8±6.9MPa compared with 47.9±10.1MPa) and hardness (8.5±0.8GPa compared with 7.7±0.4GPa) than the LLZT sample. Ionic conductivity characterizations indicate that the LLZNTH sample shows a moderate conductivity of 4.67×10^{-4} S cm^{-1} at room temperature and a low activation energy of 0.25eV.

3:40 PM

(ICACC-S6-035-2023) Thermodynamic and Thermophysical Properties of Glass-Ceramic Solid Electrolytes

M. Rohde*[1]; H. J. Seifert[1]

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

While there are numerous studies on the ionic transport in Li- or Na- conducting solid electrolytes, respectively, the thermophysical properties have been studied only in a small number of publications. Particularly, the thermal transport behavior and the specific heat of glass-ceramic Li- or Na-based titanium or germanium phosphates have only rarely been investigated up to now. In this work we have studied different compounds out of the family of NASICON (Na Superionic Conductor) structured ceramic systems, which are candidate materials for solid state electrolytes for Li-ion cells but also for systems post Lithium ion technology. Namely, LAGP ($Li_{1+x}Al_xGe_{2-x}(PO_4)_3$, x=0.3, 0.5, 0.7), LATP ($Li_{1+x}Al_xTi_{2-x}(PO_4)_3$, x≈0.5) and NATP ($Na_{1+x}Al_xTi_{2-x}(PO_4)_3$, x≈0.3) as well as NaZSiP ($Na_{1+x}Zr_2(SiO_4)_x(PO_4)_{3-x}$, x = 1.7, 2.0) substrates were prepared using a melt quenching or a sol-gel route, respectively. In order to develop a better understanding of the relationship between the specific microstructure and the ionic conductivity as well as the thermodynamic properties the samples were characterized by applying thermophysical and calorimetric measurement techniques. The ionic conductivity was measured using impedance spectroscopy while the thermal diffusivity and the specific heat were determined by Laser Flash technique and differential scanning calorimetry, respectively.

4:00 PM

(ICACC-S6-036-2023) Modified separator for trapping poly-sulphides in lithium sulphur batteries

K. K. Halankar*[1]; S. Mathur[1]

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

The lithium-sulfur battery has gained attention as the next-generation battery as it can theoretically exhibit a specific energy density that is approximately 5 times higher than that of LIBs. However, there are several challenges to overcome for the commercialization of lithium-sulfur batteries. In present work, transition metal phosphide modified separator is used to ensure prevention of dissolution of lithium polysulfides that are generated as an intermediate product with repeated charge/discharge. This modified separator provides effective trapping of lithium polysulfides, catalyzed sulfur redox, as well as suppressed formation of Li dendrites in one configuration. The polysulfide shuttle is reduced considerably which has known as the biggest obstacle to the commercialization of the lithium-sulfur battery as this problem is directly linked to the longevity and safety degradation of battery. The synergistic effect of the modified separator results in elevated sulfur redox kinetics, improved sulfur utilizations and stable charge/discharge cycling with high sulfur contents. This study extends the effects of modified separators to the Li anodes, opening up new opportunities for future developments of Li-S batteries.

S8: 17th International Symposium on Advanced Processing and Manufacturing Technologies for Structural and Multifunctional Materials and Systems (APMT17)

Polymer-based Processing

Room: Coquina Salon F (North Tower)
Session Chair: Sandrine Cottrino, MATEIS Laboratory

1:30 PM

(ICACC-S8-027-2023) Silicone-aided Advanced Additive Manufacturing of Glass-ceramic Scaffolds (Invited)

H. Elsayed[1]; F. M. Stabile[3]; J. Kraxner[2]; D. Galusek[2]; E. Bernardo*[1]

1. University of Padova, Department of Industrial Engineering, Italy
2. Alexander Dubcek University of Trencin, FunGlass, Slovakia
3. CETMIC. Centro de Tecnología de Recursos Minerales y Cerámica, Argentina

Silicone resins are attractive both as precursors of silicate bioceramics and as feedstock for additive manufacturing technologies, including masked stereolithography. The two aspects may be successfully combined operating with simple silicone-based blends, consisting of a silicone polymer mixed with photocurable acrylates. A first case study concerns wollastonite-diopside scaffolds, resulting from the inclusion of glass powders in the blend. Upon firing at 900 °C, the viscous flow of softened glass powders is mitigated by the presence of a rigid silica skeleton provided by the decomposition of silicone, so that the topology of highly porous printed scaffolds is not degraded. The final phase assemblage is defined by the glass-silica interaction. Operating just with carbonate and phosphate fillers, in a second case study, the silicone component directly yields a ceramic product (at 1100 °C) with a composition resembling well-established Biosilicate® glass-ceramics. Simple adjustments on the silicone/filler ratio allow for the obtainment of Biosilicate® glass-ceramic matrix composites, when firing in nitrogen instead of air. In this case silicone transforms into silica (reacting with oxide fillers) and pyrolytic carbon. The latter is intended to provide extra functionalities, such as enhanced absorption of IR light, useful for disinfection purposes.

2:00 PM

(ICACC-S8-028-2023) Coarse-Grained Molecular Dynamics Simulations on Wear Mechanism of Concentrated Polymer Brush Grafted on Ceramics Substrate

A. Chiba*[2]; R. Kudo[2]; M. Yokoi[2]; M. Kawaura[2]; Q. Chen[1]; Y. Asano[2]; Y. Ootani[2]; N. Ozawa[1]; M. Kubo[2]

1. Tohoku University, New Industry Creation Hatchery Center, Japan
2. Institute for Materials Research, Tohoku University, Japan

Polymer materials are lightweight and have high specific strength, chemical stability, and design flexibility. Concentrated Polymer Brush (CPB) exhibits large swelling film thickness, high compressive resistance, exclusion volume effect, and low coefficient of friction between swollen brushes. Therefore, the CPB is promising as new low-friction material and is used for modification of ceramic substrate surfaces. However, the tribological properties deteriorate due to the degradation of the CPB by wear. Since the wear occurs due to the bond breaking of the polymer chain, it is crucial to understand its mechanism based on the atomic scale dynamics. In this study, we analyzed the frictional characteristics of a CPB using coarse-grained molecular dynamics simulations. The CPB is composed of linear polymer chains. A rigid body was pressed and slid against the CPB surface to simulate the friction dynamics. The simulations show that the bond breaking occurs uniformly from root to tip of the polymer chain. Some of the broken polymer chains were expelled from the polymer brush. These polymers entangled on the brush surface and emerged into the solvent. The number of broken polymers increased with increasing polymer chain length. These results presumably involve the effect of entanglement. The obtained insights enable us to design the CPB with high wear resistance.

2:20 PM

(ICACC-S8-029-2023) Investigation of Lamination Approaches for SiC-Filled Thermoplastic Polymer Blends

O. Brandt*[1]; R. Orta Guerra[1]; R. Trice[1]; J. P. Youngblood[1]

1. Purdue University, School of Materials Engineering, USA

Ceramic co-extrusion is a ceramic forming technique where a feedrod of a ceramic/polymer mixture is heated and extruded through a reduced cross-section, reducing the size but retaining the symmetry of the original feedrod. The co-extrusion process often involves a rebundling of ceramic/polymer extrudates or a "lamination" step via warm pressing to form the final component. Lamination is critical to the overall strength as any delaminations between adjacent exudates will manifest as cracks in the final sintered body. The aim of this presentation is to describe the mechanisms that cause delamination between warm-pressed ceramic-filled thermoplastic extrudates and to present approaches to mitigate this delamination. The approaches studied varied the thermoplastic blend compositions and the binder burnout process. Mechanical tests and crack investigations were used to gain insight those variables that most contributed to successful lamination, and ultimately, crack-free sintered bodies.

Rapid Prototyping, 3D Printing, Patterning, Templates and Self-assembly

Room: Coquina Salon F (North Tower)
Session Chair: Gang Shao, Zhengzhou University

3:00 PM

(ICACC-S8-030-2023) Tailoring of ceramic powder for various additive manufacturing techniques (Invited)

A. L. Leriche*[1]; D. Grossin[2]

1. Université Polytechnique Hauts-de-France, France, France
2. CIRIMAT ENSIACET, France

Various additive manufacturing techniques are increasingly applied to the manufacturing of ceramic parts for medical or aeronautics applications. The main advantage of these techniques is the possibility of manufacturing complex shape parts with a gradient of properties. However, some issues remain to achieve mechanical properties equivalent to conventional ceramic shaping methods. This talk will highlight the main achievement of the European project called Development Of Ceramics 3D-Printing, Additive Manufacturing* (DOC-3D-Printing) in terms of powder tailoring to increase the density and the mechanical properties of printed parts. For selective laser sintering, Y-ZrO_2 and SiC powders and laser and powder bed parameters were optimized to achieve 92-97% of relative density. For robocasting, β-TCP powder, the pastes to be extruded and the drying process were set up to achieve > 92% of density and 100 MPa for compression strength. For binder jetting, an increase in the green density of alumina compact up to 35% was obtained by adjusting the size and shape of the particles. After infiltration with a slurry, green density was increased up to 55% to obtain 88% density after sintering. * This project was funded from European Union's Horizon 2020 research and innovation programme Marie Sklodowska-Curie grant agreement No 764935

3:30 PM

(ICACC-S8-031-2023) Dielectric composite materials for passive RF components manufactured by 3D printing (Invited)

J. Heintz*[1]; N. Penin[3]; C. Elissalde[2]; J. Silvain[2]; T. Fournier[4]; T. Hoang[5]; B. Loiseaux[5]; P. Pouliguen[6]

1. ENSCBP-Bordeaux INP, ICMCB, France
2. ICMCB-CNRS, France
3. ICMCB-University of Bordeaux, France
4. Plateforme Technologique CANOE, France
5. Thales Research & Technology, France
6. DGA/AID, France

The development of agile, ultra-flat and low-cost antennas operating in the microwave band is a major challenge to enable accessibility of mobile internet, especially on land and air transportation. One of the key issues is the compactness of such antenna systems, which can be achieved thanks to a sub-wavelength structuration of the material, obtained by an additive manufacturing technique. We will present new results regarding the study and development of a new polymer/titania composite featuring high values of permittivity and low losses in the microwave domain. Before carrying out 3D printing of a functional RF component by the Fused Deposition Modeling method (FDM), a significant work has been done to select a low-loss dielectric oxide powder and a low-loss polymer matrix. The preparation of the ceramic powder will be described as well as the optimization of the ceramic charge within the polymer filaments. Printable composite filaments were obtained containing up to 35 vol.% of dielectric particles and presenting high dielectric permittivity ($\varepsilon_r \sim 8$) without increasing dielectric losses (tg $\delta = 1.10^{-3}$). We will conclude by presenting an example of a demonstrator that has been successfully printed and whose results both in terms of electromagnetic behavior and reduction of the thickness and weight of the component have confirmed the interest of our approach.

4:10 PM

(ICACC-S8-032-2023) Process Principles of Additive Manufacturing to Fabricate 3D Structured Piezoceramics

S. Jang*[1]; S. Hossain[1]; H. Son[1]; S. Park[1]; C. Bae[1]

1. Korea Institute of Materials Science, Department of 3D printing materials, Republic of Korea

Additive manufacturing has opened a new way of producing novel structures of piezoceramics via design freeform in the field of sensors actuators, transducers, and energy harvesting. However, a lack of understanding about additive manufacturing processes such as resolution, printing defects, and thermal stress have restricted real usage. Herein, we demonstrated three process principles to overcome the above limitations: rheological design, printing dynamics, and thermal stress control. First, understanding the correlation between hydrogen bonding and structural effect and following mapping of printable rheology provide high resolution and shape retention. Second, we investigated printing dynamics as a function of nozzle size, printing speed, and extrusion pressure and its relation to printing defects. Finally, a method for alleviating thermal stress through a mechanism study and a quantitative approach was suggested. The manufactured piezoelectric ceramic was applied to the accelerometer and showed piezoelectric performance comparable to the conventional.

4:30 PM

(ICACC-S8-033-2023) Quantifying the effect of reactive binders when sintering 3D printed ceramic aggregates

L. O. Grant*[2]; R. Maier[2]; C. Higgs[1]; Z. Cordero[3]

1. Rice University, Materials Science and NanoEngineering, USA
2. National Institute of Standards and Technology, USA
3. Massachusetts Institute of Technology, Department of Aeronautics and Astronautics, USA

Many ceramic 3D printing technologies (e.g., binder jet 3D printing and direct ink writing) consist of mixtures of powder and organic additives which require post processing to achieve high relative densities and desirable mechanical properties. However, the high temperatures often required for densification can lead to creep and distortion of the printed parts. This challenge has motivated the use of reactive binders which decompose to form solid precipitates that reinforce interparticle contacts at temperatures below the temperature range for sintering. In this work we evaluate the effect of a reactive binder on the sintering mechanics in a model system: binder jet 3D printed TiO_2 treated with a TiO_2-forming reactive binder, aqueous titanium bis-ammonium lactato dihydroxide (TALH). Dilatometry measurements reveal that creep is slower in TALH-treated materials than in the as-printed TiO_2. Models of structural evolution in sintering powder compacts reveal that the dominant effect of TALH on the creep response is to increase the interparticle contact size, while the coordination number remains essentially unchanged because of the low initial relative density. Insights from this work are used to compare different classes of reactive binders, and we develop generalized guidelines for the design of reactive binders for creep mitigation.

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

Forming and Shaping Processes for Advanced Materials

Room: Ballroom 1-2 (South Tower)
Session Chair: Kyoung Il Moon, KITECH

1:50 PM

(ICACC-S11-007-2023) Application of Data Science Methods on the Machining of Ceramic Matrix Composites (Invited)

R. Goller*[1]; P. León-Pérez[1]; J. Macken[1]; A. Rösiger[1]

1. University of Applied Sciences, Mechanical Engineering, Germany

Machining of ceramicmatrix composites can be very challenging. Anisotropic, multi material properties require the developent of adapted machining strategies. Any damage or failure during the process cause expensive scrap especially in small series applications like satellite mirror structures or aircraft turbine components. Future high volume and high value parts e.g. turbine blades for eco fuel or hydrogen powered engines require proven conceps for quality assurance already during the production process. Further predicted maintenance has to be developed and established, to avoid the unforeseen failure of tools. In the presented work an experimental setup of the data aquisition and analysis during the machining process of CMC is presented. Corellations betwen process parameters and material properties of the mechanical machining process of CMC are demonstrated. The potential of smart data analysis in the inline quality control and predicted maintenance is given in the outlook.

2:20 PM

(ICACC-S11-008-2023) Development of a composite pressure sensor in which the change in contact area to the electrode interface dominates the piezoresistive behavior

T. Katagiri*[1]; K. Kawahara[2]; K. Niihara[1]; K. Umemoto[1]; T. Nakayama[1]; T. Miyoshi[1]

1. Nagaoka University of Technology, Japan
2. Inaba rubber Co.Ltd., Japan

Piezoresistive pressure sensors are sensors that respond to mechanical force or deflection with a change in resistance, are easy to manufacture and collect signals, and can be used at low voltages. In composites made from polymer materials, the composite exhibits high resistance when there is no load, even though most of the conductive fillers inside the composite are in contact with each other. When the external force applied to the composite increases, the resistance decreases as the contact area to the electrode increases, using the flexibility of the polymer material. Piezoresistive pressure sensors were fabricated with the property that the resistance decreases as the contact area to the electrode increases, taking advantage of the flexibility of the polymeric material.

Starting Materials: Mining, Particles, Bulk, and Functional Materials and Precursors

Room: Ballroom 1-2 (South Tower)
Session Chair: Chisung Ahn, Korea Institute of Industrial Technology

3:20 PM

(ICACC-S11-009-2023) Observation of alloying behavior Al-Cr-Fe-Ti alloy powder by changing milling energy

A. Ahn[1]; H. Park[1]; B. Choi[1]; H. Yoon[1]; K. Moon*[1]

1. KITECH, Republic of Korea

Nitride coatings deposited using Al-Cr alloys with high aluminum content are widely used to improve tool durability and wear resistance, and many studies are being conducted to design by adding 3rd and 4th alloys to improve properties. In this study, an Al-Cr-Fe-Ti quaternary alloy composition was designed and an alloy powder was prepared by mechanical alloying using planetary ball milling. The alloy powder was manufactured by changing the process time and process speed, and the milling energy according to the process parameters was calculated. The alloying behavior according to the milling energy of the prepared alloy powder was observed through SEM, XRD, DSC and PSA analysis. It was confirmed that the alloy powder was completely alloyed at a milling energy of about 3238.0×10^7 J/g and exhibited a steady state.

3:40 PM

(ICACC-S11-010-2023) Measurement of electrical waveform by applied nanosecond pulsed electric field to the sintered body

H. Saito*[1]; K. Yoshida[1]; K. Nagao[2]; K. Niihara[1]; T. Nakayama[1]

1. Nagaoka University of Technology, Extreme Energy-Density Research Institute, Japan
2. National Institute of Technology, Oyama College, Japan

The purpose of this research is to develop a method to judge the sintered state from the electrical resistance measurement by applying a nanosecond pulse voltage to the sintered body. The internal electrical resistance changes as the sintering of the particles progresses. In the measurement of ionic conductivity, it was possible to measure grain resistance and grain boundary resistance by an alternating current impedance measurement method using alternating current. In this presentation, we devised a method for judging the internal state of sintering by measuring the electrical resistance of the sintered body using a pulse voltage with a nanosecond width for the AC voltage.

4:00 PM

(ICACC-S11-011-2023) Enhancing the power generation of lead-free pyroelectric Ba(Zr Ti)O_3 with strontium (Sr) doping

N. Ngo*[2]; H. Sugiyama[1]; B. Amila[2]; J. Wiff[1]; T. Suzuki[2]; H. Suematsu[3]; T. Nakayama[2]

1. Nagaoka University of Technology, Department of Science of Technology Innovation, Japan
2. Nagaoka University of Technology, Japan
3. Nagaoka University of Technology, Extreme Energy-Density Research Institute, Japan

Waste heat recovery utilizing the pyroelectricity in ferroelectric materials gathers the attention of researchers due to the ability to convert unused heat to electricity based on its fluctuating behavior. Furthermore, with the spreading of 17 Sustainable Development Goals (SGDs), replacing conventional lead-containing materials and developing a new composition for the high-potential low-grade waste heat became essential. In this research, we report the application of lead-free Ba(Zr, Ti)O_3 with the novel electro-thermodynamic Kim's cycle for efficient heat-to-power generation and enhancing the ability by using alkaline earth metal strontium (Sr) doping. A 2.5 mol% Sr doping successfully improved the energy density by 1.4 times from 10.6 to 14.7 mJ/cm^3, and for the 5 mol% sample, the

generated energy achieved 16.7mJ/cm^3, increased by 58% compared to the un-doped (Under 200 kV/m electric field, and fluctuating heat source of 40-100 °C). Sr-doping successfully upgraded the low-temperature power generation ability of lead-free Ba(Zr, Ti)O_3, raising its potential for replacing the conventional lead-based ones in waste heat recovery applications.

4:20 PM

(ICACC-S11-012-2023) Observed changes in the fluoride doped properties of high temperature thermoelectric material-Yttrium Aluminoboride

W. Mita*[1]; K. Nishida[1]; H. Son[2]; M. Takeda[1]; T. Mori[2]; K. Niihara[1]; T. Nakayama[1]

1. Nagaoka University of Technology, Japan
2. National Institute for Materials Science (NIMS), Japan

This work describes the observation of changes in the properties of $Y_{0.62}Al_{0.71}B_{14}$, a high-temperature thermoelectric material, due to fluoride doping. Fluoride materials are explored for their suitability as sintering aids, and a comparative study is carried out to identify those that are useful as sintering aids. As additives, $Y_{0.62}Al_{0.71}B_{14}$ samples are prepared by adding 10 wt.% of AlF_3, YF_3, MgF_2 and CaF_2 to the sintered material, which have been found to be useful in previous studies. Sintering was carried out using discharge plasma sintering (SPS) at 1500 °C for 30 min under 30 MPa and in an Ar atmosphere. After sintering, the sample obtained a bulk with a diameter of 10 mm and a thickness of 2 mm. Subsequently, the bulk was measured and evaluated by X-ray diffraction (XRD), Thermal diffusivity / Thermal conductivity measurement (LF), Seebeck Coefficient / Electric Resistance Measurement System (ZEM) and Scanning Electron Microscopy (SEM). XRD showed the same crystalline state as AlF_3 in MgF_2 and YF_3. The electrical conductivity showed the same behavior for MgF_2 as for AlF_3, which showed n-type behavior. In contrast, YF_3 showed p-type behavior. These results show that the same target material can be obtained by adding fluorides other than AlF_3, and it was also discovered that the properties change.

S12: On the Design of Nanolaminated Ternary Transition Metal Carbides/Nitrides (MAX Phases) and Borides (MAB Phases), Solid Solutions Thereof, and 2D Counterparts (MXenes, MBenes)

Methods for Improving Damage Tolerance and Performance I

Room: Ballroom 3 (South Tower)
Session Chairs: Miladin Radovic, Texas A&M University; Michael Naguib, Tulane University

1:30 PM

(ICACC-S12-001-2023) Crystallographic Slip, cracking and kinking in MAX phases (Invited)

A. Srivastava*[1]

1. Texas A&M University, USA

The aim is to better understand the single crystal level mechanical response of MAX phases and unravel the nature and origin of their unconventional damage-tolerance. To this end, we first characterize the mechanical response of single crystal micropillars of MAX phases. Our results show that depending on the crystallographic orientation, the micropillars of MAX phases either undergo non-Schmid crystallographic slip, slip followed by cleavage cracking or cracking without any appreciable amount of crystallographic slip. These micropillar experiments are also complemented with crystal plasticity finite element analysis using a novel constitutive model that accounts for the non-Schmid effects on the crystallographic slip. Next, using an in-house designed and build fixture we carried out in situ SEM mechanical testing of carefully grown single crystals of these materials. The results of this exercise show that even though MAX phases readily crack along the weakly bonded crystallographic planes, onset of an abstruse mode of deformation, referred to as kinking in these materials, induces large crystallographic rotations and plastic deformation that physically heal the cracks. These results provide a better understanding of why MAX phases, instead of undergoing catastrophic fracture, demonstrate unconventional damage tolerance.

2:00 PM

(ICACC-S12-002-2023) Mechanisms of Low Wear of PEEK/MoAlB in Ethanol vs. High Wear in Alkanes (Invited)

S. Berkebile*[1]; S. Ruggiero[2]; M. Ferrera[1]; C. Matzke[2]; K. Jacques[3]; S. Gupta[2]

1. US Devcom ARL, USA
2. University of North Dakota, Mechanical Engineering, USA
3. The University of North Texas, USA

Polyether ether ketone (PEEK) is a thermoplastic polymer that exhibits strong mechanical properties and chemical resistance up to high temperatures. Because of these properties, PEEK is widely used in applications such as bearing, pistons, pumps, biomedical implants, and dental components. The addition of materials such as MAB-Phase MoAlB ceramics into PEEK form a composite material that can enhance mechanical strength and the tribological properties in sliding contact friction over that of PEEK alone. In this research, PEEK and composites of PEEK matrix with 10% MoAlB were subjected to pin-on-flat sliding mechanical interfaces lubricated with ethanol, alkanes, and mixtures thereof. Coefficient of friction, wear rate, wear mode, and surface chemical changes were determined using optical profilometry, optical and electron microscopy, Raman and infrared spectroscopy, and energy dispersive spectroscopy. Ethanol lubrication was observed to result in negligible wear, while addition of any alkane increased wear with alkane fraction. The addition of MoAlB to PEEK resulted in lower wear than PEEK alone. The mechanism of reduced wear in ethanol was through a chemical transfer film to the counterbody. PEEK materials reinforced with MAlB may be used to improve the lifespan of mechanical components in ethanol environments.

2:30 PM

(ICACC-S12-003-2023) Deformation twinning in MAX phase single crystals: A complementary experimental analysis (Invited)

A. Joulain*[1]; S. Parent[1]; H. Bahsoun[1]; G. Renou[2]; C. Tromas[1]

1. Institut PPRIME, France
2. SIMAP, France

It is well known that at room temperature, plastic deformation in MAX phases is governed by dislocations gliding in the basal plane. However, as observed in many hexagonal metals, basal slip is not sufficient to account for arbitrary deformation. The crystal must find a way to reorient and to make basal slip more favorable. For MAX phases, one way consists in developing kink bands. However, in hexagonal metals, deformation twinning is also a common deformation process allowing for local reorientation. We present a study of elementary deformation mechanisms of single-crystalline Cr2AlC deformed at room temperature by spherical nanoindentation. The orientations are chosen to inhibit basal dislocation and to promote crystal reorientation. Localized and isolated deformation twins have been identified below the indents. The combination of Atomic Force Microscopy surface observation, Transmission Electron Microscopy (TEM) characterization and Automated Crystal Orientation Mapping has provided a full 3D characterization of these twins. The TEM analysis also revealed original cross slip dislocation configurations. These results have been confirmed by micropillar compression test performed in situ in a scanning electron microscope followed by EBSD analysis of the local misorientation generated through the deformation twins and by TEM analysis in thin foils extracted from the deformed micropillars.

Design of Novel Compositions and Manufacturing Methods I

Room: Ballroom 3 (South Tower)
Session Chairs: Babak Anasori, Drexel University; Konstantina Lambrinou, University of Huddersfield

3:20 PM

(ICACC-S12-004-2023) MAX phases: Synthesis and processing by Powder Injection Moulding and Additive Manufacturing (Invited)

S. A. Tsipas*[1]; A. Jimenez-Morales[1]; E. Tabares[1]

1. Universidad Carlos III de Madrid, Spain

This work studies the viability of non-conventional processing techniques such as Powder Injection Moulding (PIM) and Composite Extrusion Modelling (CEM) for the production of near-net-shape Ti_3SiC_2 and Cr_2AlC MAX phases. These two technologies start from pelletised feedstocks and allow the production of samples with a higher design freedom, reducing post-processing steps. The objective was to produce complex-shaped parts as well as increasing the application range of MAX phases, their reproducibility and production volume. Conventional powder metallurgy processing routes were also studied. The selection of the binder system as well as the optimisation of the solid loading of the feedstocks is necessary and, for this purpose, the rheological properties were characterised. Two multicomponent binders were selected: firstly, an environmentally-friendly binder consisting of a sustainable polymer and a biopolymer and secondly, a binder with the same sustainable polymer and polypropylene. Porous MAX phases with tailored porosity were obtained by PIM, avoiding the use of spacer holder. Debinding and sintering processes were optimized. It was possible to obtain good quality parts with custom geometries through PIM and CEM for both MAX phases, suitable for industrial applications with special requirements, such as catalytic devices, filters or as high temperature heat exchangers.

3:50 PM

(ICACC-S12-005-2023) Bulk and thin film Cr_2AlC: A debated oxidation resistant MAX phase (Invited)

C. Azina*[1]; T. Bartsch[1]; J. Gonzalez-Julian[2]; P. Eklund[3]; J. M. Schneider[1]

1. RWTH Aachen University, Materials Chemistry, Germany
2. Chair of Ceramics, RWTH Aachen University, Germany
3. Linkoping University, Dept. of Physics, Chemistry, and Biology, Sweden

Cr_2AlC is one of the most promising MAX phases for harsh environment applications, as it easily releases Al to produce a dense alumina scale during high-temperature oxidation. Unfortunately, the integrity of the MAX phase is often compromised by its local decomposition into Cr-carbides, below the newly formed alumina layer. This decomposition is often accompanied by the formation of pores and leads to the rapid failure of the MAX phase component. This presentation will deal with different strategies aimed to improve the oxidation resistance of bulk and thin film Cr_2AlC. Microstructural design was optimized for bulk materials by exploring different MAX phase precursors, including MAX phase powders, and elemental and compound powders. We will also discuss the oxidation resistance of Cr_2AlC coatings and strategies employed to improve oxidation at high temperatures in air. Strategies involve doping of the Cr_2AlC MAX phase with so-called reactive elements and continuous Al supply from Al-containing MAX phases. We will also have a better look at the diffusion phenomena which occur in the MAX phase in order to suggest improved design of Cr_2AlC for harsh environments.

4:20 PM

(ICACC-S12-006-2023) Synthesis of Solid-Solution MXenes with Tunable Electronic, Optical, and Electrochemical Properties (Invited)

C. E. Shuck*[1]; Y. Gogotsi[1]

1. Drexel University, Materials Science and Engineering, USA

MXenes are potentially the largest class of 2D materials discovered so far. With a general formula of $M_{n+1}X_nT_x$, M is an early transition metal (Ti, V, Nb, Ta, etc.), X is C and/or N, T_x represents the surface groups (-O, -OH, -F, -Cl), and n = 1–4, over 30 stoichiometric phases have already been discovered. This class of materials has been widely studied owing to their exceptional properties, including hydrophilicity, scalability, mechanical strength, thermal stability, redox capability, and ease of processing. Because MXenes inherit their structure from $M_{n+1}AX_n$ (MAX) phase precursors, understanding MAX phase synthesis leads to control over flake size, defect density, and chemical composition of the resultant MXene. One understudied, yet important class of MXenes are solid-solution MXenes, where multiple elements are randomly distributed within the M layers. Herein, three interrelated solid solution systems, $(Ti_{2-y}V_y)CT_x$, $(Ti_{2-y}Nb_y)CT_x$, $(Nb_{2-y}V_y)CT_x$ are used as models to study the optical, electronic, and electrochemical properties. These MXenes exhibit tunable properties that are directly related to their chemistry. By understanding this relationship, it then becomes possible to rationally design new MXenes targeting specific properties and applications.

4:50 PM

(ICACC-S12-007-2023) Synthesis and Characterization of Novel Glass-MAX/MAB Composites

M. Dey*[1]; E. Sofowora[1]; E. Oloo[1]; S. Gupta[1]

1. University of North Dakota, Mechanical Engineering, USA

In this presentation, we report the synthesis and characterization of glass-MAX/MAB composites. As a background, glass is a viable material for different types of applications but it has different limitations like brittleness and poor thermal schock resistance. Comparatively, MAX and MAB phases are bestowed with interesting properties like damage tolerance, tailorable hardness, and triboactive behavior, among others. The goal of this work is to synthesis and characterization of composites of glass with MAX or MAB phases as reinforcements. Detailed microstructural characterization of these composites will be presented by SEM analysis. In addition, mechanical behavior and thermal shock behavior of these composites will be presented.

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

Radiation Damage, Defect Production, Evolutions, and Interactions

Room: Ballroom 4 (South Tower)
Session Chair: Tatsuya Hinoki, Kyoto University

1:30 PM

(ICACC-S13-032-2023) Spatial distribution of radiation damage in ceramics and composites (Invited)

P. A. Burr*[1]; M. I. Brand[1]

1. The University of New South Wales, Australia

Calculations of radiation damage typically rely on the theory of Norgett, Robinson and Torrens (NRT), however this assume that materials are homogeneous. For many engineering materials, especially composites, this assumption is poor. Knowing where in a material radiation damage is being produced more or less is a missing key piece of information that will aid the development of radiation tolerant materials. I will show how we have extended the NRT formalism to account for microstructural variations in materials, by including the damage caused in a phase by primary knock-on atoms that are produced in another nearby phase. The new approach converges to conventional NRT at suitably large length-scales, and agrees with binary collision approximation simulations for bulk single-phase materials. We apply the method to tungsten borides used in fusion applications, and cer-mets composites. We discover that spatial localisation of primary radiation damage can lead to damage localisation of up to 30% within a phase, and order of magnitude differences across separate phases, and that both effects can be controlled by tailoring the microstructure. In some materials it is also possible to control the total degree of damage production in a material through microstructure engineering.

2:00 PM

(ICACC-S13-033-2023) Hydrothermal Corrosion behavior of Neutron Irradiated Additively Manufactured SiC fibers

A. Seshadri*[1]; J. Pegna[2]; K. L. Williams[2]; S. Harrison[2]; K. Shirvan[3]

1. Arunkumar Seshadri, Arunkumar Seshadri, USA
2. Free Form Fibers, USA
3. Massachusetts Institute of Technology, USA

SiC/SiC composites for advanced fuel cladding concepts has been re-evaluated in light of recent developments in advanced manufacturing. Here, we describe the results of our examination into the hydrothermal corrosion and dimensional change of fibers produced by Free Form Fiber LLC with Rapid Laser Chemical Vapor Deposition (R-LCVD), exposed to neutron and gamma radiation. In contrast to radiation-induced shrinkage in commercial Hi-Nicalon Type S (HNS) fibers, the R-LCVD fibers exposed to MITR neutron irradiation exhibited stochiometric dependent swelling. Prior to and after neutron irradiation, the hydrothermal corrosion behavior of the stoichiometric R-LCVD fibers was comparable to HNS fibers. However, compared to HNS and stoichiometric fibers, the dissolution rate of non-stoichiometric unirradiated fibers containing excess silicon was significantly higher. Contrary to logic, the corrosion rate of these excess Si fibers decreased considerably after neutron radiation. The underlying mechanism behind this improvement from neutron radiation was investigated by performing post-irradiation microscopy of R-LCVD fibers. We also discussed the possibility of using high temperature thermal pretreatment to reduce thermal corrosion of R-LCVD fibers. The preliminary neutron irradiation performance of the R-LCVD-fiber imbedded composites is also reported.

2:20 PM

(ICACC-S13-034-2023) Development and qualifcation of ATF cladding materials - the H2020 ILTROVATORE experience

K. Lambrinou*[1]

1. University of Huddersfield, School of Computing and Engineering, United Kingdom

The 2011 Fukushima Daiichi event demonstrated the need for improved nuclear energy safety, thus justifying the global investments in the development of accident-tolerant fuel (ATF) cladding materials that are called to outperform commercial fuel zircaloy claddings both during nominal operation conditions and high-temperature transients/accidents. The main objective of the large-scale H2020 IL TROVATORE project (30 beneficiaries from Europe, the USA, and Japan) is the performance optimization of select ATF cladding material concepts (i.e., SiC/SiC composites, coated & surface-modified zircaloys, and ODS-FeCrAl alloys), followed by their irradiation in the BR2 test reactor in PWR-like water. This lecture describes the accelerated development of ATF cladding materials within H2020 IL TROVATORE, from the design of innovative material concepts (e.g., determination of diffusion barriers in coated zircaloy systems) to the down-selection of candidate material constituents (e.g., substrate-specific coatings) to the key performance indicators used to determine the candidate ATF cladding materials to be irradiated in the BR2 reactor. This lecture also presents preliminary results of the post-irradiation examination of the ATF claddings (i.e., uncoated & Cr-coated Optimized ZIRLO^{TM}; uncoated/coated SiC/SiC composites) irradiated in the 1st cycle of the BR2 irradiation campaign of H2020 IL TROVATORE.

2:40 PM

(ICACC-S13-035-2023) Analysis of Silicon Carbide Cladding with Fuel Performance Code BISON

G. Singh*[1]; K. Gamble[1]; A. Recuero[1]; J. Hales[1]; S. Novascone[1]

1. Idaho National Lab, USA

Silicon carbide (SiC) fiber-reinforced SiC matrix composites cladding is an accident tolerant fuel cladding concept that has potential to deliver superior performance and enhanced safety for light water reactors. BISON is a fuel performance code developed at multiple national laboratories of USA. During the last several years, BISON's capability to analyze accident tolerant fuel has expanded significantly. Several new models and physics have been added to address the need to analyze these advanced fuel concepts. The presentation will focus on the overview of BISON'S existing capability and ongoing development regarding SiC component analysis for light water reactor applications. Results from validation cases will be discussed for demonstration purpose.

Material Technologies for Enhanced Accident Tolerance LWR Fuels and Core III

Room: Ballroom 4 (South Tower)
Session Chair: Arunkumar Seshadri, Arunkumar Seshadri

3:20 PM

(ICACC-S13-036-2023) Joining of SiC-SiC Composites by a Novel Embedded-Wire CVD Technique

M. C. Schaefer*[1]

1. Free Form Fibers, USA

Embedded-Wire CVD (EWCVD) was developed by FFF as an innovative approach to produce high purity, high density CMC structures. This technique builds upon our previous work with Laser-CVD (LCVD), utilizing dimensionality of mass-transport and its large influence on CVD/CVI. An important application of EWCVD is in the long-sought goal of accomplishing joining (welding) of ceramic and ceramic-based composite components together. FFF has demonstrated homogeneous joining of SiC fiber-reinforced SiC matrix composites without the use of extraneous materials that limit the performance of the joint structure. EWCVD has also been implemented in the development of SiC thin wall composite shells for the replacement of metal-based cladding to contain uranium oxide fuel pellets in nuclear power applications. SiC is currently a candidate for nuclear fuel cladding as either a monolith, a SiC fiber-reinforced SiC matrix (SiC/SiC) composite, or a hybrid of metal and SiC/SiC composite, utilizing our LCVD SiC fibers. EWCVD is also being deployed to address this issue and form a fully dense, hermetic end cap seal. Further extension of the EWCVD technology to other thin shell applications in the aerospace field are also being explored.

3:40 PM

(ICACC-S13-037-2023) Formation of Rare Earth Silicate on Silicon Carbide Utilizing Oxidation

T. Hinoki*[1]; H. Sakai[1]; J. Lee[1]

1. Kyoto University, Japan

Silicon carbide (SiC) composites are expected to be stable in severe environment including high temperature steam for both nuclear application and aerospace application. Rare earth (RE) silicates are candidate environmental barrier coatings in high temperature steam oxidation environment. SiC materials were fabricated with RE-oxides by liquid phase sintering or reaction sintering. SiO2 was formed on the SiC by oxidation in air or steam at high temperature. The SiO2 and the RE-oxides close to the surface reacted and the RE-silicates were formed on SiC. The dense and thick RE-silicates were also formed by oxidation with RE-oxide powders.

4:00 PM

(ICACC-S13-038-2023) Mechanical properties of SiC composites neutron irradiated at ~300°C to 30dpa

T. Koyanagi*[1]; Y. Katoh[1]

1. Oak Ridge National Laboratory, USA

For economical use of SiC fiber-reinforced SiC matrix composites in light water reactor (LWR) core and fuel assembly components, their service life extended to multiple fuel cycles is desired. Owing to the limited number of irradiation studies previously conducted at LWR-coolant temperatures, this study examined SiC composites following neutron irradiation at ~300°C to 30 dpa in the High Flux Isotope Reactor. The investigated materials were chemical vapor infiltrated SiC composites reinforced with monolayer pyrolytic carbon coated Hi-Nicalon™ Type-S and Tyranno™ SA3 SiC fibers. There was no obvious change in the proportional limit stress and ultimate flexural strength of both composites from irradiation. The irradiated composites retained their quasi-ductile fracture behavior. Electron microscopy observations found micro pore evolutions in the irradiated carbon interphases, but the interphases still retained the bonding between the fiber and matrix. The results of this study indicate the feasibility of extended service of the present generation nuclear grade SiC composites to 30 dpa, corresponding to an exposure level during several fuel cycles of today's LWR. This study was supported by US DOE Office of Nuclear Energy for the Advanced Fuels Campaign.

S15: 7th International Symposium on Additive Manufacturing and 3-D Printing Technologies

Material Extrusion / Fused Deposition Modeling

Room: Coquina Salon H (North Tower)
Session Chair: Mark Du, Argonne National Lab

1:30 PM

(ICACC-S15-026-2023) Fused Deposition Modelling of Fibre Reinforced Ceramic Matrix Composites

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1. University of Birmingham, School of Metallurgy and Materials, United Kingdom
2. University of Birmingham, Ceramic Science & Engineering, United Kingdom

Ceramic matrix composites, CMCs, are finding many uses in the challenging environments that are becoming increasingly common in modern society. Fibre-reinforced CMCs have a higher toughness compared to the monolithic ceramics and can also be tailored to offer a wide range of other properties too, e.g., carbon fibre-reinforced ultra-high temperature CMCs can be used in extreme environments where temperatures can exceed 2500°C. This work focuses on using additive manufacturing for the fabrication of carbon fibre reinforced silicon carbide. The ultimate goal is to achieve the simultaneous fabrication of both the reinforcement and the matrix. The approach being investigated involves the accurate controlled use of fused deposition modelling (FDM), which it is hoped will enable flexibility in the structural and material design of fibre-reinforced CMCs, whilst being capable of producing parts of complex shape and good surface finish, potentially without a need for final machining. The influence of filament composition, surface treatment, topological optimisation of structural design and FDM parameters are currently being investigated. The research aims to improve the thermomechanical properties of fibre-reinforced CMCs via multiple approaches, reducing the demand for subsequent post-processing for densification.

1:50 PM

(ICACC-S15-027-2023) EVA-PVA Binder for Fused Deposition Modeling of Large Polymer Derived Ceramics

F. Sarraf*[1]; S. Churakov[2]; F. Clemens[1]

1. Empa, Swiss Federal Laboratories for Materials Science and Technology, Switzerland
2. University of Bern, Switzerland

Fabrication of polymer-derived ceramics using material extrusion based additive manufacturing (MEX-AM), also known as fused deposition modeling (FDM), is attractive due to thermoplastic behavior of preceramic polymers (PCPs). The released gaseous byproducts from pyrolysis of Si-based PCPs leave a glassy SiO_2 residue. This phenomenon triggers the formation of porosity and cracks. As a result, only thin structures or foams have been produced. Investigated ceramic composition in this study is a thermoplastic PCP and alumina powder to achieve 3:2 mullite. Addition of MgO additive resulted in dense ceramics at 1600°C for 5h. A mixture of Ethylene vinyl alcohol (EVA) and polyvinyl alcohol (PVA) was used to obtain suitable rheological properties for 3D printing. A 2-step debinding including solvent extraction and wick debinding was performed. Above 80% PVA removal is necessary to provide interconnected channels for efficient gas removal during crosslinking; therefore, closed spherical pore structure could be avoided in the sintered structures. Mixing EVA with a crosslinking agent enabled debinding and sintering of large printed structures without shape distortion. Furthermore, flexural strength of the wick debound and sintered extruded filaments and printed specimens was measured using four-point bending setup to determine the effect of thermal treatment and printing defects on flextural strength.

2:10 PM

(ICACC-S15-028-2023) Evaluating Extrusion Deposited Additively Manufactured Fiber-Reinforced Thermoplastic Polymers as Carbon/Carbon Preforms

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1. Composite Manufacturing and Simulation Center, USA
2. Purdue University, Department of Materials Engineering, USA

Extrusion deposition additive manufacturing of fiber-reinforced thermoplastic polymers allows embedded fibers to be oriented locally and globally without the use of molds, enabling more control over fiber architectures, the matrix thermal history, and its microstructure. Carbon/carbon composite processing can benefit from extrusion deposition additive manufacturing but thermoplastic conversion to carbon results were needed. In this study, we analyzed several short carbon fiber-reinforced thermoplastic polymers with various fiber loadings, including polyphenylene sulfide, polyetherimide, poly sulfone, polyether ether ketone, and polyether sulfone via thermogravimetric analysis and coupon distortion tests to compare carbon yields and dimensional stabilities during pyrolysis. The results showed that while polyetherimide had the highest carbon yield, the short carbon fiber-reinforced polyphenylene sulfide was the best preform for manufacturing carbon/carbon composites because of its dimensional stability. The carbonization results for an additively manufactured fiber-reinforced polyphenylene sulfide nozzle showed that slow heating rates could be used to perform shape- and size-preserving initial pyrolysis processing of more complex geometries.

2:30 PM

(ICACC-S15-029-2023) Additive manufacturing and ultra-fast high-temperature sintering (UHS) of zirconia ceramics

S. Bhandari*[3]; M. Biesuz[2]; C. Manière[1]; G. Franchin[3]

1. Chargé de Recherche CNRS Laboratoire CRISMAT, France
2. University of Trento, Department of Industrial Engineering, Italy
3. University of Padova, Department of Industrial Engineering, Italy

In the past decade, ultrafast sintering techniques have gained popularity as a way to reduce the processing time and energy consumption of the ceramic industry. However, most of the research study has focused on the field-assisted sintering of pressed powder compacts with simple shapes. Additive manufacturing (AM) allows fabricating complex geometries with fewer constraints compared to the conventional fabrication techniques. Coupling AM with field-assisted sintering would be ideal to reduce production time from days to hours. In this work, 3D printed cylindrical structures of 3YSZ with gyroidal infill pattern were fabricated using the fused filament fabrication process (FFF). This research study examines the use of ultra-fast high-temperature sintering (UHS) to densify the complex 3D structure. A systematic study was done on the sintering behavior of the as-printed, chemically debinded and pre-sintered samples. Furthermore, current-controlled experiments with the help of FEM simulation were performed to study the densification behavior. With optimized parameters, the chemically debinded samples could be almost fully densified in few seconds. There were no visible cracks on the surface of the samples. The work provides a first proof of concept on utilizing the ultrafast heating rates to sinter such complex geometries without going through a separate thermal de-binding process.

Direct Writing / Ink Jet Printing Technologies

Room: Coquina Salon H (North Tower)
Session Chair: Fateme Sarraf, Empa, Swiss Federal Laboratories for Materials Science and Technology

3:20 PM

(ICACC-S15-030-2023) Polymer-assisted direct ink writing of carbon fiber reinforced silicon carbide composites.

L. L. Balderson*[1]; T. Sun[1]; E. Opila[1]

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

A strong motivation exists for development of carbon fiber (C_f) reinforced silicon carbide (SiC) composite in order to replace nickel superalloys in aerospace propulsion systems, explicitly in hypersonic applications where upper use temperatures exceed 2000 celsius. Despite the groundbreaking potential of SiC-C_f composites, conventional fabrication technologies inhibit design capability, thermodynamic performance, and mechanical longevity of parts that serve under extreme thermal conditions. We have developed a direct ink writing 3D printing technique which utilizes a sacrificial polymer and SiC-C_f blended ink that instantaneously solidifies under the shear stress of printing. A two-stage densification process was then applied to the green part to produce components with high density and complex geometries. The roles of sintering aids, including additives and residual carbon content from sacrificial polymer burnout, was evaluated in respect to densification efficiency through solid-state sintering. Additionally, the physics underlying the effect of secondary fiber phase on enhancing fracture toughness while mitigating catastrophic brittle failure observed in monolithic SiC components was explored.

3:40 PM

(ICACC-S15-031-2023) Additive manufacturing of CMCs by DIW of preceramic polymers

F. Da Rin Betta*[1]; B. Baker[2]; R. Seabright[2]; C. Footer[2]; G. Franchin[1]; P. Colombo[1]

1. University of Padova, Industrial Engineering, Italy
2. Qinetiq, United Kingdom

In this work, a silazane-based feedstock, containing also chopped fibers and fillers, was used to manufacture complex shapes by Direct Ink Writing (DIW). The samples were then converted into silicon carbonitride-based Ceramic Matrix Composites (CMCs) with a thermal treatment in an inert atmosphere. The composition of the slurry was optimized with a square-shaped Design of Experiment (DoE) to achieve a compromise between printability and the reduction of the formation of defects during the pyrolysis process. The samples produced were first observed with a Scanning Electron Microscope (SEM) to compare the formation of cracks and to select the best performing composition, which was then used to produce samples intended for further characterization.

4:00 PM

(ICACC-S15-032-2023) Opportunities and challenges of Inkjet Printing for the fabrication of Lithium-ion batteries

K. Sztymela*[1]; F. Rossignol[1]; M. Cerbelaud[1]; M. Bienia[1]

1. Institute of Research for Ceramics (IRCER), UMR CNRS 7315, France

Owing to high energy density, high operating voltage, long cycle life, lithium-ion batteries (LIBs) are the state-of-the-art energy storage technology for application in mobile appliances and electric vehicles. The market of LIBs is continuously expanding and in the short- to medium-term future, LIBs are expected to stay the preferred battery technology. Although LIBs are well-developed, it is anticipated that the further improvement in terms of their performance, safety and cost is possible. One of the most promising strategies for improving the electrochemical performance of next-generation LIBs is three-dimensional (3D) electrode structure. Inkjet printing (IJP), as an advantageous additive manufacturing method allows for the fast deposition of thin films or complex patterns with high precision. In this work, the possibilities and challenges of fabrication of 2D/3D composite electrodes using IJP technology are presented. The composite inks with various binders were formulated and rheological measurements were performed. The printing tests were carried out, revealing a problem of process instability. 2D/3D patterns were deposited and microstructural characterisation of fabricated structures was carried out.

Binder Jetting Processes

Room: Coquina Salon H (North Tower)
Session Chair: Giorgia Franchin, University of Padova

4:20 PM

(ICACC-S15-033-2023) 3D Printed Ceramics After ISS Spaceflight

A. B. Bailey*[1]; X. Wang[1]; D. Stohr[1]

1. Alfred University, School of Engineering, USA

This work reports evaluation results of 3D printed ceramics after ISS (International Space Station) flight. A homogeneous frit enhanced batch and a pure alumina glaze dipped batch of alumina-based ceramic were designed with compositions and microstructures that would ideally be resilient and effective in the foreseen testing conditions of the spaceflight environment. After the ISS testing was completed, the specimens were examined to confirm porosity and density properties and evaluate any microstructure modifications. These results set the stage for developing a second generation

of specimens and a spaceflight mission with the goal of providing specimens of appropriate geometry and large enough quantities for mechanical testing and further radiation induced conductivity data collection.

4:40 PM

(ICACC-S15-034-2023) Reaction-bonded Joining and Densification of Additively Manufactured Silicon Carbide by Liquid Silicon Infiltration

M. Du*[1]; J. Thomas[1]; B. Ma[1]; D. Singh[1]

1. Argonne National Laboratory, USA

Joining reaction-bonded silicon carbide is a cost-effective method for fabricating ceramic components, especially for those with relatively large sizes and complicated geometries. In this study, additive-manufactured silicon carbide preforms were joined and densified by reaction bonding via liquid silicon infiltration. The silicon carbide powder was firstly printed by binder jetting technology, debound to introduce carbon, and then infiltrated by liquid silicon. To further facilitate the joining, graphite or parchment papers were introduced into the joining layer. Results showed a robust joining layer with a range of thickness of 50–500 μm, depending on the paper type and number of paper layers. High-energy synchrotron X-ray revealed that β-phase silicon carbide was formed inside the bonding layer. Finally, two additively manufactured parts with complicated channel geometry were successfully joined.

S16: Geopolymers, Inorganic Polymers and Sustainable Construction Materials

Novel Applications of Geopolymers I

Room: Coquina Salon C (North Tower)
Session Chair: Henry Colorado L., Universidad de Antioquia

1:30 PM

(ICACC-S16-023-2023) Dielectric properties of chemosynthetic alumino-silicates and metakaolin-based geopolymers and composites (Invited)

M. B. Ogundiran*[1]; J. S. Dolado[2]; G. Goracci[2]

1. University of Ibadan, Department of Chemistry, Nigeria
2. CSIC-UPV/EHU, Material Physics Centre, Spain

Consequent on the need for alternative cheap and green sources of energy, studies are ongoing on the possibility of using geopolymer as dielectrics and conductors with improved electric energy storage and conversion ability. Chemosynthetic alumino-silicates, alkali, and phosphoric acid metakaolin-based geopolymers and composites are currently being developed and characterised for electrical and dielectric applications. The measured properties include electrical conductivity, dielectric constant, dielectric loss, impedance, and modulus. The origin of these properties in geopolymers are dipole moments of water molecules, ions in geopolymers and chemical composition of the geopolymer precursors. Geopolymer dielectrics are produced using sol-gel, alkali/silicate, and phosphoric acid activation of metakaolin and local calcined clays. Geopolymer composites are prepared by incorporating conducting or non-conducting fillers to geopolymer matrix. Dielectric properties are measured with broadband dielectric spectroscopy. Potential applications of these geopolymers include energy storage as in capacitors, power inverters of electric vehicles, and energy harvesting. Geopolymers with low dielectric loss and stable dielectric constant can function as electrical insulator for electronic packaging. My presentation will explore these issues and focus on our current research in this domain.

2:00 PM

(ICACC-S16-024-2023) Geopolymer Composites Exposed to Molten Chlorides and Carbonates (Invited)

P. F. Keane*[1]; N. Stanford[2]; F. Bruno[1]

1. University of South Australia, Future Industries Institute, Australia
2. University of South Australia, STEM, Australia

Molten salts are used in a variety of applications including material quenching, chemical extractions, thermal energy storage, etc. Chlorides and carbonates are abundant, are stable at high temperatures (>600 °C), and have superior thermal properties (Cp, λ, ρ). Expensive nickel alloys are typcially used to contain molten salts due to high thermal stabily and corrosion properties. In this study, potassium metakaolin-based geopolymer composites containing System 96(c) fusible glass frit powder and alumina platelets from MicroAbrasives(c) were heated, cooled, and then exposed to series of molten Group 1 chlorides and carbonates. Both macroscopic and microscopic analysis showed which salts did not react with the geopolymer composite. Sample sets for mechanical testing were then selected, exposed to molten salts in air, and mechanically evaulated at room temperature.

2:30 PM

(ICACC-S16-025-2023) Geopolymer composite formulations as flame retardant materials (Invited)

S. Rossignol*[1]

1. IRCER, France

In the field of housing or civil engineering, construction products can play an important role in the development of flame. Nowadays, more and more studies are focused on the studies for low-carbon materials without releasing volatile organic compounds. Among the new materials, environmentally friendly aluminosilicate-based geopolymers are promising candidates. The aim of this study is to select geopolymer materials for high temperature applications. Several formulations based on different metakaolin and mineral fillers with a potassium silicate solution have been evaluated. Different formulations were carried out from cm^3 to m^3. The temperature resistance was done at 1000 and 1300 °C. Measurements with thermocouples allowed to follow the thermal evolution of the material during the tests. Structural and mechanical analyses were performed before and after the temperature test. The results show that the selected formulations make it possible to reach a resistance to 1000°C with an unexposed face reaching only a temperature of 400°C. No mechanical loss was observed during the experiment. This is due to the presence of crystalline phases such as leucite or wollastonite which are formed in-situ with the temperature increasing. These materials are therefore promising as flame retardant and fireproof.

Novel Applications of Geopolymers II

Room: Coquina Salon C (North Tower)
Session Chair: Henry Colorado L., Universidad de Antioquia

3:20 PM

(ICACC-S16-026-2023) Crystallization of nano-kalsilite via hydrothermal decomposition of kaolin clay in potassium hydroxide solution

E. F. Yusslee*[2]; N. Dahon[1]; M. Abdul Rajak[1]; S. E. Arshad[2]

1. Universiti Malaysia Sabah, Preparatory Centre for Science & Technology, Malaysia
2. Universiti Malaysia Sabah, Faculty of Science and Natural Resources, Malaysia

Nano-kalsilite ($KAlSiO_4$) was synthesized hydrothermally in a Teflon-lined stainless steel autoclave reactor by using kaolin clay and potassium hydroxide (KOH) as precursors and heating it to 190°C for 24 hours. X-ray diffraction (XRD) on the crystal formed revealed the hexagonal space group $P6_3$ with lattice parameters a=5.22Å and c=8.52 Å. The structure is characterized by AlO_4 and SiO_4 tetrahedra building an organized three-dimensional framework composed of six-membered rings of tetrahedra pointing up (U) and down (D). The formation of hexagonal nanorod of kalsilite was validated by using scanning electron microscope (SEM) and transmission electron microscope (TEM) and in line with the data calculated using Scherrer's equation. Electron dispersion spectroscopy (EDS) data revealed a homogenous distribution of potassium atoms in the nano-kalsilite framework. In addition, zeolite W and illite were found to be the major phase when the reaction was carried out at a lower KOH concentration.

3:40 PM

(ICACC-S16-027-2023) Evaluation of the effect of temperature and pressure on geopolymers for oil wells applications

U. Siciliano*[1]; J. Zhao[2]; A. C. Trindade[3]; F. d. Silva[4]

1. Pontifícia Universidade Católica do Rio de Janeiro, Department of Civil and Environmental Engineering, Brazil
2. Technische Universität Dresden, Institut für Baustoffe, Germany
3. University of Illinois at Urbana-Champaign, Materials Science and Engineering, USA
4. Pontificia Universidade Católica do Rio de Janeiro (PUC-Rio), Civil Engineering, Brazil

Geopolymers are obtained by mixing a solid aluminosilicate precursor (such as metakaolin) and an alkaline solution (known as waterglass). Currently, one of the applications searching for alternative materials is the cementing of oil wells due to the increase in downhole pressures and temperatures, where Portland cement presents great degradation issues. Geopolymers emerge as a potential alternative to reduce well insulation problems due to their thermal stability. However, their behavior must be well understood under downhole conditions, i.e., under critical temperature and pressure, but so far little research has been carried out in this sense. For this reason, the objective of this work was to evaluate the compressive strength in plain potassium-based cubic geopolymer samples (2″ side) after being cured at temperatures and pressures of up to 200 °C and 70 MPa, respectively, comparing also the data found with microstructural characterizations, such as porosimetry (MIP) and thermogravimetric analysis (TGA). The results showed that the geopolymer had its strength improved by 144 % when cured at 50 °C, and further 37 % improvement when cured at 50 °C and 3000 psi.

4:00 PM

(ICACC-S16-028-2023) Stabilization of Cohesionless Soil using Metakaolin-based Geopolymer

O. D. Huang*[1]; J. Jang[2]; S. Congress[3]; A. Puppala[4]; M. Radovic[1]

1. Texas A&M University, Materials Science & Engineering, USA
2. Terracon Consultants, Inc., USA
3. North Dakota State University, Department of Civil, Construction and Environmental Engineering, USA
4. Texas A&M University, Zachry Department of Civil and Environmental Engineering, USA

Proper soil stabilization is a crucial part of pavement design to enhance the mechanical properties of the foundation. Recently, geopolymers (GPs) have been introduced as a green alternative to traditional calcium-based stabilizers such as ordinary Portland cement (OPC) and lime for stabilization. In this study, both OPC and metakaolin-based GP were used to assess the effectiveness of GPs as a stabilizer for natural coastal soil. The samples were cured at ambient temperature for different times then tested for its mechanical properties and durability such as compressive strength, resilient modulus, capillary soaking, and linear shrinkage. Then several selected samples were characterized using various techniques such as scanning electron microscopy (SEM), and energy dispersion spectroscopy (EDS) to help understand how GP works as a soil stabilizer. Results of this study suggests that GP can be used and green alternative to OPC for cohesionless soil stabilization that are dominant in the coastal regions.

4:20 PM

(ICACC-S16-039-2023) Machine learning and analysis of microstructural evolution of porosity in geopolymer composites

J. Gruber*[1]; W. M. Kriven[2]; P. F. Keane[3]

1. University of Illinois at Urbana-Champaign, Materials Science and Engineering, USA
2. University of Illinois at Urbana-Champaign, USA
3. University of South Australia, Future Industries Institute, Australia

Amorphous self-healing geopolymers with 50 wt% KGP 35 wt% glass and 15 wt% alumina platelets were synthesized and heat treated at various temperatures above and below crystallization temperature to induce various changes in the microstructure. The resulting microstructures were analyzed specifically for their porosity, specifically values such as percent porosity, pore concentration, and pore size. Using an original machine learning program a model was created to relate heat treat temperature and time to the various porosity statistics ultimately showing how a geopolymers porosity varies with heating profiles.

S17: Advanced Ceramic Materials and Processing for Photonics and Energy

Advanced and Nanostructured Materials for Photo-voltaics and Solar Fuels

Room: Coquina Salon G (North Tower)
Session Chair: Rafik Naccache, Concordia University

1:30 PM

(ICACC-S17-027-2023) Ceramic based transparent conductive oxides for thin film solar cells (Invited)

A. Romeo*[1]; E. Artegiani[1]

1. University of Verona, Computer Science, Italy

Crystalline silicon based solar cells have achieved a very high level of development, reaching efficiencies exceeding 26%. Also, the establishment of a large production chain has strongly reduced the production costs. However thin film materials with their high absorption coefficient allow for low temperature and in-line production processes: resulting in lower production costs in respect to silicon based devices. Among them, CdTe and $CuInGaSe_2$ are the most successful in terms of reliability and scalability. However a new generation of materials is under development: composed by earth abundant and non-toxic elements. In this work, we are going to discuss the application of ceramic-based thin film layers applied on CdTe, $Cu_2ZnSn(S,Se)_4$ and Sb_2Se_3 solar cells: from the engineering and crystallization of buffer layers to the application of transparent back contact for bifacial solar cells. In particular the following will be considered: 1- $Mg_xZn_{1-x}O$ or SnO_2 important for the $CdSe_xTe_{1-x}$/CdTe (substituting CdS/CdTe typical junction), 2- TiO_2 or SnO_2 for CdSe-CdS/Sb_2Se_3 and 3- ITO and ZnO for CdS/$Cu_2ZnSn(S,Se)_4$. The physical and electrical properties of these layers, crucial for obtaining not only high efficiency but also reliability and stability in the long term, will be discussed, their effect on the growth of the subsequent layers and on the final performance of the device will be presented.

2:00 PM

(ICACC-S17-028-2023) Understanding the propagation of photons and high-energy ions through low dimensional semiconductors (Invited)

O. K. Varghese*[1]; J. Napagoda[1]; D. Waligo[1]; M. Paulose[1]; D. Chen[1]; W. Chu[1]

1. University of Houston, Department of Physics, USA

Managing the propagation of solar photons is critical to achieving high efficiency in solar energy conversion devices such as solar photovoltaic cells and photoelectrochemical fuel generating cells. Each device consists of multiple layers of materials with metallic, insulating or semiconducting properties exhibiting distinct nature of interaction with photons. Nevertheless, these layers should be designed carefully to maximize photon absorption in the semiconducting absorber layer for efficient charge carrier generation. Our studies using numerical simulations showed that the light propagation through these devices could be managed precisely by employing layers consisting of low dimensional materials with appropriate geometric features. A different simulation study showed that such materials could also manage the propagation of high-energy ions through them. Implantation of high-energy ions is a widely used method for doping layers in electronic and photovoltaic devices; however, our simulation studies revealed the potential of low dimensional materials in guiding and focusing ion beams. This presentation provides the details of these efforts in revealing the interaction of light and high-energy ions with nanostructured semiconductors.

2:30 PM

(ICACC-S17-029-2023) Optimization of Colloidal Quantum Dots for Clean Energy Technologies (Invited)

G. Selopal*[1]; H. Zhao[2]; F. Vidal[3]; Z. M. Wang[4]; F. Rosei[3]

1. Dalhousie University, Department of Engineering/ Faculty of Agriculture, Canada
2. Qingdao University, College of physics, China
3. INRS-EMT, Canada
4. Institute of Fundamental and Frontier Sciences, University of Electronic Science and Technology of China, China

Colloidal quantum dots (QDs) are potential light harvesters for emerging clean energy conversion technologies [i.e., QDs sensitized solar cells (QDSCs), and photoelectrochemical (PEC) systems for hydrogen generation], due to their promising optoelectronic properties. However, the performance of QDs-based solar energy conversion devices is still lower than the expected theoretical value, which is mainly associated with the narrow light harvesting range, slow charge injection/transfer rate from QDs to scavengers and fast non-radiative carrier recombination within the QDs. To address these challenges, core/shell engineering in which the surface of the QD core is passivated with the shell layer of different thicknesses/materials has been shown to be an effective approach. Herein, we present the synthesis of core/shell QDs with variable shell thickness while keeping the core size and then the interfacial engineering between core and shell. We will discuss the effect of different shell thicknesses, compositions, and interface engineering of core/shell QDs to improve the optoelectronic properties in terms of broadening the absorption spectrum and efficient carrier dynamics compared to the control QDs. As a proof of concept, the resulting core/alloyed shell QDs applied as a light harvester in QDSCs and PEC systems, and possible future directions will be presented and discussed.

3:20 PM

(ICACC-S17-030-2023) Water Splitting Catalysis: Strategies for Performance Enhancement (Invited)

T. A. Shifa*[1]

1. Ca' Foscari University of Venice, Department of Molecular Science and Nanosystem, Italy

The generation of clean energy from water electrolysis is a feasible solution to overcome the problems of energy crisis. In this regard, 2D materials, and transition metal phosphides have got copious attention. Here, I present strategies to enhance the catalytic performance of these materials giving a particular emphasis for transition metal chalcogenides, transition metal phosphorus trichalcogenides (MPX_3; X=S,Se), and nickel phosphides. Experimental results show that doping and hybrid material formation play a significant role in optimizing the free energy of hydrogen adsorption and desorption on the vertically oriented nanosheets of WS_2. Another compelling issue in this research area is about solving the sluggish kinetics of the other half reaction (i.e OER) in water splitting catalysis. It has remained a bottleneck in realizing efficient performance. In this regard, nickel phosphide has an excellent track of performance. I also discuss the mechanism behind the very good performance of Ni5P4 and CrOx-CuS toward electro-catalysis of OER. The metal phosphides or sulfides are not the true catalysts, rather in-situ generated metal oxides at the vicinity of phosphides/sulfides are. Moreover, I highlight the emerging layered MPX_3 nanosheets as promising materials in sacrificial agent-free photocatalytic water splitting under simulated Sun light (AM 1.5G) illumination.

3:50 PM

(ICACC-S17-031-2023) Advanced TiO2 nanotubes for enhanced photocatalytic H_2 generation and CO_2 conversion (Invited)

N. Nguyen*[1]

1. Concordia University, Chemical and Materials Engineering, Canada

Among several approaches to solving the global warming due to greenhouse gases, photocatalysis has been widely studied owing to its low complexity and sustainability. In recent years, TiO_2 has received considerable attention due to its promising photocatalytic ability to produce H_2 from H_2O and convert CO_2 into other chemicals. However, bare TiO_2 is not effective without the presence of a co-catalyst. Not only the choice but also the sizes and distribution of the catalysts significantly affect the photocatalytic activity. Here we show the formation of non-conventional TiO_2 nanotubes such as highly ordered nanostumps and spaced nanotubes by the anodization method. These advanced nanostructures provide an excellent platform for the controlled deposition of co-catalysts (sputtering-dewetting and layer-by-layer). The final heterostructures catalysts show dramatical photocatalytic enhancement in H_2 production and CO_2 reduction.

4:20 PM

(ICACC-S17-032-2023) Nanostructured Optical Limiter Photonic Thin Films for Reusable High Irradiance Shielding of High Energy Lasers

D. E. Wolfe*[1]; C. DeSalle[1]; J. Reiss[1]; P. Albert[1]; R. Romesberg[1]; S. Stepanoff[1]; M. Schmitt[2]; J. Keiper[2]

1. Pennsylvania State University, USA
2. HAMR Industries LLC, USA

The development of high energy lasers (HELs) has garnered significant interest in the advancement of photonic nanostructures as a method of protecting sensitive components or the human eye. Traditional photonics rely on absorption of electromagnetic radiation, limiting their multi-hit survivability due to thermal energy dissipation, ultimately compromising functionality. Reflective optical limiter coatings can be employed to protect subjects from high irradiances. This study highlights the engineering of multilayered thin films as reflective optical limiters that exhibit an on/off

non-linear optical response that allows for transmission at a specific wavelength at low incident irradiances, and near-100% reflection at high incident irradiances. The optical limiter coating system is comprised of a non-linear material deposited within the cavity of a Fabry-Perot filter consisting of two mirrored quarter wavelength distributed Bragg reflectors. In this study, both nitride and oxide based material systems were deposited utilizing magnetron sputtering and electron-beam physical vapor deposition, respectively. Furthermore, this investigation demonstrates broadband and multiband optical spectra through a tailoring of the coating design architecture. Experimental laser testing results validate a non-linear optical response and high irradiance multi-hit survivability.

S18: Ultra-High Temperature Ceramics

Simulations and Characterizations

Room: Coquina Salon A (North Tower)
Session Chair: Tianli Feng, University of Utah

1:30 PM

(ICACC-S18-032-2023) Accurate first-principles prediction of thermal and mechanical properties of ultra-high temperature ceramics (Invited)

T. Feng*[1]

1. University of Utah, Mechanical Engineering, USA

The fundamental thermal transport processes at ultra-high temperatures in solids remain unclear and in debate, and the predicted thermal conductivity and mechanical properties cannot match the experiment in most materials at high temperatures. Without a good understanding or accurate prediction of thermal transport, many potential revolutions in materials and technologies may not be realized. In this talk, I will show our recent efforts in the higher-order temperature-dependent first-principles-based predictions of thermal and mechanical properties for ultra-high-temperature ceramics. We include both three and four-phonon scattering as well as the temperature-dependent force constant, for the first time. By utilizing machine learning techniques, we trained interatomic potentials that have the first-principles level accuracy. Through the potentials, we could run molecular dynamics simulations to predict the thermal and mechanical properties as well. The predicted thermal and mechanical properties agree well with the experiment. The impact of grain size and defects are also studied. This work sheds important light on the fundamental thermal transport in ultra-high temperature ceramics and will pave the way toward the engineering and development of next-generation technologies with thermal management at ultra-high temperatures.

2:00 PM

(ICACC-S18-033-2023) Local bonding and carbon vacancy ordering in UHTC high-entropy carbides

T. Davey*[1]; Y. Chen[1]

1. Tohoku University, School of Engineering, Japan

High-entropy or multi-principal component ultra-high temperature ceramics, including the rocksalt structured MC_{1-x} (where M is an equiatomic or non-equiatomic mixture of metallic elements including Ti, Zr, Hf, V, Nb, and Ta), have recently generated significant interest due to their potential tuneable properties such as melting point, hardness, ductility, and oxidation resistance. The single metallic element carbides, such as zirconium carbide, are known to have a wide range of stoichiometry facilitated by significant numbers of carbon vacancies that have a tendency towards ordering at low temperature, with different trends observed in group IV and group V transition metal carbides. This work uses first-principles calculations to explore the effects of multiatomic mixing on the metal lattice on the vacancy ordering in high entropy carbides. Trends are identified considering the local atomic environment and atomic bonding features.

2:20 PM

(ICACC-S18-035-2023) Stability of GDC and MgO Ceramics in Combustion Environments for Direct Power Extraction

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2. National Energy Technology Laboratory, Albany, USA

One of the greatest challenges in the development of an open-cycle magnetohydrodynamic (MHD) power generator is material selection. The environment necessary for efficient power extraction consists of wall temperatures exceeding 1600°C, oxygen partial pressures up to 0.001 atm, and atomic vapor potassium concentration near 1% by weight. Two materials which have been hypothesized to withstand such an environment are MgO and Gd doped CeO_2 (GDC). Both have shown resistance to potassium vapor attack in inert atmospheres, have melting points far exceeding desired wall[CBT1] temperatures, and have adequate electrical properties to function as insulating and conducting components. Experiments testing these materials against a high-velocity oxy-fuel combustion plume emulating a MHD environment have shown that additional corrosion mechanisms outside of direct potassium reactions can occur in these materials due to the addition of combustion products.

Compositionally Complex UHTC III

Room: Coquina Salon A (North Tower)
Session Chair: Bai Cui, University of Nebraska–Lincoln

3:20 PM

(ICACC-S18-036-2023) Composition-Property Relationships in High-Entropy Boride Ceramics

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

Dense high-entropy boride ceramics with high phase purity were produced by two-step spark plasma sintering of precursor powders synthesized by boro/carbothermal reduction of oxides. This study demonstrates a reliable and cost-effective processing for producing dense high-entropy boride ceramics with high phase purity. High-entropy borides have wide composition ranges, while the relationships between composition and property have not been systemically studied. Processing and characterization of a series of high-entropy boride ceramics with equal metal atom ratios were investigated. The results indicated that high-entropy boride ceramics containing Cr or W had Vickers hardness values higher than 45 GPa at a load of 0.49 N. Flexural strength and fracture toughness will be measured at room and elevated temperatures. Thermal diffusivity and heat capacity will be measured using the laser flash method, and then used to calculate the thermal conductivity. This study systemically investigated composition-property relationships in high-entropy boride ceramics.

3:40 PM

(ICACC-S18-037-2023) Predicting Phase Stability of Compositionally Complex $5RE_2B_2O_7$ Type Rare Earth Zirconates

D. R. Lowry*[1]; M. Blea-Kirby[1]; J. Boro[1]; L. Jauregui[1]; N. Valdez[1];
M. Rodriguez[1]; S. Bishop[2]

1. Sandia National Laboratories, USA
2. Sandia National Laboratories, Materials, USA

This presentation will discuss the role of composition in determining the phase stability of compositionally complex $A_2B_2O_7$-type rare earth zirconates. Compositionally complex ceramics are interesting due to the ability to tailor material properties based on selected compositions. $RE_2Zr_2O_7$ materials are candidates for application as next generation thermal barrier materials. In this work 5 component rare earth zirconates, general formula $A_2Zr_2O_7$ where

A = La^{3+}, Nd^{3+}, Sm^{3+}, Eu^{3+}, Dy^{3+}, Y^{3+}, Er^{3+}, Ho^{3+}, or Yb^{3+}, were experimentally fabricated, the phase stability analyzed, and predictive formulas developed for, e.g., lattice parameter and phase stability based on ionic radii parameters. Recent compositions from the literature are shown to be stable after higher temperature anneals. Additionally, thermal expansion tuning will be discussed. Sandia National Laboratories is a multimission laboratory managed and operated by National Technology & Engineering Solutions of Sandia, LLC, a wholly owned subsidiary of Honeywell International Inc., for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525. SAND2022-0317 A.

4:00 PM

(ICACC-S18-038-2023) Ab Initio prediction and experimental preparation of $(HfNbTaTiZr)B_2$ structures based on the different molar ratios of transition metals

I. Zhukova*[1]; V. Kombamuthu[2]; M. Tatarkova[2]; D. Zagorac[3]; A. Kovalčíková[5]; I. Dlouhy[4]; B. Matovic[3]; P. Tatarko[2]

1. Slovak Academy of Science, Institute of Inorganic Chemistry, Slovakia
2. Institute of Inorganic Chemistry, Slovak Academy of Sciences, Department of Ceramics, Slovakia
3. Institute of Nuclear Sciences, Belgrade University, Serbia
4. Institute of Materials Science and Engineering, Czechia
5. Institute of Materials Research, Slovakia

High-entropy borides (HEB) are a new class of ultrahigh temperature ceramics. Until now, most of the research interest has been focused on the HEB materials with equimolar composition of transition metals. Among all of the HEB materials, (Ti-Zr-Hf-Nb-Ta)B_2 is one of the most investigated compositions. The present work was aimed at studying the effect of various molar ratios of five transition metals using density functional theory, with the compositional disorder being treated using the special quasi-random structures technique (SQS). Employing the Alloy Theoretic Automated Toolkit software, the SQS supercell (5×2×2) with 60 atoms was selected for the present calculations. The goal was to understand the effect of individual metal elements, different atomic positions and concentrations on the formation of the diboride structure. The different molar ratios selection criteria were based on the solubility of atoms in each other, formation and cohesive energies. The bulk, shear and Young's modulus of non-equimolar structures were studied and compared to experimental data. The individual HEB compositions were synthesized using boro/carbothermal reduction of oxide precursors, followed by Spark Plasma Sintering at the temperature range of 1800–2000°C. The lattice parameters of the materials were calculated using Rietveld refinement of XRD patterns.

Poster Session B

Room: Ocean Center Arena

5:00 PM

(ICACC-P053-2023) Effects of resin pocket length on cracking behavior in CFRP laminate with ply discontinuity

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1. Tokyo University of Science, Mechanical Engineering, Japan
2. Newcastle University, School of Engineering, United Kingdom

Ply termination that is often used to alter the laminate thickness or fiber orientation, accommodate changes in structural geometry, or create a hybrid composite by changing the ply material usually creates ply discontinuities in composite structures. When an internal ply is terminated, a resin-rich pocket is formed at the edge of the dropped ply which leads to stress concentrations and damage initiation. In this study, the mechanical properties and damage behavior in laminates with varying resin pocket lengths were elucidated. A simple specimen geometry of unidirectional CFRP laminates with no alternating thickness is used, with the aim of exploring the behavior of the resin pocket. Experimental observations revealed that a single crack occurred in a very short resin pocket and delamination initiated from the tips of the crack at lower applied stress compared to the laminates with longer resin pockets. On the other hand, a longer resin pocket may form several cracks prior to initiation of delamination where the observed cracks typically occur within the resin pocket, rather than at the interfaces with the discontinuous ply, and are curved towards the center of the pocket. It is demonstrated that the number of cracks in the resin pocket and crack locations are susceptible to the resin pocket length and the geometry's uniformity in the thickness and width direction of the specimen.

(ICACC-P054-2023) Deformation and molecular orientation analysis of tricalcium phosphate / poly(lactic acid) composite screws molded by extrusion die forging

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1. Salesian Polytechnic, Mechanical and Electronic Engineering, Japan
2. Tokyo Metropolitan University, Mechanical Engineering, Japan

The composite of tricalcium phosphate (TCP) and poly(lactic acid) (PLA) had been attracted the attention as a material of bone fixation devices. However, application of this TCP/PLA composite was limited to low-load regions, because the strength was insufficient. Molecular orientation by forging was focused to improve the strength of TCP/PLA composite in this study. The deformation behavior of forging with compressive plastic deformation and the orientation behavior of solid-state deformed polymer are complex. In this study, TCP/PLA screw was focused as a bone fixation device, and was molded by extrusion die forging. The purpose of this study was clarification of the molecular orientation behavior in TCP/PLA screw molded by the die forging. At first, the mechanical property of TCP/PLA was measured under temperature between the glass transition and the melting point of PLA. Next, the deformation of TCP/PLA billet during the die forging was calculated using a finite element method (FEM). The extrusion die forging is a method of extrusion drawing and molding of threads at the same time. Therefore, the deformation during extrusion drawing and forging of threads were calculated, and were superimposed. The molecular orientation obtained by the die forging in TCP/PLA billets was calculated using a chain network model from the strain resulted FEM.

(ICACC-P055-2023) Improvement of powder injection molding process using cellulose nanofibers

T. Osada*[1]; S. Kobayashi[2]

1. Tokyo Metropolitan University, Japan
2. Tokyo Metropolitan University, Mechanical Engineering, Japan

Powder Injection Molding (PIM) process is suitable for fabricating small and complex shaped ceramics or metal parts with high production volume. In this study, in order to improve the PIM process, cellulose nanofibers(CNF) were focused. There are problems such as deformation during debinding and sintering due to the addition of a large amount of binder. On the other hand, the strength of the green compact decreased with decreasing binder contents. In this study, cellulose nanofibers were added with a binder during mixing in order to improve the handling of green compacts, and decrease the deformation during debinding and sintering. Effects of CNF contents on the mechanical properties were investigated.

(ICACC-P056-2023) Mode I fracture toughness of adhesively bonded CFRP joints under cyclic freeze-thaw conditions

K. Kitagawa[1]; S. Oshima*[1]; T. Takeda[2]; H. Kumazawa[2]; K. Koichi[1]

1. Tokyo Metropolitan University, Department of Aeronautics and Astronautics, Japan
2. Japan Aerospace Exploration Agency (JAXA), Aviation Technology Directorate, Japan

The effects of freeze-thaw cycles on the mode I fracture toughness of adhesively bonded CFRP joints were studied. The fracture toughness specimens were immersed into water and exposed to the thermal cycles consisting of thawing (+60 °C) and freezing (−50 °C).

Two types of fracture toughness specimens were prepared; without mechanical loading (non-stressed) and with mechanical loading using a wedge (stressed) during the environmental conditioning. The fracture toughness of non-stressed specimens decreased, whereas that of stressed specimens increased. The fracture mechanisms are discussed based on the results of the fracture toughness tests, the tensile properties of bulk adhesives, and the fracture morphologies of specimens.

(ICACC-P057-2023) Residual Strength Evaluation for Composite cylinders using Plate Specimen

S. Kobayashi*[1]; T. Osada[2]

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2. Tokyo Metropolitan University, Japan

In this study, as a new method for evaluating the internal pressure strength of the composite vessel after the impact test, an evaluation method using a plate specimen was proposed, and its validity was examined. The impact test was carried out under the conditions determined by finite element analysis, and then the internal pressure test of the composite vessel and the tensile test of the plate specimen were carried out. As a result, in the impact test, it was confirmed that the composite vessel and the plate specimen were damaged on the same scale and the residual strength was similar.

(ICACC-P058-2023) AE Based Damage Characterization of CFRP with Considering AE Sensor Response Function

A. Garg*[1]; T. Sakai[1]

1. Saitama University, Japan

The utilization and acceptability of composites, particularly CFRP, is increasing at a very rapid pace in recent times. Apart from its wide usage in Aerospace industry, it now finds application in diverse areas like automotive, renewable energy and other sectors also. But still, given its complex damage mechanisms, their reliability remains a big concern as well as a challenge to its wide acceptability. So understanding their damage mechanism is very much essential. The Acoustic Emission (AE) method has been widely used to determine and characterize the various type of damages in the composites. But there is huge variation in these characterization result. The reason for these variation can be due to difference in the AE sensor, Material, Experimental setup. So if we want to improve the characterization results, we have to focus on reducing the effect of these parameters. Therefore, in this study, to reduce the effect of AE sensors we proposed an evaluation method that takes into account the response function of AE sensors. The response function of the AE sensor was obtained from the response to the input signal using a swept-sine waveform with a function generator. By considering the response function, the relationship between the peak frequency and the waveform could be more accurately represented. Using this method, the damage evaluation of CFRP could be performed more accurately.

(ICACC-P060-2023) Splitting Progress in Unidirectional Carbon Fiber Reinforced Plastics

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2. Tokyo Metropolitan University, Japan

The energy release rate associated with splitting progress in unidirectional (UD) carbon fiber reinforced plastics (CFRP) laminates is derived using shear-lag type stress analysis. Fatigue tests were conducted on UD-CFRP with three types of carbon fibers. It is clarified that the splitting progress rate is expressed by Paris's law type relation.

(ICACC-P061-2023) Fabrication of Biocompatible Oxide Ceramic Parts by Injection Molding

T. Osada*[1]; Y. Nagai[1]; S. Kobayashi[2]

1. Tokyo Metropolitan University, Japan
2. Tokyo Metropolitan University, Mechanical Engineering, Japan

In this study, we proposed a new fabrication method of artificial bone materials using Ceramic Injection Molding (CIM) and verified the mechanical properties of the fabricated compacts. Since ceramic parts can be machined in the green parts from CIM, it is possible to manufacture custom-made artificial bones that match the defective parts. Powder loading, binder composition, and content were changed for controlling the porosity of sintered compacts. As a result, bending strength slightly decreased with increasing porosity, however, it is enough for artificial bone.

(ICACC-P062-2023) High Thermal Conductivity Reaction-bonded Diamond/SiC Composites

J. Wang*[1]; J. Coppola[1]; N. Coombs[1]; M. Aghajanian[1]

1. II-VI Inc., USA

Reaction bonded Diamond/SiC/Si composites are fabricated by the reactive infiltration of molten Si into preforms of diamond particles. Molten Si reacts with diamond, forming SiC and leaving residual Si in the matrix. Reaction bonded Diamond/SiC/Si composites have favorable properties including enhanced hardness and wear resistance, high thermal conductivity and stiffness, low thermal expansion, providing value in applications such as semiconductor equipment and thermal management. In this study, we optimized the microstructure of the Diamond/SiC/Si composites by controlling the diamond particle size, mixing ratio and infiltration process, to eliminate residual Si in the matrix. The optimized diamond/SiC composites, with no residual Si in matrix, have very high stiffness and thermal conductivity. This study correlates the starting diamond powder size and mixing ratio with the microstructure, mechanical and thermal properties (stiffness, CTE and thermal conductivity) of the reaction bonded composites.

(ICACC-P063-2023) ASTM International Standards for Properties & Performance of Advanced Ceramics–Helping Our World Work Better with Standard Worldwide

M. G. Jenkins*[1]; S. T. Gonczy[2]; J. Salem[3]; J. Westbrook[4]; G. D. Quinn[5]

1. Bothell Engineering and Science Technologies, USA
2. Gateway Materials Technology, USA
3. NASA Glenn Research Center, Materials and Structures, USA
4. Corning Incorporated, SP-FR-04, USA
5. National Institute of Standards and technology, Materials Measuremenet Sciences Division, USA

Physical and mechanical 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 in its 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-P064-2023) The effect of BN coating layer thickness for LSI processed SiC/SiC composites

S. Kim*[1]; I. Han[1]; H. Bang[1]; S. Kim[1]; Y. Seong[1]; S. Lee[1]

1. Korea Institute of Energy Research, Republic of Korea

SiC/SiC composites are applied to high-temperature parts of aircraft engines and in various industries. An appropriate interfacial coating such as BN is applied to the SiC fiber for the SiC/SiC composites. It is not only improve the toughness and mechanical properties, but also protect the fibers during the high-temperature process. In this study, the behavior of BN coating on SiC fiber was confirmed during process of SiC/SiC composites using the Liquid Silicon Infiltration. Damage to the coating was confirmed due to the phenomenon that B is diffused out during process in which a high-temperature Si-C reaction is induced. As a result, the mechanical properties of the composites were also decreased. On the other hand, as the thickness of the BN coating increased, the mechanical properties improved, but at the same time, the porosity of composites increased. It could be explained by introducing the 'pore open factor', and it was analyzed that the change in the fiber volume fraction according to the coating thickness also contributed to the increase in porosity. As a result of the study, the fiber coating of the SiC/SiC composites protects the fiber from high-temperature process and at affects the formation of pores. Therefore, it was confirmed that applying an interfacial layer with an appropriate thickness for the LSI process is a factor that can maximize the densification and mechanical properties.

(ICACC-P065-2023) A study on the SiC slurry and SiC slurry tape for LSIed-SiC$_f$/SiC composite

Y. Seong*[1]; S. Lee[1]; S. Kim[1]; S. Kim[1]; H. Bang[1]; I. Han[1]

1. Korea Institute of Energy Research, Republic of Korea

In order to apply a superalloy with a temperature limit of 950 °C to H-Class gas turbine parts with TIT(Turbine Inlet Temperature) of 1600 °C, the temperature is lowered by 150 °C with thermal barrier coating and more than 500 °C with air cooling technique. This results in a significant reduction in the power output and efficiency of the gas turbine. Therefore, the development and application of CMC material with non-cooling technology is considered as the only alternative for the required lifetime and stable operation of parts that require high temperatures for high output/high efficiency gas turbines. In order to improve the properties of CMC materials, fiber protection coating technology, high strength matrix manufacturing technology, and composite densification technology must all be secured. In this study, we have studied the ceramic filler based slurry for high strength matrix using liquid silicon infiltration(LSI) method. Specifically, SiC slurry was applied for the purpose of increasing strength by suppressing residual silicon in SiC$_f$/SiC. The mechanical properties(tensile strength/creep/fatigue) tests are also conducted at 1315 °C. Additionally, SiC slurry tape was studied using tape casting process to secure mass productivity by increasing the reliability of SiC$_f$/SiC material.

(ICACC-P066-2023) New process and fabrication techniques for zirconia-toughened alumina (ZTA) ceramic gelcasting methods

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Yttria (3mol%) stabilized zirconia (YSZ, 50-80nm) and alumina (30-40μm) powders combined with <1wt% graphene oxide (6-28μm) shows considerable improvement in flexural strength and toughness. These ceramic powders combined with water soluble binders have varying viscosities due to the solids content and ability to stay in solution. Polyethylene glycol (PEG), Polyvinylpyrrolidone (PVP), and Polyvinyl Alcohol (PVA) were combined with ammonium phosphate and other salts to disperse ceramic powders in solution. Gel formers such as starches, alginates, and agar were studied to determine the optimum gelation time for the solutions. For shear rates below 10/s for slips and gels, the viscosity measurements were useful for predicting the maximum solid loading (>50 vol%) of the gel for optimum flow in the mold. As the viscosity of the gel increased the flow through the mold decreased, therefore vacuum was applied to improve flow and prevent air pockets during molding. A green body drying was determined to be 500°C for 2 hours for crack prevention and optimum densification through thermogravimetric analysis of the final gel product. It is expected that these new process and fabrication techniques would result in simple and repeatable gelcasting processes for engineering ceramic materials.

(ICACC-P067-2023) A study on the properties change of SiC/SiC ceramic matrix composites after high temperature long-term fatigue test

S. Lee*[1]; I. Han[1]; S. Kim[1]; Y. Seong[1]; S. Kim[1]; H. Bang[1]

1. Korea Institute of Energy Research, Republic of Korea

SiC/SiC has characteristics such as high thermo-mechanical stability and fracture toughness. It is widely used in high-temperature parts in military fields. In this study, the SiC/SiC composites were exposed to a long-term high temperature in air to confirm the oxidation characteristics used in a high temperature environment and the properties change due to oxidation behavior were analyzed. The samples were manufactured by MI process with multi-coated SiC fiber and conducted 5 X 10^6 cycle fatigue test without failure in conditions of air during 164hour, 10.0Hz, R=0.05, monotonic tensile load. After fatigue test the retained tensile test was conducted. As a result, the specimens subjected to the fatigue test showed improved strength and decreased porosity compared to the specimens exposed to high temperatures short-term. It was confirmed that as the oxidation exposure time increased, the pores were filled and the density of the composites increased. The increase in density was due to the formation of Cristobalite Silica around the pores and was confirmed using XRD/SEM/TEM etc. This oxidation behavior is considered to have a positive effect on the reliability improvement of SiC/SiC composites mainly used at high-temperatures.

(ICACC-P068-2023) Electrochemical study of Inconel 600 to TiC-Composites joints using a BNi3 alloy

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1. Instituto Tecnológico Superior del Sur de Guanajuato, Ingeniería en Sistemas Automotrices, Mexico

This work shows the electrochemical study of dissimilar joint of inconel 600 to TiC/Ni immersed in synthetic seawater. The composite-inconel 600 assemblies were brazed with BNi3 alloy by brazing technique, in an inert atmosphere at work temperature of 1100°C. The corrosion rate (CR) was evaluated from the technique of electrochemical noise (EN). In addition, the corrosion potential measures as a function of the time were carried out to get the stabilization time of the anodic and cathodic reactions. Electrochemical measurements were carried out under static conditions, ambient temperature and atmospheric pressure, the electrochemical results show that the susceptibility of localized corrosion of composite sample was higher than the joints. The corrosion process is of mixed type, that us to say a process of transfer of load influenced by a mechanism of mass. Analysis by scanning electron microscopy (SEM) revealed that mechanism corrosion was cells differential aeration (difference of concentration of oxygen).

(ICACC-P069-2023) Synthesis of carbides by reactive sintering from binders for 3D printing

D. Valasek*[1]; D. Salamon[1]

1. Brno University of Technology, CEITEC - Advanced Multifunctional Ceramics, Czechia

The distribution of carbon during reactive sintering of heavy carbides (WC and similar) is crucial due to the low diffusion of carbides. Sintering of pure carbides by reactive sintering requires high temperature and pressure or special techniques such as Flash sintering. In this work, we are using reactive carbon prepared by partial debinding prior to sintering. Tungsten carbide (WC) was prepared by reactive sintering of powder obtained by this novel approach to investigate the influence of different weight percentages of tungsten on phase formation and hardness of the final product. Several homogenous mixtures of tungsten powders and resin with various tungsten-to-carbon ratios were prepared and transformed into reactive carbon by the debinding process. The resulting powder was sintered by using Spark Plasma Sintering (SPS) apparatus at 1600 °C under the pressure of 50 MPa. The sintered samples were observed using Scanning Electron Microscopy, and an X-Ray Diffraction analysis was performed. In the end, the Vickers hardness of the samples was measured on highly dense samples. It was found that the initial tungsten-to-carbon ratio influences the presence of additional phases in the system. Both WC and tungsten semi-carbide (W_2C) were found in the samples. It was proved that the debinded resin for 3D applications could be used as a highly reactive and homogeneous carbon source during reactive sintering.

(ICACC-P070-2023) Effect of fine-bubbles on enhanced charge transfer reaction in glass CMP process

S. Mochizuki*[1]; S. Suda[1]

1. Shizuoka University, Engineering, Japan

Chemical mechanical polishing (CMP) is indispensable engineering or technologies for planarizing glasses or semiconductor substrates. Charge transfer reactions would occur by applying shear stress between glass and abrasives. Then, soften hydration layer would be formed on the surface of glasses as chemical polishing of glass CMP process. Ceria and colloidal silica are generally used as glass CMP abrasives. Hydroxide ions on the surface of these abrasives are contributed to the charge transfer reactions. As a novel abrasive material instead of these solid abrasives, we focus on fine bubbles. Fine bubbles are generally charged negatively in water, and they have highly concentrated hydroxide ions in the vicinity of gas-liquid interface of bubbles. The hydration layer is evaluated by measuring the electric potential change under CMP process. Soda lime glass was used as a work piece and p-type Si wafer was used as a poor chemical reactive substrate. NH_4NO_3 water solutions with or without fine bubbles was used as slurry. The electric potential change for the fine-bubbles solutions was 10 times larger than that for the bubble-free solutions. Hydroxide ions in the fine-bubbles vicinity were enhanced the charge transfer reactions, and the fine-bubbles would work as abrasives similarly to colloidal silica.

(ICACC-P071-2023) Fabrication of Carbon Fiber Reinforced Ceramic Matrix Composites

M. Rasheed*[1]; S. Mujib[1]; G. Singh[1]

1. Kansas State University, Mechanical and Nuclear Engineering, USA

Ceramic matrix composites (CMCs) are suitable candidates for aerospace and aircraft components due to the composites' lightweight structure, high mechanical strength, and high temperature stability. In this context, Si-based precursor-derived ceramics (PDCs) have processing flexibility that allows to achieve desired shape and control of the final ceramic product. PDCs have shown high resistance to various loads, deformation, and creep. In this study, SiCN/CF, and Si(B)CN/CF CMC mini-composites were fabricated with carbon fiber (CF) reinforcement using Polysilazane-based single-source liquid precursors. A polysilazane-based precursor was mixed with boron-containing precursors to synthesize Si(B)CN polymeric precursor. The homogenous solutions of the precursors were then allowed to infiltrate CFs via a drop-coating process. The coated CFs were cross-linked at 180C for 16 h and later pyrolyzed at 800C for 30 min in argon atmosphere to achieve CMC mini-composites. The SiCN/CF and Si(B)CN/CF composite bundles exhibited high mechanical strength, averaging a max. stress of 390 and 110 MPa respectively under tensile load. The CMC mini-composites also showed improved oxidation resistance up to 1500 °C, when compared to base CF material.

(ICACC-P072-2023) Effect of Al_2O_3 and Carbon Fiber Addition on Thermal Conductivity and Tensile strength of Polyamide-6

M. Ijiri*[1]; S. Ishida[1]; T. Osada[1]; S. Kobayashi[1]

1. Tokyo Metropolitan University, Japan

Polyamide-6 (PA6) has excellent wear resistance and impact resistance, but its low thermal conductivity and mechanical strength require the addition of fillers. In this study, we investigated the optimum contents of Al_2O_3(AO) and Carbon fiber (CF) as fillers to improve the thermal conductivity and tensile strength of PA6. In PA6 with only AO added, the Young's modulus and tensile strength decreased when the content was over 40 vol%, but the thermal conductivity increased. However, the tensile strength decreased at a content of 35 vol% or more. Furthermore, in order to improve the thermal conductivity, the total filler ratio was fixed at 40 vol% and the samples were prepared. The thermal conductivity improved when the AO content was high. When the AO content was high, the thermal conductivity improved, but the Young's modulus and tensile strength decreased. The cause was thought to be the presence of a gap at the interface between the filler and the resin.

(ICACC-P073-2023) A fractal analysis of crack branching and the relationship to fractographic parameters

D. P. DeLellis*[2]; N. A. Mecholsky[3]; G. D. Quinn[1]; A. Krause[2]; J. Mecholsky[4]

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Fractal analysis is a powerful tool for quantitatively characterizing tortuous features. It is useful in failure analysis because it relates the fracture surface features via a fractal dimensional increment, D*, to fracture toughness. Some work has also demonstrated that the fractal dimension of the crack branching pattern, the crack branching coefficient (CBC), can be related to fracture stress. The present work describes the application of fractal analysis to crack branching patterns in borosilicate glass disks broken in biaxial flexure. It is shown that a larger CBC is correlated with a greater strength. Furthermore, it presents newly identified relationships between the fracture surface fractal dimension, the crack branching coefficient, and the fracture energy. Combining these analyses allows for a more thorough fractographic examination through the observation of fracture via multiple features.

(ICACC-P074-2023) Processing and mechanical properties of ZrO_2-Zr ceramic-metal composites

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ZrO2 (3Y-TZP) matrix composites reinforced with Zr metallic particles were fabricated by colloidal processing route followed by spark plasma sintering (SPS). The microstructure and mechanical properties of this novel ceramic–metal composite were investigated. Compacts with high densities (98% of the theoretical density) and microstructures with uniformly distributed metallic particles in the ceramic matrix were obtained. The fracture toughness of composites was slightly higher than the values corresponding to monolithic zirconia. Scanning electron microscope (SEM) observations of the crack path showed that the major contributions to toughening were the resulting crack blunting and branching that occurs at crack tips in the metallic particles before the onset of crack propagation.

(ICACC-P075-2023) Repair Method of Blast Furnace Hot Stove Combustion Chamber

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Blast furnace hot stove, apparatus for preheating air blown into a blast furnace, an important step in raising the efficiency of iron processing. Blast furnaces are served by three stoves. While two are being heated, the air blast passes through the regenerative chamber of the third stove on its way to the blast furnace. Hot-blast stoves in particular consist of Silica Refractories, which take a long time to cool down. Therefore, it is difficult for workers to cool and repair Refractories. And operating with two hot stoves has a huge impact on blast furnace production. Our hot stove combustion's hot blast outlet part was damaged. It had to be repaired. We had a lot of thought on how to repair that part. At first, hot stove should not be cooled, so I thought about making heatshield and repairing it. This, however, caused difficulties in safety or repair times. Another method of repairing Ceramic Welding in glass melting furnaces was found. The combustion chamber set brick has been repaired for Ceramic Welding within the shortest time, and it has been used well so far without damage.

(ICACC-P077-2023) Preferred-oriented BEAZ grains Protective layer for enhanced chemical stability toward Strontium

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Low temperature conducting electrolyte is vital for the higher performance of solid oxide fuel cells. Unlike conventional oxide ion (O^{-2}) conductors, proton (H^+) conductors exhibit high ionic conductivity owing to its reduced activation energy and have high theoretical electrical efficiency in the target temperature window. A new family of conductors such as hexagonal perovskites were studied for its inherently oxygen deficient hexagonal layers which have shown good results thus far. Herein, an innovative synthesis method has been devised to develop a new promising Ni doped $Ba_5Er_2Al_2ZrO_{13}$ hexagonal perovskite with highly oxygen deficient hexagonal (h) layers. The total conductivity investigated by impedance spectroscopy was in the order of 10^{-2} Scm^{-1} at 600 °C which is promising corresponding to their counterpart electrolytes such as $Ba_5Er_2Al_2ZrO_{13}$ and $Ba_5In_2Al_2ZrO_{13}$. Furthermore, it is evident from the analysis that bulk conductivity is dominant compared to grain boundary conductivity. HAADF confirms the Ni at the 001 plane, insinuating the accumulation of Ni in the grain boundaries, resulting in enhanced grain boundary conductivity as well. According to the results of the EBDS analysis, the hexagonal perovskite has a preferred orientation at plane 0001 that is favourable for proton conductivity.

(ICACC-P078-2023) Cation diffusion and electrode properties in LSM/CeO_2 nanocomposite cathode

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SOFC cathode materials generally have low heat-resistant temperatures, and they are easily decomposed or degraded by sintering at high temperatures that are suitable for co-sintering cells. (La, Sr) MnO_3 (LSM) shows relatively high resistance to decomposition and low reactivity during sintering at high temperatures. But it shows low cathodic properties because of poor oxygen diffusion. We developed LSM/CeO_2 nanocomposites in order to combine electronic conduction of LSM and oxygen diffusion of CeO_2. Highly dispersed LSM/CeO_2 nanocomposites were obtained by glycine method. The nanocomposite showed advantages in cathodic properties, but it brought about interdiffusion between LSM and CeO_2. Thus, cation diffusions were investigated by XRD and SQUID. The two analysis methods clarified apparent La and Sr diffusion from LSM into CeO_2 lattice, but they did not show clear evidence of Mn diffusion. The diffusion of La led to increase oxygen conductivity of CeO_2, but that of Sr would reduce cathodic properties of LSM. The diffusion behavior of Sr also was also different from that of La during sintering at high temperatures. Therefore, diffusion of La and Sr in the nanocomposite would be controllable by optimizing sintering processes.

(ICACC-P079-2023) Effect of the CGO solution properties on the long-term performance of CGO infiltrated Ni-YSZ SOEC electrodes

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The purpose of this work is to study and optimize the infiltration of Cerium Gadolinium oxide (CGO) in the Ni-YSZ electrode of SOEC in order to improve the long-term electrochemical performance of the cell. The Ni-YSZ electrode of fuel-electrode supported cells is infiltrated using CGO solutions of different concentrations but targeting the same CGO loading (185 mg/cm^3). The cells' performance and degradation are comprehensively analyzed and quantified via operando impedance spectroscopy. Post-mortem analysis of the cells provides extra info about the microstructure changes and the CGO agglomeration. The parameters such as the solvent type (water/ ethanol), the used surfactant, and the solution concentration are further considered for the optimization of the infiltration procedure. The cell infiltrated in 3 cycles with 1 M CGO solution (Cell-1M) is operated in a continuous galvanostatic operation for 5270 h. A similar cell infiltrated in one cycle with 3M CGO solution (Cell-3M), exhibits inferior long-term performance when operated in a continuous galvanostatic mode compared to the Cell-1M. For this reason, Cell-3M is operated for 3030 h in an intermittent galvanostatic operation with intervening periods of in-situ heat treatment. The results show that intermittent galvanostatic operation can extend the life-time of the cell that exhibited initial inferior performance.

(ICACC-P080-2023) Optimal microstructure for fabrication anode-supported SOFC by microwave selective sintering

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Co-sintering of thick anode, thin electrolyte and thin buffer layer is essential to fabricate anode-supported or metal-supported solid oxide fuel cells. However, delicate sintering or additive control is required to obtain adequate anode and electrolyte microstructures by co-sintering using conventional furnace. For example, co-sintering of the half cells composed of 8 mol% yttria stabilized zirconia (8YSZ) electrolyte and NiO/8YSZ anode easily bring about over-sintering of NiO of the anode. (La, Sr) (Co, Fe) O_3 cathode materials also require the introduction of buffer layer to prevent reactions with electrolyte and the co-sintering of anode, electrolyte, and buffer layer. However, sintering temperature of CeO_2 buffer layer is generally higher than that of 8YSZ, and the allowable co-sintering conditions for appropriate microstructures would be restrictive. We then focused on microwave sintering, which has selective heating properties. The microwave selective sintering was much affected by microstructure of calcined compacts as compared to dielectric loss factor of materials. The selectivity of microwave sintering was thus controlled using adequate calcination conditions. Adequate calcination led to anode/8YSZ/CeO2 half cells with adequate microstructures as well as anode/8YSZ half cells by microwave co-sintering.

(ICACC-P081-2023) Change in SOFC cathodic properties of $La_{1-x}Sr_xFe_{1-y}Ni_yO_3$ by sintering high temperatures

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SOFC is one of the most efficient energy generation systems, and advanced power density per unit volume would be required to spread extensively. Co-sintering among electrodes and electrolyte is one of the most important challenges to overcome for highly efficient compact SOFC. (La, Sr) (Co, Fe)O_3 is widely used as a high-performance cathode material, but it easily react with fluorite electrolyte by sintering at high temperature. We investigated $La_{1-x}Sr_xFe_{1-y}Ni_yO_3$ (LS_xFN_y) which would be a candidate cathode that is applicable to co-sintering by providing both sufficient heat resistance and high cathodic properties. LS_xFN_y (x=0.0-0.4, y=0.0-0.2) powder was synthesized by glycine method. LS_xFN_y ceramics obtained by sintering at 1200°C or 1400°C showed a single phase of perovskite structure regardless of sintering temperature. Electronic conductivity was increased with increased Sr and Ni composition (x, y), and it was much affected by A site composition as compared to B site. But no significant degradation was observed by sintering at 1400°C. On the other hand, oxygen diffusion coefficient was much suppressed by sintering at high temperature of 1400°C and excess dissolution of B site. Oxygen diffusion was important for cathodic properties of LS_xFN_y and the optimization of B site of LS_xFN_y would be essential to obtain the applicable cathode materials.

(ICACC-P082-2023) Metal-Supported Solid Oxide Fuel Cell Using Proton Conducting Electrolyte for Direct Ammonia Utilization

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Metal-supported solid oxide fuel cells (MS-SOFCs) have better thermal and mechanical properties compared to conventional cermet anode-supported SOFCs. But the different physical and chemical properties between metal and cermet materials, especially the difference in thermal expansion coefficients (TEC) and sinterability, cause critical issues in the fabrication and performance of the cells. In this study, MS-SOFCs have been successfully fabricated with Ni metal anode and proton-conducting electrolyte $BaZr_{0.1}Ce_{0.7}Y_{0.1}Yb_{0.1}O_3$ (BZCYYb (1711)) at 1350 °C. The performance of the Ni-supported cells is evaluated with a triple conductive Pr_2NiO_4 (PNO) cathode. The cell shows a peak power density of 0.72, 1.05 W/cm^2 at 700 °C for NH_3 and H_2 fuel, respectively. With an upstream NH_3 pre-decomposition design, we demonstrate a 340 h stable operation of MS-SOFC in NH_3 at a constant current density of 350 mA/cm^2 at 600 °C.

(ICACC-P083-2023) Numerical Modeling of Long-Term Performance Degradation for Solid Oxide Fuel Cell with Multiple Degradation Mechanisms

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The long-term performance of solid oxide fuel cells (SOFCs) is affected by multiple degradation mechanisms. The dominant degradation mechanism can also change with the operating time. In the present study, a robust numerical model with multiple, selectable degradation mechanisms is developed to extend the capability of the multiphysics simulations of realistic SOFCs. This numerical model utilizes the microstructural properties either calculated from a phase field model or estimated from a reduced order model. Then with the microstructural properties at each time stage, the polarization curves and electrochemical impedance spectrum of fuel cells are simulated. To validate the numerical model, the predicted performance degradation at each time stage is compared with experimental datasets. Parametric studies are also performed to investigate the long-term cell performance under various operating conditions. Finally, thorough analysis of the long-term performance degradation reveals the change in dominant degradation mechanism over time. The evolution of species transport processes and charge transfer processes within both electrodes are also analyzed. This study provides deeper insights of long-term performance degradation, indicating appropriate mitigation strategies at different stages of operation to extend the cell lifetime.

(ICACC-P084-2023) Oxidation inhibitor ceria-layers for SOC's metal support by laser powder bed fusion

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The oxidation/corrosion of SOC's metal supports during the operation in the harsh environment is a critical issue that limits the long-term stability of metal-supported SOCs. Oxidation-resistant coatings are commonly used to mitigate the degradation of metal supports. However, the irregular and high-tortuosity inner surface of conventional porous metal support challenge the uniformity and effectiveness of the coating. This work shows a 3D printed porous 410L steel support with a novel ceramic multi-coating. 410L stainless steel supports are optimized with regular-distributed straight gas channels by laser powder bed fusion to reduce the tortuosity of the inner surface/channels. The coating is made by combining electrophoretic deposition (EPD) and infiltration. It is based on uniform and dense $Ce_{0.9}Gd_{0.1}O_2/CeO_2$ composite layers that passivate the steel by forming $MnFe_xCr_{2-x}O_4$ and various rare-element-based perovskite phases. The catalytic activity of $Ce_{0.9}Gd_{0.1}O_2/CeO_2$ towards H_2O and O_2 provides chemical protection, dynamically reducing the oxygen activity at the steel/ceria interface. The multi-coated 410L supports show excellent electro-chemo-mechanical stability and superior corrosion resistance under the test conditions of 750 °C/air-3% H_2O/122 h, exhibiting a parabolic rate constant of 0.26×10^{-15} g^2 cm^4 s^{-1}.

(ICACC-P085-2023) Composite glass-zirconia sealings for solid oxide electrolyzers fabricated by 3D printing

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SOE technology is gaining a lot of attention as a way of converting electricity into alternative energy carriers, such as the synthetic fuels. The need for reliable sealings which prevent mixing of oxidant and fuel during operation of SOC requires dedicated designs of seals, optimized composition as well as reliable and low-cost fabrication technique. One of the most promising methods is the 3D printing. The process is waste-free and less complex when compared to tape casting. Moreover, due to the low mechanical strength of the softened glass which commonly makes the seal, hybrid or composite gaskets with enhance properties might be required. The study was focused on the development of glass and glass-zirconia composite pastes suitable for robocasting. A number of different compositions was prepared and used for 3D-printied seals. Evaluation of the rheological properties of the pastes complemented the study. Applicability of these seals was verified in stacks during long-term operation under reference electrolysis conditions. The discussed method makes it possible to obtain seals suitable for the SOC stack operated as an electrolyzer. Moreover, the process can be successfully applied in large-scale production at reduced costs. The presented research was financially supported by the National Centre for Research and Development, Poland, within project no. LIDER/1/0003/L-12/20/NCBR/2021.

(ICACC-P086-2023) Microstructural changes of active Ni fuel electrode/electrolyte interface in solid oxide fuel cells

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The morphological evolutions of the nickel (Ni) stripes on porous yttria-stabilized zirconia (YSZ) layers are investigated under 60 hours potentiostatic solid oxide fuel cell (SOFC) operation. The porous YSZ layers with high and low porosity (ε_{High} and ε_{Low}), large and small pores (D_{Large} and D_{Small}) are fabricated by screen-printing technique. Ni migration towards the electrode/electrolyte interface is observed for both SOFC_$\varepsilon_{High}D_{Large}$ and SOFC_$\varepsilon_{Low}D_{Small}$ cells with enhanced adhesion between Ni and YSZ after SOFC operations. Ni enrichment is found close to the electrolyte surface of SOFC_$\varepsilon_{High}D_{Large}$. Porous YSZ layer with small pores and low porosity can effectively prevent Ni migration and lead to better electrochemical performance. The strong constraining force originates from large pore curvature and corresponding pinning effect from YSZ backbone.

(ICACC-P087-2023) Proton Ceramic Cells for electrochemical reactions: materials and cells development

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This presentation gives an overview of the European WINNER project, started in January 2021. The project is developing an efficient and durable technology platform based on electrochemical proton conducting ceramic (PCC) cells designed for unlocking a path towards commercially viable production, extraction, purification and compression of hydrogen at small to medium scale. This is undertaken in the project in three applications: ammonia cracking to hydrogen or power, dehydrogenation of hydrocarbons, and reversible steam electrolysis. WINNER is developing innovative cell architectures with tubular geometry with a specific focus on the integration of multifunctional electrodes and a novel pressure-less current collection system. The experimental activities are steered by a multi-scale multi-physics modelling platform which addresses all length scales (from atomistic to process). Advanced in-situ and operando characterisation methods are applied to establish correlations between performance and degradation mechanisms upon operation of the cells in these applications. We will present the results achieved so far in the project, spanning from electrode materials development to cell test evaluation in relevant process conditions and development of engineering models describing the various applications.

(ICACC-P089-2023) Thermal Conductivity of Oxide Materials with Metallic Electrical Conductivity

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Research for electrode materials has been mostly limited to metal materials, which have lower high temperature stability, oxidation resistance, and corrosion resistance than ceramic materials. Moreover the high thermal expansion coefficient (16.6 ppm/K for Cu) can be a weak point for simultaneous firing with ceramic materials. This research reports a CaVO3-δ perovskite oxide that has excellent electrical conductivity as much as Ti metal (2.410105 S/cm) which maintains a stable electrical conductivity value regardless of changes in oxidation/reduction atmosphere. A non-redoxable electron-cloud-channel mechanism is proposed as a mechanism to explain the stable electrical conductivity without atmospheric dependence and phase stability of the CaVO3-δ perovskite oxide. It was confirmed that vanadium excess defects and oxygen pores in a single phase and perovskite structure were implemented as donors. A solid-state reaction method, a high-temperature reduction process, and a spark plasma sintering equipment (SPS) were applied to manufacture the CaVO3-δ perovskite conductors and the process parameters were optimized. The CaVO3-δ oxide can be applied to various devices demanding an electrode with high durability.

(ICACC-P090-2023) Modeling Effects of Coulomb Forces in Explosions

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The problem of Coulomb-forces-driven destabilization and explosions found multiple application in conjunction with the development of laser-based technologies (see, for instance, Bulgakova et al. (2005), and references therein.) The phenomenon of our particular interest is the considerable growth of explosion intensity due to the electrostatic forces accompanying penetration of solid projectiles through metals. Such evidence has been noticed in the publication of Marakhtanov and Marakhtanov (2002). In this report we begin with a simple model of a radial Coulomb explosion of an electrically charged spherical gaseous layer. This model permits quite elementary technical analysis. It is particularly important for understanding the mostly robust features of Coulomb explosions. Also, it provides a relevant exact solution for validation and verification purposes when dealing with more sophisticated models.

(ICACC-P092-2023) Development of sustainable CO_2 capture system by seawater electrolysis using optimized hydrogen evolution electrode

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Seawater electrolysis under adequate conditions enables us to extract CO_2 from atmosphere with decomposition of $CaCO_3$. The seawater electrolysis CO_2 capture system would be a promising candidate as effective CO_2 capture and utilization systems since the seawater electrolysis generates pure aragonite fine power and $CaCO_3$ is much used for industrial material such as cement and steel. Dissociation of bicarbonate ion to carbonate is accelerated with hydroxide ion obtained by the electrolysis and $CaCO_3$ precipitates in vicinity of cathode. However, the deposited $CaCO_3$ was easily fixed on the cathode and current density of the electrolysis steeply decreased during the electrolysis. For improving sustainability of capturing CO_2, we investigated hydrogen evolution electrode to generates thin H_2 gas layer on cathode surface to prevent adherence of $CaCO_3$. Hydrogen generation efficiency and time-course change in CO_2 capture rate were investigated using Ni-Fe alloy coating prepared by electroplating. The efficiency was altered by changing alloy compositions. The $CaCO_3$ detachment from the surface of cathode would be much affected by surface microstructure as well as Ni-Fe composition. The formation of H_2 gas layer was much effective for sustainable seawater electrolysis, and the optimization of cathode microstructure would be essential for long-term stable CO_2 capture system.

(ICACC-P093-2023) Catalyst support design for durability improvement in PEMFC

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Cost reduction issue requires research for reducing PGM (Pt group metal) loading contents in a catalyst of a Polymer Electrolyte Membrane Fuel Cell (PEMFC) by using a carbon support having a high surface area. The durability issue of the carbon support demands continuous improvement in corrosion resistance of the support materials. The degradation of the catalyst with the carbon support occurs by the particle coarsening and detaching between

the Pt catalyst and support during practical electrochemical operation conditions such as current load cycling, high current, and start-up/shut-down. Several advanced nanostructured carbon-systems including CNTs, reduced graphene oxide (RGO), and SiC were considered in this study as alternative supports with better tolerance for carbon corrosion than conventional carbon black support. We tried to coat graphene layers on a new support material to improve mechanical strength and durability by using a CVD method. It was necessary to design process conditions including design of source materials to secure the crystallinity and conductivity of the graphene layer. The electrochemical properties of the produced support were evaluated by a CV curve measurement and ECSA, XRD, FE-SEM, eds. The results were compared with the case of the commercial carbon black support.

(ICACC-P095-2023) Intercalative hybridization of CdS nanoparticles and N-doped TiO_2 nanosheets for efficient solar-driven hydrgen production

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We have synthesized an intercalative hybrid photocatalysts of 0D CdS nanoparticles (CdS NPs) and 2D N-doped TiO_2 nanosheets (N-TiO_2 NSs) for efficient hydrogen production under visible light irradiation. For the intercalative hybridization, the aqueous suspension of CdS NPs with the positively charged surface and narrow size distribution in 2-4 nm was slowly dropped into the colloidal suspension of exfoliated N-TiO_2 NSs. A combinatorial analysis of powder X-ray diffraction and transmission electron microscopy clearly demonstrates the formation of well-ordered intercalation structure, indicating the sequent interstratifications of CdS NPs and N-TiO_2 NSs. The photocatalystic test of the hybrid composites was carried out for H_2 production under visible light irradiation ($\lambda \geq 420$ nm). The present hybrid composites have better photocatalytic performance than individuals. Further, this was found to be 1.3 times better than that of the hybrid system of CdS NPs and exfoliated TiO_2 NSs without N-doping. From the obtained results, the present synthetic process guarantees the stability of CdS NPs against photo-dissociation. In this presentation, we will discuss in detail about synthesis procedure, photocatalytic ability, photostability, and photocatalytic mechanism for the present hybrid composites.

(ICACC-P096-2023) Development of Ag-sensitized Eu^{3+}-doped luminescent zeolites for sensing

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Zeolites are crystalline porous materials with significant applications in greenhouse gas capture like CO_2 and NO_x, heavy metals, hydrocarbons and other dangerous contaminants for the environment. Moreover, the porous crystalline nature of zeolites allows a homogeneous dispersion of luminescent species, which could add sensing functionality to the material. Rare earth ions (RE^{3+}) are particularly interesting for this scope because their photoluminescence (PL) emissions have a clear and identifiable spectral shape and long luminescence lifetimes. Among them, Eu^{3+} emission in the red is sensitive to the surrounding environment, potentially responding to the absorption or desorption of contaminant species. In this work, two different commercial zeolites (5A and 13X) were doped by Eu^{3+} ions and Ag aggregates via ion-exchange in solution. Ag can provide a significant increase of the Eu^{3+} PL intensity and a widening of the excitation band, together with an additional blue emission. Optimization of the synthesis process supported by a detailed structural and optical characterization as well as preliminary testing on the sensing capabilities will be presented.

(ICACC-P099-2023) Oyster Mushroom Gills: As decorative art piece

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Ceramic products are often used for cutlery but can be utilized as beautiful decorative art piece. In this study, ceramic decorative piece was conceptualized from oyster mushroom gills and were casted using different baking tools. The texture, structure was crafted in 3D forms in multiple designs which include tableware vase, lamps and decorative pieces. In conclusion, the paper presented innovate ceramic art pieces designed using unconventional methods.

(ICACC-P100-2023) Development of an ML Potential for SiC

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Interatomic potentials govern the physics of atomistic simulations and provide immense computational savings compared to ab initio molecular dynamics (MD). However, these reduced computational requirements may come with noticeable reductions in accuracy over the ab-initio methods. Recently, there have been large advances in machine learning techniques, which have steadily been adopted by the material science community, ushering in the data-driven field of material informatics. Our goal is to develop a machine-learned interatomic potential (MLIP) for the silicon carbide (SiC) system, intended for simulating extreme environments (e.g., shock loading). We use an artificial neural network trained on ab initio calculations to fit an MLIP for the SiC system. The training data includes multiple polytypes as we aim to capture phase transformations observed under extreme loading conditions approaching ab initio accuracy with lower computational costs compared to current potentials. While MLIPs may not be as physically interpretable as their traditional counterparts, we expect that their flexible form will enable the identification of connections across length scales that are otherwise missed in more traditional approaches. Efforts for SiC MLIP development will be discussed, along with network architecture optimization.

(ICACC-P101-2023) Effect of Plasma Nitiriding & Oxynitriding Temperature on Wear and Corrosion Resistance of the AISI 4140 Steel

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This work presents the effect of nitriding/oxynitriding process at different temperature on the corrosion resistance and wear behavior of the quenching-and-tempering-treated AISI 4140 steel. Prior to conduct the oxynitriding process, the AISI 4140 was plasma nitrided at variable process temperature to form optimal compound layer thickness. Subsequently, the plasma oxynitriding was performed on the plasma nitrided AISI 4140 steel at different temperature under distilled water vapor atmosphere, which results in the formation of oxide thin layer on the nitrided AISI 4140 steel. Microstructure and phases of the plasma oxynitrided samples are investigated, indicating that phase formation of the oxide layer is strongly dependent on processing temperature during plasma oxynitriding: formation of Fe_3O_4 is preferred over Fe_2O_3 at lower processing temperature. Also, it is believed that ε-$Fe_{2\text{-}3}N$ phase formed by nitriding process plays an important role to promote the formation of Fe_3O_4 phase during plasma oxynitriding.

(ICACC-P102-2023) Wear of PEEK/MoAlB Composites in Ethanol and Alkane/Ethanol mixtures

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Polyether ether ketone (PEEK) is a polymer thermoplastic material that is currently used in applications including biomaterials, and in bearing/pump components that may contain ethanol which leads to reduced part life and increased maintenance cost. Using a ternary ceramic material known as MoAlB, the PEEK material was made into a polymer matrix composite. Tribological testing was performed on a PEEK/10% MoAlB polymer matrix composite using a high frequency reciprocating rig (HFRR) with 52100 steel counter-body and alkane/ethanol fuel lubrication mixtures. Characterization was performed using sliding friction, wear through mass loss and volumetric wear, elemental and chemical behavior using Scanning Electron Microscopy and x-ray and infrared spectroscopies. It was found that wear and mass loss in 100% ethanol was very low, followed closely by 90% ethanol in decane and the highest wear was seen in 10% ethanol with decane. Further testing is planned for more harsh parameters, longer testing period, and completing equivalent testing on pure PEEK material.

(ICACC-P103-2023) Exploration of PEEK-based Composites for Multifunctional Applications

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In this poster presentation, we will present some of the recent studies in the synthesis and characterization of PEEK-based composites. The team will review different types of manufacturing processes of PEEK-based composites. The microstructure, mechanical and triboactive behavior of these composites will be presented. A systematic study of tribological behavior of these composites in different solvents will be presented.

(ICACC-P104-2023) Ultrafast Laser Bessel Beam Cutting of Glass

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Ten unique compositions of optically transparent ceramics were tested for single-shot and multi-shot cutting with ultrafast laser pulses shaped into a high aspect ratio Bessel beam by way of an axicon lens. Cutting is performed on sapphire, indium tin oxide (ITO), quartz, X-cut lithium niobate (XLN), Z-cut lithium niobate (ZLN), and several different compositions of zinc barium borate (ZBB) glass. Characterization of cutting energies, cut roughness, and cut radius was performed with optical profilometery and spectroscopy.

(ICACC-P105-2023) Understanding the Effects of Polar Topological Phases in Epitaxial Oxide Super Lattices on Phonon Scattering Rate

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1. University of Virginia, Materials Science and Engineering, USA
2. UCBerkeley, MSE/Physics, USA

Epitaxial oxide superlattices and heterostructures give rise to spatially complex topological configuration such as polar skyrmions and vortices. Some of the emergent properties of these stable quasi-particle objects in epitaxially grown (PbTiO3)n/(SrTiO3)n (PTO/STO) superlattices have been extensively studied. However, a detailed study of the effects of these topological phases on the scattering mechanisms and resulting thermal properties was unexplored. Since the type of topology introduces different long to medium range order, in this study we aim to investigate phonon relaxation time as one of the key parameters involved in both thermal transport and coherent phonon phenomena. We perform Time-domain Brillouin scattering (TDBS), an ultrafast laser spectroscopy technique, on the PTO/STO superlattices and trilayers to interrogate the underlying changes in phonon dynamics. The displacements and the length scales associated with the existing topological phases in these superlattices change phonon lifetimes where all of these phases have collective topological modes. In other words, we reveal the effect of (anti) ferroelectric displacements near phase boundaries on phonon dampening due to different degrees of frustrated acoustic phonons.

(ICACC-P107-2023) Enhanced piezoelectric properties of tungsten-modified $Bi_5Ti_3FeO_{15}$ ceramics for high-temperature piezoelectric applications

Q. Wang*[1]; E. Liang[1]; C. Wang[1]

1. Shandong University, School of Physics, State Key Laboratory of Crystal Materials, China

$Bi_5Ti_3FeO_{15}$ (BTF), a multiferroic four-layer Aurivillius phase compound, has received much attention in recent years due to its ferroelectric and magnetic properties, as well as magnetoelectric coupling behavior. Despite numerous researches have been done on BTF, its piezoelectric properties are still insufficiently investigated. In this work, the dielectric, piezoelectric, and electrical properties of tungsten-modified BTF with compositions of $Bi_5Ti_3Fe_{1-x}W_xO_{15}$ (BTF-100xW) have been investigated in detail. The results indicate that the substitution of W^{6+} for Fe^{3+} effectively increases the resistivity and electrical homogeneity, and reduces the dielectric loss tanδ at high temperatures, resulting in enhanced piezoelectric properties. Remarkably, a high piezoelectric constant d_{33} of 23 pC/N, which is three times that of pure BTF, and a high Curie temperature T_C of 794 °C are achieved in BTF-3W. Meanwhile, BTF-3W also exhibits excellent thermal stabilities of electrical properties and the electromechanical coupling characteristics up to 400 °C. These results demonstrate that tungsten-modified BTF are promising candidate for the applications of high-temperature piezoelectric devices.

(ICACC-P108-2023) Enhanced energy storage performance of BNT-based lead-free relaxor ferroelectric ceramics

J. Li[1]; Q. Wang*[1]; C. Wang[1]

1. Shandong University, School of Physics, State Key Laboratory of Crystal Materials, China

The development of dielectric ceramic capacitors with excellent energy storage performance plays a major role in meeting the requirements of miniaturization and integration for advanced pulsed power capacitors. $Bi_{0.5}Na_{0.5}TiO_3$ (BNT)-based ceramics are considered as one of the most promising energy storage materials owing to a large spontaneous polarization (~40 μC/cm^2). Unfortunately, the high remnant polarization (P_r), large coercive electric field (E_c) and low breakdown strength (BDS) severely limit the enhancement of recoverable energy storage density (W_{rec}) and energy storage efficiency (h). Herein, BNT-xCa$(Al_{0.5}Ta_{0.5})O_3$ (BNT-xCAT) ceramics were prepared through traditional solid-state reaction method, and the crystal phase, microstructure, and electric properties were investigated in details. The results showed that CAT-doped BNT can effectively enhance the BDS, and improve the relaxor behavior on account of the formation of polar nanoregions (PNRs). Therefore, a large W_{rec} of 3.95 J/cm^3 and a high h of 76.42%, as well as excellent temperature and frequency stabilities, and fast discharge speed ($\tau_{0.9}$ = 78 ns) were simultaneously achieved in the BNT-0.08CAT. All these demonstrate that this system has potential application in pulse power system.

(ICACC-P109-2023) Data-driven strategies to create artificial intelligence model with humanlike image discrimination: Example of toughened silicon nitride ceramics

R. Furushima*[1]; Y. Maruyama[1]; Y. Nakashima[1]; M. Ngo[1]; T. Ohji[1]; M. Fukushima[1]

1. National Institute of Advanced Science and Technology, Japan

Fracture toughness of silicon nitride (Si_3N_4) ceramics was evaluated directly from their microstructures via deep learning using a trained artificial intelligence (AI) model. Totally 156 data sets containing microstructural images and relative densities were prepared from 45 types of Si_3N_4 samples as input feature quantities (IFQs) and were correlated to the fracture toughness as an objective variable. The data sets were divided into two groups. One was used for training, resulting in creation of regression models for two kinds of IFQs: the microstructures only and combination of the microstructures and the relative densities. The other group was used for testing validity of the created models. As results, the determination coefficient was approximately 0.8 even when using only the microstructures as the IFQs and was further improved when adding the relative densities. It was revealed that fracture toughness of Si_3N_4 ceramics was well evaluated from their microstructures. Furthermore, gradient-weighted class activation mapping as visualization tool was also conducted to help us to understand which part the trained AI model focused. The AI well recognized the difference between grains and secondary phases, which was also one of the indices for image discrimination by human. Therefore, the trained AI model executed a certain kind of humanlike image discrimination, which contributed to high prediction accuracy of fracture toughness in the Si_3N_4 ceramics.

Thursday, January 26, 2023

Emerging Materials and Sustainable Manufacturing Technologies in a Global Landscape: Symposium in Honor of Dr. Tatsuki Ohji

Tatsuki Ohji Honorary Symposium V

Room: Coquina Salon D (North Tower)
Session Chairs: Hisayuki Suematsu, Nagaoka University of Technology; Jerzy Lis, AGH University of Science and Technology

8:30 AM

(ICACC-HS-028-2023) Synthesis of β-MoO_3 nanosized powders by pulsed wire discharge (Invited)

H. Suematsu*[1]; N. M. Chu[2]; T. Kitagawa[1]; T. Do[1]; T. Nakayama[1]; K. Niihara[1]

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

A γ-ray emitting isotope of ^{99m}Tc has been used in various radiopharmacies and is produced from its mother nuclide of ^{99}Mo. ^{99}Mo has been prepared from highly enriched uranium (HEU) spent fuel in nuclear reactors. Because of the nuclear security risks of HEU, a new preparation method using a nuclear reaction by neutron irradiation of α-$^{98}MoO_3$ targets has been investigated. We have found that, instead of conventional α-MoO_3, β-MoO_3 shows superior ^{99}Mo extraction characteristics to water because of the larger spacing between Mo-O layers. For this application, synthesis of β-MoO_3 nanosized powders have been attempted. A Mo wire with a diameter of 0.25 mm and a length of 25 mm was placed between electrodes in a chamber filled with O_2-N_2 mixed gas at 25-100 kPa at a constant O_2 partial pressure of 25 kPa. The electrodes were connected to capacitors of 30 μF charged at 3-6 kV. The pulsed large current evaporated the Mo wire within a few ms. The Mo vapor was cooled by and reacted with the O_2-N_2 mixed gas to form β-MoO_3 particles. From X-ray diffraction, with the decrease in the total pressure, the β-MoO_3 content was increased and the particle size was decreased. It was thought that the low total pressure condition allowed the Mo vapor to expand largely and then cooled quickly. Since β-MoO_3 is stable below 450 °C, this is the reason to yield the high β-MoO_3 content at the low total pressure.

8:50 AM

(ICACC-HS-029-2023) Tailoring Microstructure of Ceramics by colloidal processing and external fields (Invited)

T. S. Suzuki*[1]

1. National Institute for Materials Science, Ceramics Processing Group, Japan

The physical properties of materials depend on the crystal axis. Design of the crystallographic orientation in ceramics is expected to improve their functional and mechanical properties. Many researchers have reported that the textured ceramics were produced by the Templated Grain Growth method, hot forging, one directional extrusion, etc. On the other hand, we have achieved the control of orientation even in diamagnetic ceramics by a strong magnetic field. The particles become rotated to an angle that minimizes the system energy. The driving force for the magnetic alignment is a magnetic torque generated from the interaction between the magnetic anisotropy of crystals and the applied magnetic field, thus easy magnetization axis can be aligned parallel to the magnetic field and one-dimensional orientation can be achieved. If the orientation axes of platelets aligned due to a geometrical effect are different from that aligned by a magnetic field, the multidimensional orientation can be controlled, when the gravity force or the shear stress for aligning the anisometric particles was combined with a magnetic field as the other force. Furthermore, when we align the direction of the pore-forming agents having a fiber shape such as short nylon, we can control to align the tubal pores.

9:10 AM

(ICACC-HS-031-2023) Development of Materials Engineering at AGH University of Science and Technology (Invited)

J. Lis*[1]; D. B. Kata[1]; R. Wisniowski[1]

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

Strategies and progress in composite materials, prepared by combination of rapid synthesis and 3D shaping technologies from a scientific approach to a promising industrial applications are discussed. It responds and aims to contribute current needs in production of very effective parts for power plant industry. The systematic review of different forms of metal – ceramic composites shows that grain boundaries control final properties of their high temperature applications. To tailor of phases appeared at grain boundaries the combination of 3D shaping by selective laser sintering (SLS) and rapid solidification is showed. SLS is a method that uses energy delivered to material in form of laser beam in order to melt and rapid solidify new structures shaped in precise and sophisticated geometrical objects. It is demonstrated in ceramic-metal composite explored in: Inconel 625-WC and Inconel 625-NbC systems. Thus technology of preparation new class metal – ceramic polycrystals is manifested. Finally the room and high temperature applications of these composites are exanimated to demonstrate their applicability.

10:00 AM

(ICACC-HS-032-2023) Additive Manufacturing of structural ceramics (Invited)

D. B. Kata*[1]; J. Lis[1]; R. Wisniowski[1]

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

Additive Manufacturing (AM) is widely known and appreciated due to its flexible and customized manufacturing capabilities. Today, the AM technology has contributed to almost all engineering areas that include mechanical, materials, industrial, aerospace, electrical and most recently biomedical engineering. The applicability of AM is shown by appropriate assessment based on the two material systems, namely: (1) AlN-graphene nanocmposite; (2) Inconel 625 – WC composite. The anisotropic AlN-graphene nanocomposites were shaped by AM technology. The structures, morphologies, and microstructures of those hybrids were examined by X-ray diffraction, SEM and TEM methods. It is showed that additive manufacturing significantly influenced on thermal anisotropy and microstructure appearance. The results were correlated with thermal conductivity of the samples carried out by the laser pulse method. Chemically and structurally homogenous Inconel 625 – WC composite coatings were prepared by Additive Manufacturing. Because of chemical composition of Inconel 625 superalloy, secondary carbides of WC, W_2C, NbC, (NbW)C, $W_6C_{2.54}$ and $(W,Cr,Ni)_{23}C_6$ were detected in the intergranular spaces by XRD analysis. They appeared in form of eutectic with typical fishbone-like structure in samples containing increased amount of WC - 30 wt %. High cooling rate during AM was the reason of fine microstructure in produced material.

10:30 AM

(ICACC-HS-033-2023) Bonding and densification mechanism between particles in the RTIC phenomenon of fine ceramic particles (Invited)

J. Akedo*[1]

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

Dense and highly adhesive lamination of functional ceramic materials is the key to the realization of electronic devices, energy, and environment-related devices. The room-temperature impact consolidation phenomenon (RTIC) of ceramic particles (a phenomenon in which ceramic particles solidify at room temperature without sintering), which was clarified by research on the aerosol deposition method, is characterized not only by the fact that it is a room-temperature process, but also by the fact that it is denser than the conventional thermal spraying method. It has a very important meaning as a principle for obtaining a ceramic coating with high adhesion. Until now, the densification mechanism of ceramic coatings by the AD method has been explained by the plastic deformation of colliding fine particles. However, there are many unclear points about the mechanism of plastic deformation and interparticle bonding of ceramic fine particles at room temperature. In this presentation, I will discuss the mechanism of plastic deformation of ceramic fine particles at room temperature based on reports on elucidation of the mechanism of the AD method, which has recently begun to be studied in various places, and on the results of examinations of reflection film formation by the AD method and compression fracture tests of fine particles.

10:50 AM

(ICACC-HS-034-2023) Unique Route to Grow Single-Crystals of NonstoichiometricTerbium Oxides and Alumina Electrochemically (Invited)

N. Imanaka*[1]

1. Osaka University, Applied Chesistry, Japan

The nonstoichiometric phases of fluorite-related rare earth oxides have been regarded difficult to grow in a single crystal form because they possess high melting points ans are usually obtained as mixtures of some R_nO_{2n-2m} (n) 7, 9, 11, 12, 16, 19, 24, 29, 39, 40, 48, 62, and 88; m) 1, 2, 3, 4, 6, and 8) phases. Here, our unique dc electrolysis method can be simply applied to grow a specific nonstoichiometric phases in a single crystal form selectively at moderate temperatures at 800 or 900 °C, which is fur below the melting point. By dc electrolysis of trivalent terbium ion conducting $Tb_2(MoO_4)_3$ solid, single crystals of nonstoichiometric rare earth oxide phases such as $Tb_{16}O_{30}$ ($Tb_{O1.875}$), $Tb_{24}O_{44}$ ($TbO_{1.833}$), and $Tb_{11}O_{20}$ ($TbO_{1.818}$) were artificially grown. The selective growth of a specific phase has been successfully realized by controlling the oxygen partial pressure, the electrolysis temperature, and also the voltage applied during the electrolysis. The growth of high-quality single crystals has been evidenced by the selected area electron diffraction analysis method. Similarly, the presently developed electrochemical method can be simply applicable at moderate temperatures around 900 °C for the single-crystal growth of alumina, and, therefore, it becomes possible to artificially grow such an intermediate phase of delta-alumina in a single-crystal form.

S1: Mechanical Behavior and Performance of Ceramics & Composites

Fracture Mechanics, Failure Analysis, and Fractography

Room: Ballroom 5 (South Tower)
Session Chairs: Jonathan Salem, NASA Glenn Research Center; Kevin Strong, Sandia National Laboratories

8:30 AM

(ICACC-S1-042-2023) A Meso-Scale LEFM Approach to Understanding CMC Interlaminar Strength (Invited)

J. D. Baker*[1]

1. Rolls-Royce Corporation, USA

Damage initiation and propagation in CMCs is driven by smaller scale features. This body of work applied linear elastic fracture mechanics to understand the interlaminar failure process at a meso-scale (fiber tows and matrix). The fiber interphase coating is designed to toughen the material yet can be treated as initial flaws. The flatwise tensile (FWT) test is modelled as an approximately isotropic material containing multiple cracks that span a fiber tow. Cracks are explicitly modelled in finite element analysis (FEA) and the non-dimensional stress intensity factors (SIFs) were extracted using local crack tip displacements. Interlaminar strength is then directly related to the SIF magnitudes. The effects of various fiber tow nesting positions are explored to understand expected material test scatter. The model correctly identified the first-ply bias of the FWT test and found a feasible modification to achieve more accurate interlaminar tensile (ILT) strength.

9:00 AM

(ICACC-S1-043-2023) Statistical modeling of mechanical lifetime in glass and ceramic (Invited)

S. Grutzik*[1]; T. Diebold[2]; K. T. Strong[3]

1. Sandia National Laboratories, Materials and Failure Modeling, USA
2. Sandia National Laboratories, Material Mechanics and Tribology, USA
3. Sandia National Laboratories, Material Mechanics and Triboloty, USA

The stochastic nature of brittle material reliability is commonly characterized using Weibull statistics. In addition, lifetime is often estimated in terms of subcritical crack growth. In practical application many restrictive assumptions are often employed, such as the assumption that subcritical flaw population evolution does not change the strength distribution or stress state does not change in time. In this work, the time to failure under static four-point bending was measured for barium titanate ceramic and Schott S8061 glass in a controlled atmosphere at 50% and 95% relative humidity. Measured failure times are compared to a hazard function (or rate of failure) calculated using beginning-of-life Weibull strength statistics and Paris Law subcritical crack growth parameters in a unified, time-dependent probabilistic manner. The effects of crack tip stress relaxation and strategies to deal with non-constant stress are also discussed.

9:30 AM

(ICACC-S1-044-2023) Finding the "Real" Fracture Toughness of Glass: Draft ASTM Standards Using CNSB, DCB, DT, SEPB Test Methods

M. G. Jenkins*[1]; J. Salem[2]; J. Westbrook[3]; E. Aaldenberg[3]; B. M. Sundaram[3]; G. D. Quinn[4]

1. Bothell Engineering and Science Technologies, USA
2. NASA Glenn Research Center, Materials and Structures, USA
3. Corning Incorporated, USA
4. National Institute of Standards and technology, Materials Measuremenet Sciences Division, USA

Finding the "real" fracture toughness of a material requires proper procedures and strict attention to detail exemplified by such ASTM International standards as E1820 for metals and C1421 for ceramics. A key requirement is an atomistically sharp pre-crack in the test specimen. In metals, the precrack is initiated by cyclic fatigue. In ceramics, pop-in precracking or hardness test indentation precracking are used. Another requirement is a fracture test geometry with a valid stress intensity factor (SIF). For both metals and ceramics, single-edge-cracked flexure specimens with well-known SIFs are used. Glasses, while brittle, may not be as easily tested using ASTM C1421. One difficulty is the availability of glasses as thin plates. Interferences such as slow crack growth in glass need consideration. Therefore, several draft ASTM standards are being jointly developed by Committee C14 on Glass & Glass Products and Committee C28 on Advanced Ceramics for fracture toughness testing of glasses using CNSB (chevron notch short bar), DCB (double cantilever beam), DT (double torsion) and SEPB (single edge precracked beam) test configurations. The draft standards address the following -- test specimen geometries/preparation, test fixtures, test equipment, interferences, testing modes/procedures, data collection, calculations, reporting requirements, precision/bias.

10:10 AM

(ICACC-S1-045-2023) Alumina toughened zirconia composites with time-dependent strength improvement

Z. Pedzich*[1]; A. Wojteczko[1]; M. Grabowy[2]

1. AGH University of Science and Technology, Department of Ceramics and Refractory Materials, Poland
2. CEREL, Ceramic Department, Poland

Tetragonal zirconia is well commercialised but still very promising ceramic material due to its relatively high strength and fracture toughness. Usually used stress tests are rather fast and they can not deliver information about possible changes of material's strength during long-lasting exploitation. Long-term loading of tetragonal zirconia components in humid environment (in air or water) leads to decrease in strength due to stress corrosion. This phenomenon is known as the subcritical cracking. With the above limitation in mind, an attempt was made to create composite in order to reduce the effect of stress corrosion cracking. The ATZ (alumina toughened zirconia) type composites were prepared using reactive sintering process utylizing different zirconia powders. Materials obtained by means of such procedure revealed very fine microstructure and had extremaly high fracture toughness on the level of 12 $\text{MPam}^{0.5}$. Subcritical crack propagation phenomenon in mentioned materials were tested using constat stress rate test. Calculations were made in order to estimate the survival time of the material working under loads in air only for materials where change of strength for different stress rates was observed, which indicated significant limitation of subcritical crack propagation phenomenon occurrence.

10:30 AM

(ICACC-S1-046-2023) 3D Mapping of Glass Indentation Stress Fields

A. J. Bellafatto*[1]; I. Reimanis[2]

1. Colorado School of Mines, Materials Science, USA
2. Colorado School of Mines, USA

Crack initiation associated with contact damage is of great interest for glass that experiences mechanical loads. The stress field under an indent is dependent on a variety of factors, including the glass type, indenter acuity, amount and type of plasticity, and the presence of cracks. In the present work, Raman mapping is used to describe the stress state in silica and soda lime silicate after indentation. Details of the measurement, including stress calibration and spatial resolution are described.

10:50 AM

(ICACC-S1-047-2023) Evaluating Mechanical Properties of FAST-processed Hard Ceramics via Multiscale Indentation and Holistic Elastoplastic-Fracture Analysis

D. E. Wolfe*[1]; C. DeSalle[1]; C. Ryan[1]; R. Slapikas[1]; R. Sweny[1]; R. Crealese[1]; P. Kolonin[1]; S. Stepanoff[1]; S. Divilov[2]; H. Eckert[2]; C. Oses[2]; M. Esters[2]; D. Brenner[3]; W. Fahrenholtz[4]; J. Maria[1]; C. Toher[5]; E. Zurek[6]; S. Curtarolo[7]

1. Pennsylvania State University, USA
2. Duke University, USA
3. NC State University, USA
4. Missouri University of Science & Technology, Dept. of Materials Science and Engineering, USA
5. University of Texas at Dallas, USA
6. State University of New York at Buffalo, USA
7. Duke University, USA

Materials with superior mechanical properties in extreme conditions have attracted substantial attention in the development of hard materials for manufacturing, aerospace, energy, and defense applications. To improve the wear, abrasion, and erosion resistance of these materials while maintaining thermomechanical properties, composites and ceramics have been developed as super-hard materials. Field-Assisted Sintering Technology (FAST) has been instrumental in advancing the fabrication, joining, and repair of these hard ceramic materials without the need for soft or weakening sintering aids/binders. However, characterizing the mechanical properties of these materials can be difficult for facilitating scalable R&D efforts. This study presents the development of novel mechanical property characterization techniques via multiscale indentation by applying coupled microindentation and nanoindentation on hard FAST-processed materials. In addition to deconvoluting multiscale hardness and deformation effects at various length scales, this methodology facilitates insight into the elastoplastic and fracture responses through loading curve analysis, in-situ scanning probe microscopy, and indentation fracture resistance (IFR) analysis.

11:10 AM

(ICACC-S1-048-2023) Microstructural Effects on Polycrystalline Ceramic Fracture as Revealed via Synchrotron Radiation

S. F. Gorske*[1]; M. Ramesh[2]; J. Park[3]; P. Kenesei[3]; H. Sharma[4]; J. Almer[3]; P. Voorhees[2]; K. Faber[1]

1. California Institute of Technology, Materials Science, USA
2. Northwestern University, Materials Science and Engineering, USA
3. Argonne National Lab, Materials Physics and Engineering, USA
4. Argonne National Lab, Computational X-ray Science, USA

In anisotropic polycrystalline materials, microstructure is a key determinant in crack propagation. As opposed to traditional fractography methods, which rely on post mortem analysis of fracture surfaces to identify characteristics of the crack path, synchrotron radiation reveals in-depth information as a crack grows. By combining the methods of micro-computed X-ray tomography, high-energy diffraction microscopy, and mechanical testing, the shape and location of a growing crack front can be monitored in real time, along with the shape, size, and orientation of the grain structure around the crack. We present the first use of these combined methods applied to a polycrystalline ceramic material, aluminum oxynitride (AlON). The AlON samples were machined into a double-cleavage drilled compression sample, with which stable crack growth under a compressive, displacement-controlled load was obtained. We discovered that the large grains (on the order of 100 μm) provided barriers to crack growth, with the crack growing intergranularly around favorably-oriented grains before impinging at grains through which the crack had to gain enough stress energy density to move. This process thus elucidates the energy considerations of an in-situ crack interacting with regions of changing fracture toughness and elastic properties, which is key information for future phase-field modeling efforts.

11:30 AM

(ICACC-S1-049-2023) Damage Control Measures in Composites: BVID Damage Progression

K. Jribi*[1]; J. Gosse[2]; D. Neill[2]; A. Mello[1]

1. Embry-Riddle Aeronautical University, Aerospace Engineering, USA
2. Computational Engineering Software, LLC, USA

A major challenge with composites is the fact that there is still no accepted standard to define the damage and its propagation compared to well-studied metallic structures. This research follows the guidelines of national Agencies with the initiative to create an efficient simulation-based approach that would predict the potential initiation of damage propagation in a composite structure. The predictive analysis will incorporate the Onset method which is a physics-based methodology for assessing the onset of irreversible deformation. The Onset methodology utilizes the critical dilatation and distortion of the constituent materials of the composite material system. The assessment follows the micromechanical de-homogenization of the deformed composite material system. As a result, there is a limited dependency on parameters extracted from representative experiments. The approach consists of extracting the critical dilatation and distortion for each constituent material from tested coupons, developing a sequence of statically indented coupon tests to simulate low-velocity impact damage, establishing the catastrophic load environments for the indented coupons subject to in-plane uniaxial compression, and numerically analyzing the indented coupons using the critical dilatation and distortion of the constituent materials to numerically derive the critical in-plane compression loads to the critical values of from test.

11:50 AM

(ICACC-S1-050-2023) Soldered Tensile Failure Stress of Ceramics in Multilayered Ceramic Capacitors

T. J. Rogers*[1]; K. T. Strong[2]; J. Yang[2]; S. Grutzik[3]; R. Wheeling[1]

1. Sandia National Laboratories, USA
2. Sandia National Laboratories, Material Mechanics and Triboloty, USA
3. Sandia National Laboratories, Materials and Failure Modeling, USA

Weibull strength size-scaling can be successfully used to predict the strength of a ceramic part subjected to tensile stress from a standard laboratory flexure test. However, it is unknown whether this can be applied when the ceramic has a complex thermal residual stress field from a joining process such as soldering. The ceramic dielectric from multilayered ceramic capacitors was chosen for this study as it is a very common commercial ceramic that is soldered. Tape cast coupons were acquired from the manufacturer and biaxial flexure was used to measure the tensile strength using a ring-on-ring (RoR) fixture. Additional samples were fabricated with a soldered copper disc in the center of the coupon. The area with the soldered disc was place on the tensile side of the RoR fixture to measure the solder strength. Finite element analysis was used to simulate the solder stress of the ceramic coupons. The simulated ceramic was then placed in biaxial stress the same as the experimental fixture. A probability of failure was predicted using the Weibull parameters from the unsoldered ceramic strength tests. The experimental and computation results are compared.

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

Environmental Barrier Coatings II

Room: Flagler C (South Tower)
Session Chairs: Bryan Harder, NASA Glenn Research Center; Katherine Faber, California Institute of Technology

8:40 AM

(ICACC-S2-046-2023) A multiscale 3D investigation approach for characterizing damage in coatings and woven ceramic matrix composites

H. Bale*[1]

1. Carl Zeiss Research Microscopy Solutions, USA

Textile composites with woven ceramic fiber tows in a ceramic matrix are important for many structural applications and represent a new class of integrally woven ceramic-matrix-composites for high-temperature applications. For high-performance and reliability, a key issue is irregularities and defects in the textile reinforcement, which compromise strength and life. Environmental barrier coatings further expand the capabilities for corrosive environments. Through complex microstructural layouts derived by weaving bundles of fibers, including functional coatings on the fibers and incorporating several strategies to enhance the toughness of bulk composite excellent properties can be achieved. However understanding damage in such complex architectures has remained extremely challenging. Recently, high resolution X-ray imaging capabilities that were previously only available at synchrotrons have been transferred to analogous lab-based instruments, enabling non-destructive detailed characterization including in situ and 4D imaging experiments, albeit at a different time scale than synchrotrons. We present a unique characterization workflow in EBC/TBC coatings and bulk CMC's using correlative X-ray microscopy aided with deep-learning based algorithms for data-reconstruction and segmentation, combined with FIB-SEM and electron microscopy to characterize cracks networks.

9:00 AM

(ICACC-S2-047-2023) Coating Processing and Materials Testing with the Plasma Spray- Physical Vapor Deposition (PS-PVD) Facility at NASA Glenn Research Center

B. J. Harder*[1]; L. C. Hoffman[1]; M. Kulis[1]; K. Lee[2]; M. J. Presby[1]

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

The Plasma Spray-Physical Vapor Deposition (PS-PVD) facility at NASA Glenn Research Center was established in 2009 for the purpose of generating coatings with a variety of architectures and applications for the thermal and environmental protection of hot section components for gas turbine engines. While the PS-PVD system bridges the architectural gap between conventional thermal spray and vapor phase techniques, it has several advantages as well as new challenges in the processing of thermal and environmental barrier coatings (T/EBCs). In this work, components of T/EBC systems for use above 1482°C that were processed with PS-PVD will be discussed regarding composition, architecture, and durability. In addition, initial studies utilizing the PS-PVD system as a high heat flux torch for materials testing will be discussed. Plasma temperature, heat flux measurements and ionized species will be discussed as a function of plasma conditions and the resulting potential degradation for materials in high-speed aero environments.

9:20 AM

(ICACC-S2-048-2023) Plasma Sprayed Disilicate-based Environmental Barrier Coatings: a comparative study

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

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

Disilicate based environmental barrier coatings (EBCs) are the current best option to protect ceramic matrix composites (CMC) components that are used in current and forthcoming power generation and aircraft/spacecraft turbine engines from the combustion atmosphere generated in operation. These materials are usually deposited by atmospheric plasma spray (APS) in the form of coatings that have a high amorphous character and a shift from the original chemical composition caused by the spraying conditions. In this work is presented a comparative study of three disilicate materials; $Y_2Si_2O_7$, $Yb_2Si_2O_7$ and $YYbSi_2O_7$ deposited by APS in the form of coatings on SiC substrates previously coated with a Si bond coat. Their amorphous character, compositional changes, phase and microstructure evolution with thermal treatments are thoroughly discussed Their behavior against burner rig thermal cycling is presented and correlated to the nature of each coating

9:40 AM

(ICACC-S2-049-2023) Varying processing parameters in the development of slurry-based oxide bond coat for environmental barrier coatings

R. I. Webster*[1]; K. Lee[1]; B. J. Puleo[1]

1. NASA Glenn Research Center, USA

Silicon carbide (SiC)-based ceramic matrix composites (CMCs) are replacing nickel-base superalloys in some hot-section aircraft engine materials due to their lower density and higher working temperature. CMCs require an environmental barrier coating (EBC) for protection from corrosive combustion species. Current-generation EBCs consist of a rare earth silicate topcoat and a silicon bond coat; however, the relatively low melting point of silicon (1414°C) limits the upper use temperature of these coatings. To extend the capability of next-generation EBCs, an oxide-based bond coat capable of withstanding temperatures of up to 1480°C has been developed at NASA Glenn Research Center. In this study, the oxide-based bond coat was applied to monolithic SiC via a slurry coating process. Parameters including slurry particle size and viscosity were varied, and coating performance was evaluated after steam cycling at 1480°C. The effect of including different rare earth constituents in the bond coat was also explored.

10:20 AM

(ICACC-S2-050-2023) Effect of Cycle Frequency on Steam Oxidation of Environmental Barrier Coatings

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1. Oak Ridge National Lab, USA

Rare earth silicate environmental barrier coatings are required for protection of SiC components from the combustion environment in turbine applications. A primary failure mode for EBCs is the oxidation of the silicon bond coat, where SiO_2 formation at the interface can eventually result in EBC delamination. While aerospace turbines may be better replicated in a laboratory with rapid furnace cycles, land-based gas turbines for power generation may operate for longer durations in baseload before cooldown. In this work, the steam oxidation behavior of EBCs were analyzed at 1350°C for both 1-h and 100-h cycles to assess EBC durability as a function of cycle time. Results were utilized to develop an updated lifetime prediction model for EBCs. This research was sponsored by the U. S. Department of Energy, Office of Fossil Energy and Carbon Management, Advanced Turbine Program.

10:40 AM

(ICACC-S2-051-2023) In-situ Synchrotron Studies of Environmental Barrier Coatings Under Steam and Air Conditions at Elevated Temperatures

B. Herren[1]; C. Chuang[2]; J. Almer[2]; K. Lee[3]; K. Faber*[1]

1. California Institute of Technology, USA
2. Argonne National Laboratory, Advanced Photon Source, USA
3. NASA Glenn Research Center, USA

It is well established that water vapor in combustion atmospheres is deleterious to passivating SiO_2 scales on SiC/SiC ceramic matrix composites (CMCs). Although environmental barrier coatings (EBCs) are known to provide some level of protection against water vapor recession in such systems, diffusion through state-of-the-art EBC systems is still known to result in oxidation of the EBC bond coat and CMC, and potentially lead to failure. In order to study this phenomenon using synchrotron radiation, a custom induction furnace was designed for use with in-situ steam and dry exposures at temperatures as high as 1320°C. We describe here the model of the custom furnace for synchrotron use. Further, we demonstrate the first examples of EBC studies with the environmentally controlled furnace. These include a comparison of dry air vs. steam environments on thermally grown oxides in the plasma-sprayed $Yb_2Si_2O_7$/Si/SiC/SiC system. In-situ synchrotron studies were also conducted to reveal crystallization of $Yb_2Si_2O_7$ plasma spray powders and measure thermal expansion coefficients of doped-$Yb_2Si_2O_7$ plasma spray powders. The latter work demonstrates the ease of rapid temperature change in the induction furnace.

11:00 AM

(ICACC-S2-052-2023) Temperature Dependent Thermal Expansion Anisotropy of Rare Earth Disilicates Via Synchrotron X-Ray Scattering

A. Salanova*[1]; I. Brummel[1]; E. J. Opila[1]; J. Ihlefeld[1]

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

In this work, the temperature dependent anisotropic thermal expansion behavior of rare earth disilicates for thermal/environmental barrier coating applications will be presented. Hot stage X-ray diffraction experiments were preformed using synchrotron radiation and in-lab instrumentation. Lattice parameters and thermal expansion were then determined using the X-ray scattering data, showing the direction of maximum thermal expansion varies with temperature. The shifts correspond to changes in eigenvectors with respect to temperature, manifesting as distinctions in shear components in the tensor and lattice parameter changes. The composition of the rare earth disilicate changes the anisotropic behavior, leading to mixed and high entropy (HE) disilicate compositions with unique thermal expansions. Initial efforts to elucidate the phenomena leading to these behaviors using pair distribution function analysis will be presented.

11:20 AM

(ICACC-S2-053-2023) Foreign Object Damage (FOD) in Environmental Barrier Coatings (EBCs)

L. C. Hoffman*[2]; M. J. Presby[1]; J. L. Stokes[1]; B. J. Harder[1]

1. NASA Glenn Research Center, Environmental Effects and Coatings Branch, USA
2. HX5, LLC., USA

Foreign object damage (FOD) is one of the key damage/failure modes for environmental barrier coatings (EBCs) operating in gas turbine engines. While FOD has been extensively investigated for ceramic matrix composites (CMCs), there is limited research and understanding on the FOD response of EBCs. This work explores the FOD behavior of NASA's Generation II Ytterbium Disilicate EBC deposited on monolithic SiC Hexoloy SA. FOD testing was conducted at room temperature using a 1.59-mm steel ball projectile with particle velocities ranging from 50 – 300-m/s at normal incidence angle. The impact damage was characterized by optical profilometry and scanning electron microscopy (SEM) of the cross-sections. The effect of environmental exposure (steam cycling) on the FOD resistance will also be discussed.

S3: 20th International Symposium on Solid Oxide Cells (SOC): Materials, Science and Technology

Electrolytes

Room: Ponce de Leon (North Tower)
Session Chair: Scott Barnett, Northwestern Univ

8:30 AM

(ICACC-S3-044-2023) Strategies for Achieving high performance in Solid Oxide Electrochemical Devices using LSGM at KICET (Invited)

T. Shin*[1]

1. Korea Institute of Ceramic Engineering & Technology, Hydrogen Energy Materials Center, Republic of Korea

Many studies have been reported for not only efficient production of hydrogen via SOECs but also high-performed SOFCs, and $La_{1-x}Sr_xGa_{1-y}Mg_yO_{3-\delta}$ (LSGM), a substitute for yttria-stabilized zirconia (YSZ) has emerged as a rising candidate for the electrolyte due to its superior ionic conductivity even in intermediate temperature (≤1073K). However, a few essential requirements must still be addressed for the widespread use of commercial LSGM-based electrochemical devices; their higher reactivity with electrodes compared to the YSZ-based electrolyte makes it difficult to find a cost-effective manufacturing process and compatible materials. In this regard, several strategies for advanced manufacturing have been carried out for the competitive commercialization of LSGM-based SOCs at KICET: (a) lowering temperature processing, (b) non-touchable electrode coating processing, and (c) buffer layer. Our innovative activity on the recent LSGM-based SOCs will be discussed in detail. Heverin, the fabricated cell exhibits an outstanding maximum power density of 1.40 W cm^{-2} with the ohmic resistance of 0.131 Ω cm^2 at 1073K through the effects of the modified electrode surface via the simple-step sonic spraying technique. When the higher electrolysis performance was, subsequently, achieved on reverse SOC operation mode; a high current density of 1.01 A cm^{-2} at 1073 K, when applied to 1.3 V.

9:00 AM

(ICACC-S3-045-2023) Defect Thermodynamics and Transport Properties of Proton Conducting Oxide $BaZr_{1-x}Y_xO_{3-\delta}$ (x≤0.1) Evaluated Based on Density Functional Theory Modeling

Y. Lee*[1]; Y. Duan[1]; D. C. Sorescu[1]; W. Saidi[1]; D. Morgan[2]; T. Kalapos[1]; W. K. Epting[1]; G. Hackett[1]; H. W. Abernathy[1]

1. National Energy Technology Laboratory, USA
2. University of Wisconsin-Madison, Materials Science and Engineering, USA

Density functional theory-based thermodynamic modeling was performed to determine the effect of humidity and H_2/O_2 gas pressure on the defect chemistry and transport properties of proton conducting oxide $BaZr_{1-x}Y_xO_{3-\delta}$ (x≤0.1). The charge defect analysis, together with the hydration and oxidation reaction energies, was carried out to predict the Brouwer diagram and the transport properties relevant for solid-oxide cell applications. It is shown that the oxygen vacancies introduced to compensate the extra Ba vacancies may cause a slight increase in the concentration of hydroxyl and proton species upon hydration of Ba deficient $BaZr_{1-x}Y_xO_{3-\delta}$, while the overall proton diffusivity is predicted to decrease due to increased proton hopping barriers upon trapping near the Ba vacancies. The defect model developed was demonstrated to be capable of describing the experimental transport properties of $BaZr_{0.9}Y_{0.1}O_{3-\delta}$ in the solid-oxide cell operating conditions. The role played by the acceptor-bound holes, the self-regulated Fermi levels of $BaZr_{0.9}Y_{0.1}O_{3-\delta}$ due to the intrinsic and hydrogen point defects, and the two key chemical reactions (oxidation and hydration reactions) upon the conductivities of holes, protons, and oxygen vacancies under the hydrogen-rich and oxygen-rich conditions at various humidity levels, will be discussed.

9:20 AM

(ICACC-S3-046-2023) Conductivity Increase as a Result of Eliminating Short-Range Ordering in a Scandia-Zirconia Electrolyte

S. Zhang*[1]; C. Savaniu[1]; J. T. Irvine[1]

1. University of St Andrews, School of Chemistry, United Kingdom

Solid oxide cell electrolytes and structures have been greatly optimised to offer high performance and good durability for commercial implementation. This has highlighted an underlying issue where the slow, long-term conductivity degradation in the generally used fluorite type oxide-ion conductors known to be related to reorganisation of their defect short-range structures. Scandia stabilised zirconia (SSZ) offers the highest ionic conductivity in doped zirconias due to the smallest mismatch between the dopant and host cations, meaning the system is more prior to defect ordering. Depending on the composition, its ionic conductivity ages more significantly than other doped zirconias on high temperature annealing. Here, we report a magnesia and india co-doped SSZ composition that offers high ionic conductivity, 0.14 at 850C and 0.4 at 1000C, improved phase stability in both long- and short-range, and long-term conductivity stability. By annealing the sample at 850C, the conductivity behaviour at different time-domain is correlated to its microdomain structural evolution. We show the initial of conductivity increase over time as a result of eliminating the defect short-range ordering using electron and powder neutron diffractions, resulting in a long-term stable disordered structure with improved conductivity. We will discuss driving forces for the breakdown of defect ordering.

9:40 AM

(ICACC-S3-047-2023) Measurement of mechanical properties of thin zirconia substrates

C. Steinborn[1]; M. Kusnezoff*[1]

1. Fraunhofer IKTS, Germany

One of the most critical issues in cell and stack manufacturing is the mechanical failure in ceramic electrolyte of cermet substrate. There are several standards for the measurement of mechanical strength of ceramic substrates all based on assumption of mechanical failure by bending distance, which is considerably lower than thickness of ceramics. These approach fails for very thin substrates (<200 μm) which bending up to failure event is much higher than substrate thickness. Up to date ball-on-three-ball (B3B) and bending tests have been adapted for such type of testing. The mechanical analysis of existing testing procedures performed by ANSYS shows that related volume experiencing maximal tension strength for samples under test is small and decreases significantly with increased bending and decreased thickness of the substrate. To provide the mechanical properties relevant for typical electrolyte volume of electrolyte supported cells tension stress testing methodology has been developed. The values for mechanical strength and Weibull module obtained from testing of electrolytes with size of 40 mm x 120 mm x 0,09 mm are compared with B3B results obtained on small samples resulted in a good agreement. The developed methodology allows to assess the properties of electrolytes with thickness down to 40-60 μm utilizing big test volumes.

Fuel Electrode

Room: Ponce de Leon (North Tower)
Session Chair: Prabhakar Singh, University of Connecticut

10:20 AM

(ICACC-S3-048-2023) Metal Oxide Electrocatalysts for High Temperature CO_2 Splitting in Solid Oxide Electrolysis Cells (Invited)

V. Birss*[1]; H. Ansari[1]; A. Bass[1]; S. Bouzidi[1]

1. University of Calgary, Department of Chemistry, Canada

Perovskite oxides, having a general formula ABO_3, are a versatile class of high temperature materials that are of increasing interest as electrocatalysts in solid oxide fuel cells (SOFCs) and in solid oxide electrolysis cells (SOECs). One of their main advantages as that they exhibit mixed ionic and electronic conductivity and thus their entire exposed surface area is electrochemically active, contrary to the case of metal/ceramic composites, where reactions occur on at the triple phase boundary. In our group, we have been working on a promising class of perovskite electrocatalysts, $La_{0.3}M_{0.7}Fe_{0.7}Cr_{0.3}O_{3-\delta}$ (M = Sr,Ca) (LMFCr), materials that have been demonstrated to be highly active in both air and H2 fuel environments, and both in the SOFC and SOEC mode of operation. LMFCr is also highly active for both CO_2 reduction and CO oxidation, thus opening the door to symmetrical, reversible solid oxide cells that can serve either to store energy by converting CO_2 to CO or generate electricity via CO oxidation. In this talk, the focus will be on the effect of transition metal doping of LCFCr, which leads to the ex-solution of metal nanoparticles as well as a micro-roughened LCFCr surface. Together, this leads to a significant enhancement in the kinetics of CO_2 reduction but especially of CO oxidation.

10:50 AM

(ICACC-S3-049-2023) Solid Oxide Cell Degradation and Failure due to Siloxane Deposition (Invited)

R. Milcarek*[1]

1. Arizona State University, School for Engineering of Matter, Transport and Energy, USA

High fuel flexibility characterizes solid oxide fuel cells in comparison to other fuel cell technologies, but challenges remain with contaminants. Well-known contaminants like carbon and sulfur poisoning have received considerable attention. Silicon poisoning, resulting from insulating materials or siloxane in biogas, has receive much less attention. Data suggests that poisoning related to silicon can result in rapid and irreversible degradation of the anode even with ppb level contamination. This presentation provides an overview of experimental studies on siloxane contamination research and suggest future areas of exploration. Cyclic structure siloxane is shown to be stable with evidence suggesting it adsorbs near the anode surface on Ni and yttria-stabilized zirconia. Moist fuels resulting in hydroxylation of the yttria-stabilized zirconia surface increases the rate of siloxane deposition while the presence of water decreases carbon deposition on Ni. While yttria-stabilized zirconia is prone to contamination in the presence of water, siloxane deposition on Ni is more rapid in the presence of dry fuel. Long-term exposure or elevated concentrations of siloxane result in amorphous SiO_2 phases with some carbon present. Silica blocks diffusions channels in the anode resulting in failure due to mass transport limitations.

11:20 AM

(ICACC-S3-050-2023) Enhancing exsolution electrode performance by infiltration

S. Wang*[1]; P. V. Hendriksen[1]; B. Sudireddy[1]

1. Technical University of Denmark, DTU Energy, Denmark

Alternatives to Ni:YSZfuel electrodes are desirable to enhance robustness and stability of solid oxide cells. In this work, A-site deficient lanthanum doped strontium titanate ($La_{0.47}Sr_{0.33}Ti_{0.94}Fe_{0.03}Ni_{0.03}O_{3-\delta}$, LSFNT) is investigated as fuel electrode in prepared state or after infiltration with ceria. It is observed that LSFNT exsolves nanoparticles in reducing atmosphere, likely helping performance but overall electrode polarization resistance (Rp) is large (~8.3 Ωcm^2 at 850 °C in 3%H_2O/97%H_2) in the "as-reduced" state. To promote the electrochemical performance, the LSFNT fuel electrodes were infiltrated with CGO ($Ce_{0.9}Gd_{0.1}O_{2-\delta}$) or Ni-CGO. After optimizing infiltration process (number of infiltration cycles, heattreatment temperature), greatly improved electrode performance was observed with Rp of 0.05 Ωcm^2 (CGO) and 0.09 Ωcm^2 (Ni-CGO) at 850 °C in 50%H_2O/50% H_2. These values are two to three orders of magnitude smaller than Rp of pure LSFNT electrode (3.1-26 Ωcm^2). At lower temperatures (650°C) these electrodes actually performed better than state-of-the-art Ni-YSZ electrodes. Rp of 0.38 Ωcm^2 and 0.76 Ωcm^2 in 3%H_2O/97 %H_2 is measured respectively for CGO and Ni-CGO infiltrated LSFNT electrode, whereas the Rp of standard Ni-YSZ fuel electrodes under similar conditions is 3.65 Ω cm^2. The long-term stability of the infiltrated LSFNT electrodes is currently being evaluated and will be discussed in detail in the paper.

11:40 AM

(ICACC-S3-051-2023) Investigation of Ce(Mn, Fe)O_2/La(Sr)Cr(Mn)O_3 composite as oxide cathode for high-temperature CO_2 electrolysis

S. Lee*[1]; T. Shin[2]

1. Korea Institute of Ceramic Engineering and Technology (KICET), Republic of Korea
2. Korea Institute of Ceramic Engineering & Technology, Energy Materials Center, Republic of Korea

High-temperature CO_2 electrolysis using solid oxide electrolysis cells (SOECs) has tremendous potential for reducing carbon emissions and storing energy from renewable sources. For decades, the nickel-based cathode was widely employed for SOFC/SOEC. However, the nickel-based cathode is easily deactivated when directly exposed to the CO/CO_2 redox reaction during high-temperature CO_2 electrolysis. The superior-robust perovskite oxide La(Sr)Cr(Mn)$O_{3-\delta}$ (LSCM) is suggested because of its high redox stability. However, it still required the adding precious catalyst for high-performance. Therefore, this study aims to further improve the performance of the LSCM electrode material by compositing it with Ce(Mn, Fe) O_2 (CMF). Finally, to analyze the LSCM-CMF composite for CO_2 electrolysis at high temperatures, the electrochemical performance is discussed in detail.

12:00 PM

(ICACC-S3-052-2023) Conversion of Methane to Valuable Chemicals over SFM Anode-based Metal Supported Solid Oxide Cells

B. Hu*[1]; M. Tucker[2]; f. Rosner[2]; H. Breunig[2]

1. Lawrence Berkeley National Laboratory, Energy Storage and Distributed Resources, USA
2. Lawrence Berkeley National Laboratory, USA

Electrochemical oxidative coupling of methane (OCM) in solid oxide cells (SOC) has demonstrated high selectivity by pumping oxygen ions through an electrolyte membrane. The oxygen ion flow is controlled by operating temperature, current density, and cell voltage. Here, the use of metal-supported solid oxide fuel cells (MS-SOFC) for OCM is demonstrated and optimized. The Lawrence Berkeley National Laboratory MS-SOFC is based on a porous symmetrical structure that endures fast heating and cooling. Scanning electron microscopy is utilized to assess the impact of processing and operating parameters on the electrode and support morphologies. The mass transport of chemical species is largely affected by the porosity of MS and electrode. The optimization of the cell structure and process parameters has improved the mass transport and the impact on the conversion rates will be presented. The selectivity of the electrochemical OCM conversion has been analyzed by an online gas chromatograph. Catalyst compositions, operating temperature, and flow rate greatly impact the conversion rate and selectivity. Finally, the stability and degradation mechanisms of $Sr_2Fe_{1.5}Mo_{0.5}O_{6-\delta}$ (SFM) anode-based SOCs will be presented. This research demonstrates technical progress in catalyst and device development in electrochemical oxidative coupling of methane for valuable chemical synthesis.

S4: Armor Ceramics - Challenges and New Developments

Current Developments in Boron Carbide and Advanced Ceramics I

Room: Coquina Salon B (North Tower)
Session Chair: Chris Marvel, Louisiana State University

8:30 AM

(ICACC-S4-010-2023) Bond-level Representation of Deformation and Fracture During Tensile Shock Loading of Boron Carbide (Invited)

G. Subhash*[1]

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

Bond-level deformation mechanisms during dynamic tensile fracture in boron carbide (BC) along [111] and at 90° to [111] were investigated using molecular dynamic simulations by propagating two tensile shock fronts of equal amplitude from opposite ends of a domain to meet at the center and cause fracture. In both cases, BC exhibited a linear-elastic axial stress-strain response up to the yield stress followed by non-linear behavior that differed significantly in the two orientations. Along [111], post-yield softening was followed by an almost perfectly plastic response. In contrast, a post-yield hardening was observed at 90° to [111] followed by an abrupt loss of strength. Spallation along [111] was accompanied by micro-crack initiation normal to the loading direction while at 90° to [111], spallation was preceded by crack formation at ~45° to the loading direction. These observations were explained by analyzing the deformation of the 41 bonds in the unit cell of Boron carbide. It was noted that the onset of plastic deformation along both crystal orientations is triggered by breakage of B-B inter-icosahedral bonds followed by B-C icosahedral chain bonds. The sequence in which the bonds break and the fraction of total number of broken bonds in given orientation result in different macroscopic (domain-level) responses.

9:00 AM

(ICACC-S4-011-2023) Development of Deep Neural Network Learned Interatomic Potential for Shock Simulations of B_4C

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1. University of Florida, Material Science and Engineering, USA
2. University of Florida, Mechanical and Aerospace Engineering, USA

Due to recent innovations in computing power, data-driven methods like machine learning (ML) have risen in popularity in the field of solid mechanics. For example, ML models have been fit to material potential energy surfaces (PES) due to their ability to reach ab initio accuracy with significantly reduced computational cost. Neural networks (NNs) are deep learning methods that can leverage the flexibility of biological neural pathways to learn the PES of complex, highly covalent materials in extreme environments. The development of such interatomic potentials (IP) is non-trivial and requires systematic investigations. This work presents the development of a NN-based IP for boron carbide (B_4C), specifically the training data generation, model development, and model validation. The breadth of computational and experimental literature available on B_4C allows for thorough validation of the IP. Preliminary results indicate a run-time speedup of nearly two orders of magnitude in shock simulations with the NN-based IP as compared to ReaxFF-based simulations. This increase in efficiency can radically improve the predictive nature of computational investigations previously unattainable with conventional approaches.

9:20 AM

(ICACC-S4-012-2023) Effect of TiB_2 Addition on the Properties B_4C

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1. Rutgers University, Material Science and Engineering, USA

The aim of this project is to synthesize TiB_2 powders with a high average grain size and to use these powders in composite production to increase the fracture toughness of B_4C-TiB_2 composites when it still has high density and hardness. To reach this aim, in this study, first, TiB_2 powders with two different average grain sizes were synthesized. Previous studies have shown that high density and good microstructure cannot be achieved since commercially available powders contain high levels of impurities, so it was essential to synthesize powders in-home. Then, TiB_2 with a high average grain size was added to B_4C at the rate of 2-3-5 wt.% while fine TiB_2 was added to 5 wt.% and composites were produced. All samples reached ~99% density, high hardness (~30GPa), and high elastic modulus. On the other hand, homogeneous microstructures were obtained. The highest fracture toughness was obtained at samples made with 5 wt.% coursers TiB_2 addition 3.65 $MPa.m^{1/2}$. When this value is compared with the fracture toughness of additive-free Starck B_4C, an increase of 58% was observed. In addition, a 52% increase was obtained when compared to the toughness of commercially available PAD B_4C.

9:40 AM

(ICACC-S4-013-2023) A Mechanism-based Approach towards Improving the Impact Performance of Boron Carbide

P. Malhotra[1]; A. Zare*[1]; M. He[1]; J. Moreno[1]; M. Shaeffer[1]; K. Ramesh[1]

1. Johns Hopkins University, Mechanical Engineering, USA

Boron carbide (BC) is desirable for protection applications, but its impact performance is limited by brittle fracture and loss of shear strength. Studies of commercial BC identified 2 contributing failure mechanisms: brittle fracture initiated from processing-induced carbon-rich microstructural heterogeneities and amorphization at high pressures. To control these mechanisms, 2 new ceramics were prepared: BC with minimal carbon-rich heterogeneities to mitigate crack nucleation and Si-doped BC to mitigate amorphization. Both ceramics were also reinforced with TiB_2 inclusions to improve fracture toughness. Impact performance of the new and commercial BC is compared through canonical sphere-on-plate impact experiments in the HyFIRE facility at the HEMI. WC projectiles were launched at BC plates that were backed by an Al block at low (1.2–2 km/s) or high (3–4.5 km/s) impact velocities. Low velocity shots didn't result in complete penetration of the BC plate and their impact performance was measured through characterization of crater attributes in the BC plates using X-ray micro-CT. Electron microscopy was also performed on the impacted plates to characterize the activated failure mechanisms. In contrast, high velocity shots resulted in perforation of the BC plate and penetration into the Al block and their impact performance was assessed by characterizing the crater attributes in the Al block.

Current Developments in Boron Carbide and Advanced Ceramics II

Room: Coquina Salon B (North Tower)
Session Chair: Arezoo Zare, Johns Hopkins University

10:20 AM

(ICACC-S4-014-2023) Microstrucutre, properties and ballistic tests of strong B_4C-TiB_2 composites densified by gas pressure sintering

D. Sciti*[1]; S. Failla[2]

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2. National Research Council of Italy - Institute of Science and Technology for Ceramics, Department of Chemical Science and Materials Technologies (DSCTM), Italy

B_4C-TiB_2 composites with classical 75/25 vol ratio were sintered by pressureless sintering with and without gas pressure application in the final stage of densification, using a novel prototypal furnace. A small fraction of WC was introduced through high energy milling of the starting powders with WC-Co spheres. High energy milling facilitated the densification thanks to incorporation of WC impurities acting as sintering aid, and size reduction of the starting boride and carbide powders. For the sake of comparison, a hot pressed B_4C-TiB_2 composite with the same composition and powder treatment was fabricated and tested. Strength, stiffness and toughness of the ceramic densified at 2050°C via gas pressure sintering were even better than those of the composite hot pressed at 1900°C, whilst the hardness was lower. Depth of Penetration tests on plates with 3 to 5 mm thickness demonstrated that the gas pressure sintered material had a superior performance compared to the hot pressed one, and the experimental results were further validated by statistical analyses. This work also revealed that hardness was not the property spotting the best ballistic performance.

10:40 AM

(ICACC-S4-015-2023) Boron Carbide-Silicon Carbide composites for next generation armour

H. Payne*[1]; L. J. Vandeperre[1]; F. Giuliani[1]

1. Imperial College London, Materials, United Kingdom

Modern warfare is developing rapidly; technology is becoming more advanced and the modern soldier requires more of it. With the increase in equipment there is an increase in weight which reduces mobility. Threats have also developed, in such environments greater protection can be required. These two results shows a need to produce lighter armour with a higher level of protection. Boron Carbide (B_4C) has shown great potential for use in armours. It has a high hardness and low density, but it sometimes fails prematurely. This has been explained by a phase transformation involving polytype collapse. This research aims to mitigate structural breakdown by microstructural design. It is hypothesised that a composite containing nano grains of B_4C and Silicon Carbide (SiC) can mitigate the polytype breakdown, whilst combining the low density of B_4C with the ballistic reliability of SiC. Current work has focused on creating robust powder production techniques, raman analysis of amorphous regions and investigation of amorphous regions using TEM. Composites have been indented and mapped across depth using Raman spectroscopy to determine the spread of amorphisation. TEM samples have focused on developing robust methods to lift out and thin heavily cracked indents. Large area images with high resolution have then been produced using montaging such that features can be examined holistically against those of B_4C.

11:00 AM

(ICACC-S4-016-2023) New armor ceramic material based on Silicon carbide for multi-hit ballistic performance

B. Pérez Román*[1]; J. Pérez Gallego[1]; J. Rubio[1]

1. Institute of Ceramics and Glass, CSIC, Chemical-Physics of Surfaces and Processes, Spain

This project is focused on the development of a new reaction-bonded Si_3N_4-SiC ceramic composite material for multi-hit ballistic capability armor plates with low areal density and high structural strength. The formation mechanism of Si_3N_4 into the reaction-bonded Si_3N_4-SiC composite was studied. Ceramic parts were developed by slip casting and pressureless sintering. The green parts of different shapes and dimensions were processed by slip-casting of the ceramic slurry into moulds of plaster of Paris. Followed by a thermal treatment of the green bodies into a N_2 atmosphere at T<1650C where a silicon nitridation and α-/β-Si_3N_4 occur. Sintered parts have been characterized by means of XRD, FE-SEM, and in terms of mechanical properties. Armor systems were prepared for Si_3N_4-SiC plates with UHMWPE backing layer to evaluate the multi-hit ballistic performance with different ammunitions under the threat established by NIJ level III Standards. The results showed a lightweight Si_3N_4-SiC composite material, with the combination of mechanical properties, microstructure, and grain size to produce low microcracking density after single and multiple impacts due to the ability of meso-structure (interconnected pores, binder phases, and large SiC grains). The samples of successful designs armor plates adequate high ballistic performance including satisfactory multi-hit performance have been demonstrated.

11:20 AM

(ICACC-S4-017-2023) Improvement of the mechanical properties of TiB2 for armour applications using different additives and sintering techniques.

S. Taraborelli*[1]; S. Failla[2]; D. Sciti[3]

1. University of Parma, Chemical science, life science and environmental sustainability, Italy
2. National Research Council of Italy - Institute of Science and Technology for Ceramics, Department of Chemical Science and Materials Technologies (DSCTM), Italy
3. ISTEC-CNR, Italy

TiB2 is a promising material in several fields including impact resistant armor, seals, cutting tools, crucibles and wear resistant coatings given its physical, mechanical and chemical properties, in particular thanks to the combination of high hardness and exceptional wear resistance. It is however very difficult to sinter below 2000°C, also under mechanical pressure, and is limited by its low fracture toughness. By using sintering additives, it is possible to improve the sintering process and increase the mechanical properties since the additives react with oxidized layers to form secondary phases. In this study, we explored different preparation methods, various combinations of additives (B4C, Si3N4 and MoSi2), and sintering techniques (hot pressing and pressureless sintering). Thanks to the synergy between optimized process and tailored composition, an almost fully dense material was obtained at 1700°C with hardness of 24.4 ± 0.2 GPa and fracture toughness of 5.4 ± 0.2 MPa m0.5. However, the highest hardness value (30 ± 1 GPa) was obtained for samples sintered by pressureless sintering, featuring a core-shell grain structure.

11:40 AM

(ICACC-S4-018-2023) Surfing boundary conditions to characterize effective fracture-toughness of brittle composites

Z. Hossain*[1]

1. University of Delaware, USA

Despite century-long successes in the field of fracture mechanics, the understanding of toughness-structure correlation remains elusive, regardless of the scale of the structure. For over a century now it has been possible to describe effective properties (such as effective stiffness) that are characterized by variational principles. Nonetheless, it remains a challenge to establish a notion of effective toughness that is governed by the criticality of the material. To design new ceramic-based armor materials, it is essential to develop robust characterization tools that can describe effective fracture toughness as a function of the nano-microstructural attributes of the composite. Under the conventional uniform remote loading at the macroscopic boundary, initial cracks grow in an unstable manner due to wild spatial variations in the singularity of the elastic fields across the nano-microstructure. In this talk, we will present a new boundary condition called the 'surfing boundary condition' that enables the determination of effective fracture toughness as a function of the nano-microstructures at multiple scales. It also makes it possible to explore the evolution of crack-path in complex heterogeneous media. The approach reveals the fundamental design insights for devising brittle composites possessing controllable anisotropic and asymmetric fracture toughness.

S6: Advanced Materials and Technologies for Rechargeable Energy Storage

Diagnostics and Materials Characterization for Lithium Batteries I

Room: Coquina Salon E (North Tower)
Session Chairs: Chongmin Wang, Pacific Northwest National Lab; Yu Katayama, Osaka University

8:30 AM

(ICACC-S6-037-2023) X-ray Spectroscopic Studies of Energy Storage Systems (Invited)

M. Balasubramanian*[1]

1. Oak Ridge National Laboratory, USA

There is significant interest worldwide to develop new battery systems for transportation and grid-storage applications. Development of new transformational technologies will require tailoring advanced materials and incorporating additional chemical or electrochemical processes. Using X-ray spectroscopy methods, we seek to capture the fine details of the redox behavior (electronic structure) and the complex local structure of battery materials. Such studies provide unique insight to develop structure-property correlations that are important to predict long-term battery performance and also help tailor new novel materials with high energy and power densities and stable crystal structures. In this talk, we will discuss recent X-ray studies of energy storage materials.

9:00 AM

(ICACC-S6-038-2023) Chemical States Visualization of Cathode Active Materials by X-ray Spectroscopic Ptychography (Invited)

N. Ishiguro*[1]

1. Tohoku University, International Center for Synchrotron Radiation Innovation Smart (SRIS), Japan

X-ray ptychography imaging using coherent X-rays from synchrotron radiation is one of the most promising tools for visualizing the mesoscopic structures in high space-resolution of 10-nm order or less, and a combination with XAFS spectroscopy gives information about their local structure and chemical states (e.g., element composition, valence, local structures, etc.). Here, we report some demonstrations of nano/mesoscale chemical state visualization of practical cathode active material particles using the X-ray spectroscopic ptychography techniques. We have conducted X-ray spectroscopic ptychography measurements to spinel-type lithium nickel-manganese oxides (LNMOs) particles at X-ray energies around Mn and Ni K-edge and succeeded in reconstructing 2-dimensional spatially-resolved XAFS and phase spectra. The chemical maps of the Mn/Ni density and valence and the electron density were successfully estimated by the curve-fitting analysis of the spatially-resolved XAFS, and the phase spectra and data mining of the chemical maps were also performed, indicating the existence of heterogeneous phase domains. We would also like to report the first demonstration of chemical state visualization of sulfur in cathode active materials of Li-S battery by spectroscopic ptychography in tender X-ray region.

9:30 AM

(ICACC-S6-039-2023) Anionic redox in Li-ion cathode materials: A spectroscopy point of view (Invited)

M. Sougrati*[1]

1. CNRS ICGM, France

Li-ion batteries (LIBs) are a key component of energy storage systems, including portable electronics and electric mobility engines. Demand for LIB is growing rapidly with the need for higher energy densities, longer lifetime and safer operation. Over the past decade, the performance of Li-ion batteries in terms of specific capacity has advanced sufficiently to bring them closer to the theoretical maxima expected for conventional cationic redox. Among the investigated solutions, the use of anionic redox (as à compliment to cathodic one) seems to be one of the most promising ideas. In this case, not only metals are involved on the electrochemical reactions, but also their "ligands". The most studied anions with a possible redox are based on oxygen and sulfur (O_2^{n-} and S^{n-}, $n<2$). Although the anionic redox offers a serious option to improve the energy density of LIBs (and NIBs), it brings new challenges in terms of fading mechanisms' understanding. In this talk, the principle, the mechanism and the limitations of materials exhibiting anionic redox will be explained through serval examples of iron[1], tin[2] and antimony[3] containing materials. The role of spectroscopies in mechanisms understanding will be highlighted.

Diagnostics and Materials Characterization for Lithium Batteries II

Room: Coquina Salon E (North Tower)
Session Chairs: Nozomu Ishiguro, Tohoku University; Mahalingam Balasubramanian, Oak Ridge National Lab

10:20 AM

(ICACC-S6-040-2023) In-situ and cryo-TEM diagnosis of SEI layer characteristics in rechargeable battery (Invited)

C. Wang*[1]

1. Pacific Northwest National Lab, USA

Electron microscopy based diagnosis, both in-situ and ex-situ, appears to be one of the essential methods for gaining insights as how an electrode material failure, therefore feeding back for designing and creating new materials with enhanced performances. In this presentation, I will focus on recent progress on using ex-situ, in-situ, operando and cryo-scanning transmission electron microscopy for probing into the structural and chemical evolution of interfacial process for lithium ion batteries, representatively such as Li and Si anode. I will highlight several recent key observations, which even appear to be well documented, while essentially are poorly understood, therefore limiting 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 the bottlenecks for advancing electrode materials design for better batteries. In perspective, challenges and opportunities for developing in-situ electron microscopy for probing both functional and structural materials will also be discussed.

10:50 AM

(ICACC-S6-041-2023) Operando Understanding of the Electrode/Electrolyte Interface in Lithium Batteries (Invited)

Y. Katayama*[1]

1. Osaka University, SANKEN, Japan

(Electro) chemical reaction at the electrode/electrolyte interface, including the formation of solid electrolyte interphase, is crucial in improving the performance of lithium batteries. Numerous operando techniques, for instance, X-ray photoelectron spectroscopy, Raman spectroscopy, and infrared spectroscopy, have been performed to study the reaction proceeding on the electrode surface. This talk will bring the most recent understanding of the electrode/electrolyte interface during battery operation. We will discuss the chemical reaction during the open circuit voltage, the (electro) chemical reaction during the charge-discharge of the batteries, and the role of the electrode/electrolyte interface in those (electro) chemical reactions. The molecular-level understanding of the (electro) chemical processes at the electrode/electrolyte interface by operando attenuated total reflection (ATR) infrared spectroscopy will be particularly discussed. We also extend this technique to probe the electrode/electrolyte interface of the polymer electrolytes, which is considered a next-generation electrolyte. Our findings highlight the importance of operando studies of the interface to capture reaction intermediates and suggest design strategies for more stable high-energy electrode and electrolyte materials.

11:20 AM

(ICACC-S6-042-2023) Design of new ionic conductor used as an electrode or solid electrolyte for non-aqueous batteries (Invited)

V. Pralong*[1]; J. Jean[1]; A. Sagot[1]; A. Neveu[1]

1. CNRS ENSICAEN, France

One of the major challenges of the 21st century is our ability to solve energy-related problems caused by ever-higher consumption, demography, and standard of living. It is therefore imperative to anticipate this energy demand and this in the context of sustainable development. Storage technologies are highly dependent on the materials used and it is necessary to search for new materials with advanced properties that are also ecological and economical. Manganese-based oxide materials are promising cathodes for alkaline ion batteries due to their high energy density, low cost, and low toxicity. We notice that the system A-Mn-O (with A=Li, Na, K) is extremely rich in terms of original structures and we will show new materials in this system. In addition, with the aim of making lithium batteries safer, the scientific community is looking in recent years to replace the liquid solvents used as electrolytes with a solid ionic conductor compound. Several families of materials have been developed, leading to major improvements in this technology (NASICON, perovskites, Garnets). In addition, thiophosphate family is widely explored and several compounds have been discovered in the pseudo-binary Li_2S-P_2S_5 -P_2O_5 diagram. In this presentation, we will discuss the structure-properties relationships for these phases and detail our strategy to design a suitable ionic conductor.

S7: 17th International Symposium on Functional Nanomaterials and Thin Films for Sustainable Energy Harvesting, Environmental, and Health Applications

Metal Oxide Nanostructures and Chalcogenides for Energy, Environmental and Water-splitting Applications I

Room: Coquina Salon G (North Tower)
Session Chair: Muhammet Toprak, KTH Royal Institute of Technology

8:30 AM

(ICACC-S7-001-2023) Advances in Photon-harvesting Technologies for Perovskite Absorbers and Water Splitting Reactions (Invited)

S. Mathur*[1]

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

Metal oxide nanostructures with hetero-contacts and phase boundaries offer unique platform for designing materials architectures for energy harvesting applications. As viable alternative to water electrolysis, photoelectrochemical (PEC) water splitting has emerged as a competitive technology being capable of converting solar energy directly into chemical energy using stable and efficient photocatalysts for solar hydrogen production. Even though the potential of hematite thin films for water splitting applications are widely accepted, researchers are still tackling the 'rust challenge'. We report here on the influence of external magnetic fields applied parallel or perpendicular to the substrate during plasma enhanced chemical vapor deposition of hematite (α-Fe_2O_3) nanostructures. Hematite films grown from iron precursors showed pronounced changes in crystallographic textures depending upon whether CVD was performed with or without external magnetic field. Investigations on the water splitting properties of the hematite films in a photoelectrochemical reactor revealed superior photocurrent values of hematite photoanodes deposited in external magnetic field. In addition, the role of emerging hybrid perovskites in tandem PEC-PV set-ups for photo-assisted water splitting reactions will be discussed.

9:00 AM

(ICACC-S7-002-2023) Role of Heterojunctions in Metal Oxide Heterostructures for Energy and Environmental Applications (Invited)

N. Pinna*[1]

1. Humboldt-Universität zu Berlin, Department of Chemistry, Germany

Heterostructures made from metal oxide semiconductors are fundamental for the development of high-performance gas sensors and electrocatalysts. In this lecture I will introduce hierarchical heterostructures composed of p-, n-type and insulating metal oxide shells. Precisely controlled films of alternating metal oxides can be uniformly deposited onto the inner and outer walls of the CNTs and onto mesoporous substrates by atomic layer deposition. The morphological, microstructural and electrical characteristics of the heterostructures were investigated. Electrical resistance measurements highlighted the large influence of the metal oxides thickness and charge carriers types, suggesting that the conductivity of the electrodes are dominated by Schottky barrier junctions across the heterojunctions. The behavior of the heterostructured electrodes in sensors and in electrocatalysis applications was investigated for low concentrations of volatile organic compounds and pollutants, and the oxygen evolution reaction in acidic media, respectively. For examples, the gas sensing response of the heterostructures showed a strong dependence on the thickness of the metal oxide shell layers and the type of heterostructures formed. On the other hand, the electrocatalytic performances are strongly related to the surface catalyst layer and the electrical conductivity of the electrode.

9:30 AM

(ICACC-S7-003-2023) New generaton of chalcogenide and phosphide catalyst for water splitting and thermoelectric applications (Invited)

D. Chua*[1]

1. National University of Singapore, Materials Science & Engineering, Singapore

Today's catalyst goes beyond traditional expensive noble metals, especially in the area of electrochemistry where new generation of chalcogenides and phosphide have been shown to be highly effective. We we shall like to report on a new generation of 0D, 1D and 2D nanocomposites. ational design and synthesis of material with controlled structures and morphologies from nanoscale to microscale are of utmost importance in order to achieve optimal performances. We shall like to report our work in chalcogenide and phosphide family where electrocatalyst such as NiP have been shown to be highly promising for water splitting. We will further show that other types of chalcogenides such as MoS and WS2 also exhibit interesting properties for water splitting while CuS has potential in PEC applications. By redesigning the materials, we will show that some of the chalcogenides such as SnS shows promise as thermoelectric materials.

Metal Oxide Nanostructures and Chalcogenides for Energy, Environmental and Water-splitting Applications II

Room: Coquina Salon G (North Tower)
Session Chair: Thomas Fischer, University of Cologne

10:20 AM

(ICACC-S7-004-2023) Synthetic approach to metal chalcogenide functional materials for sustainable energy conversion by molecular building block assembly

V. Brune*[1]; D. Patrun[1]; Z. Aytuna[1]; S. Mathur[1]

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

Layered materials beyond omnipresent graphene such as chalcogenide-based materials that possess tunable and defined crystallographic structures and elemental compositions offer a broad portfolio of potential applications in energy storage and conversion devices. The lacking control of large scale and homogeneous formation of chalcogenide-based materials MX_y (X = S, Se; Y = 1, 2; M =metal) in commercial formation processes motivated us to develop a uniform synthetic approach to variety chalcogenide-based materials. Complete characterization of synthesized complexes enabled their controlled decomposition to energy-efficient materials. Herein we report a reliable synthesis approach of molecular building blocks to form (air) stable precursor classes $[M\{S(C_2H_4)_2NMe\}_y]$ ($M = Mo^{IV}, W^{IV}, Ti^{IV}, Zr^{IV}, Hf^{IV}, Nb^{IV}, Sn^{IV}, y = 2; M = Ge^{II}, Sn^{II}, Pb^{II}, Ni^{II}; y = 1$), which deliver the desired functional material. These synthesized complexess enabled the formation of homogeneous assemblies of inorganic nanostructured materials by thermal decomposition. These extraordenary molecular precursors deliver an economic approach for large scale synthesis of chalcogenide-based functional materials for application in sustainable energy conversion processes.

10:40 AM

(ICACC-S7-005-2023) Hydrogen Evolution Reaction with Metal Chalcogenide Nanofilms as Catalysts in Electrochemical Water Splitting.

D. Patrun*[1]; L. Jürgensen[1]; S. Mathur[1]

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

In recent years, hydrogen gained interest as energy storage for sustainable harvested energy. A common method for hydrogen production is the electrochemical water splitting. Promising electrode materials for this method are metal sulfides and selenides, whereby thin film coatings of these materials can be used. Since the availability of precursors for metal chalcogenides is limited, the demand for novel molecular structures is high. In this work, nanoscale thin films of metal sulfides were produced by chemical vapor deposition (CVD), for which the design of precursors was tailored in a targeted manner. Different metal chalcogenides were obtained by the variation of ligand classes and replacement of the heteroatom. The synthesis of precursors containing sulfur and selenium was successful. Complexes with the transition metals of cobalt and nickel were synthesized and provide ideal precursors for CVD. The conversion could be demonstrated by various analytical measurement methods. Thin films of Ni_3S_2 were successfully generated. With the application in the photoelectrochemical cell (PEC), its suitability as a novel material with good electrical properties was demonstrated. Thus, thin films of metal chalcogenides represent promising materials as catalyst in the water splitting reaction.

11:00 AM

(ICACC-S7-006-2023) Metal Alkoxides Derived High Entropy Oxides: A Design Strategy For Efficient Oxygen Evolution Reaction (OER) Electrocatalysts

Z. Aytuna*[1]; A. Bhardwaj[1]; M. Wilhelm[1]; S. Mathur[1]

1. Institute of inorganic Chemistry, Department of Chemistry, Germany

High entropy materials (HEO) have gained enormous attention in recent years due to various application possibilities, based on the different attributes of each implemented metal cation and the synergy in between. The tailorable characteristic of the materials makes them a great opportunity, to choose and tune the metals and thus the properties. Therefore, different precursors and the synthesis approaches are finetuned to obtain a high entropy system, by using metal alkoxides, especially metal isopropoxides of titanium, vanadium, tin, zirconium, and hafnium. Furthermore, the synthesized HEO was analyzed with different characterization methods like X-ray photoelectron spectroscopy (XPS), X-ray absorption spectroscopy (XAS), X-ray diffraction (XRD), and scanning electron microscopy (SEM). The material was tested for oxygen evolution reaction since high entropy materials have also gained high interest in terms of catalytical activity in the field of renewable energy. Based on that, the electrochemical water splitting reaction was catalyzed by the synthesized HEO $[(V,Sn,Ti,Zr,Hf)O_2]$ and superior catalytical activity with an overpotential of 301 mV and an electrochemically active surface area of 3375 Fcm^{-2} could be obtained. Furthermore, the material is tested on stability by a chronoamperometry measurement, showing great stability over 90 hours.

11:20 AM

(ICACC-S7-007-2023) Understanding of Degradation Process on the Photoelectrochemical Energy and Materials

S. Kang*[1]

1. Chonnam National University, Department of Chemistry education, Republic of Korea

Hydrogen (H_2) is one of the most widely used essential chemicals worldwide. It has applications in the production of many potential chemicals, especially carbon-free energy carrier. Here, a solar-powered photoelectrochemical cell based on a Ni-Pi (Nickel phosphate) cocatalyst covered crystalline ~30 nm thick TiO_2 layer on porous Etched Gallium arsenide (ET-GaAs) photoanode is described, converting water into O_2 to achieve photocurrent density (Jph) of 23.52 mA/cm^2 and 25.77 mA/cm^2 at 0 V versus reversible hydrogen electrode (briefly marked as V_{RHE}) and 1.23 V_{RHE}, respectively, under one sun illumination in strong alkaline electrolyte. Herein, the TiO_2 as a surface protecting layer exhibits the high-density crystalline structure and at the same time, the carriers move easily through the oxygen defects in TiO_2 layer to make a fast charge transport rate as required for the highly efficient and durable GaAs photoanode for over 50 h. Furthermore, the Ni-Pi co-catalyst provides a highly efficient charger transfer pathways for solar water oxidation with a meaningful shift (~ 100 mV) of onset potential (V_{on}), continuously boosting the photostability to 110 h-testing for PEC water oxidation with maintaining a steady J_{ph} of over 25 mA/cm^2.

11:40 AM

(ICACC-S7-008-2023) Facile synthesis of high-surface area anatase from $TiOSO_4.2H_2O$ as excellent photocatalyst

T. L. Nguyen*[1]; D. Seo[1]

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

Titanium dioxide (TiO_2) has been one of the most important materials in photocatalysis, water splitting and dye-sensitized solar cells, and yet there remains challenges in controlling morphologies for optimized performance due to the lack of readily available precursors with easy control of their reaction in solution. Recently, titanyl sulfate dihydrate ($TiOSO_4.2H_2O$, TSD) has been considered as a sustainable and cost-effective precursor, yet it still faces the same issue due to its solid-state nature. Our studies overcome this problem by (1) discovering the nonaqueous chemistry of TSD via its facile exfoliation in an organic solvent at a high concentration up to 57 wt%, due to its one-dimensional structure and Brønsted acidity; and (2) employing that controllable nonaqueous chemistry of TSD for synthesis of Ti-based materials. To demonstrate that, TSD in methanol solution is used to prepare highly porous anatase bulk material or submicron-thick coatings via a simple base treatment (ammonia in methanol or in water), following by washing and drying. The products are characterized by powder X-ray diffraction, thermogravimetric analysis, Fourier transform infrared spectroscopy, Raman spectroscopy, gas sorption analysis and microscopic studies. The bulk samples have shown high surface area, ranging from 300 to 460 m^2/g, with excellent photocatalytic activity towards methylene blue.

S9: Porous Ceramics: Novel Developments and Applications

Structure and Properties of Porous Ceramics

Room: Coquina Salon H (North Tower)
Session Chairs: Manabu Fukushima, National Institute of Advanced Industrial Science and Technology (AIST); Surojit Gupta, University of North Dakota

8:30 AM

(ICACC-S9-001-2023) Exploration of DDGS-PLA and HAP-PLA Scaffolds for Tissue Engineering (Invited)

M. Geigle*[1]; E. Eades[1]; S. Javaid[1]; A. Thorn[1]; S. Gupta[1]

1. University of North Dakota, Mechanical Engineering, USA

In this presentation, we will present a novel method for manufacturing scaffolds from environmentally friendly benign sources. The scaffold will be composed of Poly Lactic Acid (PLA) or Distiller's dried grains with solubles (DDGS) and Hydroxyapatite (HAP) where KBr will be added as a pore-former to achieve three different types of porosity, for example 70%, 90%, and 95%. These compositions will be characterized for microstructure and mechanical strength. The high porosity sample could not be adequately tested for compressive strength due to difficulties in machining. Detailed behavior of these scaffolds in Simulated Body Fluid (SBF) will be also tested.

9:00 AM

(ICACC-S9-002-2023) Mechanical Behavior of Calcium Phosphate - Alumina Ceramic Composite Porous Scaffolds Coated with Poly(lactic acid)

Y. Zusho*[1]; S. Kobayashi[2]

1. Tokyo Metropolitan University, Mechanical Systems Engineering, Japan
2. Tokyo Metropolitan University, Mechanical Engineering, Japan

In this study, porous ceramic composite scaffolds consisting of calcium phosphate, with excellent biocompatibility, and alumina, with high mechanical properties, have been prepared by a gelation freeze-casting method. The composite of ceramics with different properties has resulted in scaffolds with high pore orientation and mechanical properties. We proposed a novel hybrid porous scaffold with enhanced mechanical properties by immersing a porous ceramic composite in a chloroform solution of poly(lactic acid) (PLA). The coating conditions of PLA were examined by compression tests and microstructural observations. The volume fraction of the PLA coating increased in proportion to the concentration of the PLA solution. When the amount of alumina in the composite ceramics was 50 %(v), Young's modulus and compressive strength increased 2.6 and 2.1 times, respectively, at a concentration of 9% (w/v) of PLA solution. This was owing to the repair of surface defects by the PLA coating. Furthermore, fracture behavior was monitored using acoustic emission techniques. While the PLA coating did not affect the type of the main fracture of the porous scaffold, it affected the type of fracture during collapse, with tensile fracture dominating. The hybridization of ceramics and polymers improved the mechanical properties of the scaffolds.

9:20 AM

(ICACC-S9-003-2023) Porous Silicon Nitride Ceramics for RF Radomes Fabricated by Slip Casting

A. Kimery*[1]; C. Martinez[1]; R. Trice[1]

1. Purdue University, Department of Materials Engineering, USA

Radomes are used to protect the antenna of aircraft during high-speed flight while still maintaining transmission of electromagnetic waves. Silicon nitride has attracted interest as a radar frequency (RF) radome material because of its low dielectric constant and loss, and suitable high temperature mechanical and thermal properties. Porosity can be added to silicon nitride to further lower its dielectric constant. In this study, a slip casting method was used to fabricate silicon nitride with varying levels of porosity using fugitive carbon black. Microstructure and physical properties will be reported.

9:40 AM

(ICACC-S9-004-2023) Evolution of crack path in porous carbides

Z. Hossain*[1]

1. University of Delaware, USA

In this presentation, we will discuss the role of crystallography in the formation of crack paths in porous silicon carbide. By applying a multiscale framework, we explore the elastic interaction between a crack-tip and pore-edge and find that depending on the symmetry of the crystallographic plane, the crack can dodge the pore (as if there were no pore present on its path) or get annihilated at the pore. If the crack path is terminated at the pore, its continued evolution requires a renucleation event that takes place at the opposite end of the pore and requires much higher elastic energy compared to that required for the initiation of the crack propagation. As a result, pore enhances the effective toughness of the material. This finding paves a pathway on how to design crack-path a priori to control crack paths and enhance the toughness of composites containing porous materials.

10:20 AM

(ICACC-S9-005-2023) Properties and estimation of mullite based thermal Insulators prepared by gelation freezing Route

M. Fukushima*[1]

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

Mullite based thermal insulators were fabricated by freeze-drying gels containing calcined various types of raw materials such as mullite, calcined kaolinite, nano-sized silica or alumina, followed by sintering. The resultant porosity could be varied from 70% to 95% by solid loadings in the initial slurry. The relationship among porosity, microstructure, compressive strength, and thermal conductivity was examined. Those properties were also estimated by simulation using modeling of overall pore morphology, resulting in good agreement.

10:40 AM

(ICACC-S9-006-2023) Understanding thermal stability in doped zirconia aerogels for high temperature applications

N. Olson*[1]; J. Meyer[1]; H. Guo[2]; F. Hurwitz[3]; J. L. Stokes[4]; J. A. Krogstad[5]

1. University of Illinois at Urbana-Champaign, Materials Science & Engineering, USA
2. Universities Space Research Association, USA
3. NASA Glenn Research Center, Materials Chemistry and Physics Branch, USA
4. NASA Glenn Research Center, Environmental Effects and Coatings Branch, USA
5. University of Illinois at Urbana-Champaign, Materials Science and Engineering, USA

The high porosities and low densities of ceramic aerogels offer outstanding insulative performance in applications where weight is a critical factor. The high surface-to-volume ratios and specific surface areas provide extremely low thermal conductivity, but also contribute to rapid densification of the pore structure at elevated temperatures. This densification diminishes their favorable properties and inhibits use of aerogels in high temperature applications. The purpose of this work is the establishment of a design framework for thermally stable, highly porous materials. Here we will review our recent studies on the effects of synthetic parameters and dopant chemistry on the evolution of zirconia-based aerogels. The structural evolution was studied to 1200 °C using nitrogen physisorption, scanning and transmission electron microscopy, and x-ray diffraction. The role of dopant identity and concentration in thermal stability was elucidated. In context of the design framework, dopant chemistry is an aggregate for many closely related material properties, each of which may contribute to aerogel structural evolution. To develop a truly predictive design framework for ceramic-based aerogels, systematic and comprehensive evaluation of thermodynamic and kinetic properties must be performed in conjunction with studies on structural evolution.

11:00 AM

(ICACC-S9-007-2023) Porous composites from cold sintering applied to insulating and hygroscopy

E. Kamseu*[1]; Z. Ngouloure[1]; L. K. Tiogning-Djiogue[5]; A. Akono[2];
F. Andreola[4]; B. Nait-Ali[3]; S. Rossignol[3]; C. Leonelli[4]

1. Local Materials Promotion Authority, Cameroon
2. Northwestern University, Civil and Environmental Engineering, USA
3. University of Limoges, IRCER, UMR 7315, France
4. University of Modena and Reggio Emilia, Department of Engineering Enzo Ferrari, Italy
5. University of Yaoundé 1, National Advanced School of Engineering, Cameroon

The extent of dissolution and polymerization/polycondensation of metakaolin added biomass in alkaline solution was exploited to design highly porous insulating geopolymers using sodium polyacrylate and calcium silicate to enhance the hygroscopic properties. 2.5 to 7.5 wt% of sodium polyacrylate and 20-40 wt% of calcium silicate efficiently embedded into the geopolymer gel were able to increase the absorption and desorption capacities between 30 and 50% maintaining the thermal conductivity at 0.13-0.16 $W.m^{-1}K^{-1}$ up to 70% of the relative humidity and 0.22-0.28 $W.m^{-1}K^{-1}$ when the relative humidity is above 80%. The extension of the reactivity of metakaolin added biomass conducts to homogeneous gel in which specific surface area is improved enhancing the hygrothermal properties. The pore size (essentially nano and meso) and their interconnectivity make the porous matrices achieved promising candidates for the core vacuum insulation and regulation of the thermal comfort for Net zero Energy buildings. A Machine Learning (ML) method, the polynomial regression was used to ascertain the close correlation between the thermal conductivity, Temperature, moisture absorption and time. The increase of nano and meso pores increase of the tortuosity of the porous matrices and explain the improvement of the hygrothermal properties. The changes into the microstructure with the increase of the volume of fine pores.

11:20 AM

(ICACC-S9-008-2023) Development of porous phyllosilicate-based ceramics using freeze tape casting

K. Barry[2]; G. Lecomte-Nana*[1]; M. Seynou[2]; P. Blanchart[1]; C. Peyratout[1]

1. IRCER, ENSIL-ENSCI, University of Limoges, France
2. University Joseph Ki Zerbo de Ouagadougou I, Burkina Faso

In the present study, kaolinite- and halloysite-rich clays were used to manufacture porous ceramics shaped by freeze tape casting (FTC). A commercial kaolin (KCR) (provided by Imerys), and two kaolinite-rich (KMY) and halloysite-rich (HCR) clay from Cameroun were used as starting clays. The optimized suspensions were prepared using dolaflux B11, polyvinyl alcohol (PVA22000) and polyethylene glycol (PEG300° as dispersant, binder and plasticizer respectively. FTC was used in order to include a multiscale ordering thanks to ice templating effect within kaolinite or halloysite-based samples. The as-obtained freeze tape casting samples were freeze-dried prior to their sintering at 1200 and 1300°C. The total porosity and biaxial flexural strength were determined. The porosity of dried samples was very similar. The stress to rupture values for KCR and KMY were greater than for HCR. The sintering of all samples at 1200°C lead to an increase in porosity values (close to 70%). KCR samples stress to rupture were significantly improved in comparison to HCR samples. The latter trend resulted from the type and amount of mullite within these samples. Despite the great porosity of sintered samples, their mechanical resistance was less affected. This behavior suggested a profitable effect of lamellar pores, thanks to FTC. Sintering at 1300°C allowed improving some properties of the HCR samples

11:40 AM

(ICACC-S9-009-2023) Incorporating Ni catalysts into freeze-cast SiOC porous materials

K. Yu*[1]; K. Faber[2]

1. Caltech, Materials Science, USA
2. California Institute of Technology, USA

Porous SiOC structures produced through freeze casting and pyrolysis of polysiloxane pre-ceramic polymers are known for their directionally permeability and thermally stability. These structures are promising catalyst support materials for high-temperature reactions, such as the Ni-catalyzed Sabatier process that produces methane from carbon dioxide and hydrogen. However, incorporating large quantities of catalysts can change the rheological and thermal properties of the solution, disrupting the desired templating created during freeze casting. This ultimately limits the maximum achievable catalyst loading if introduced prior to freeze casting. Additionally, these catalysts are dispersed within the pore walls, further reducing the exposed surface sites available for catalytic activity. This work focuses on a novel method of sol-gel infiltration of pyrolyzed SiOC to increase the amount of Ni catalyst loading. This is achieved by separating the freeze casting and SiOC conversion from the catalyst incorporation process. The resulting two-step, sol-gel derived Ni-SiOC structures are compared to one-step, solution-derived structures, with an emphasis on Ni catalyst loading. The evolution of Ni from its precursors during the processing steps is characterized using X-ray diffraction, while both Ni-SiOC microstructures are compared using SEM.

S10: Modeling and Design of Ceramics and Composites

Modeling of Ceramics and Composites I

Room: Coquina Salon A (North Tower)
Session Chair: Gerard Vignoles, University Bordeaux

8:30 AM

(ICACC-S10-001-2023) Distinctive Mechanical and Functional Properties Due to Glide Dislocations in Zinc Sulfide (Invited)

K. Matsunaga*[1]

1. Nagoya University, Materials Physics, Japan

High-purity ZnS single crystals having the zincblende structure can undergo extraordinarily large plastic deformation even at room temperature in complete darkness (Oshima, Nakamura, and Matsunaga, Science (2018)). In normal light-illumination environments, however, ZnS single crystals immediately fracture in a brittle manner after only a few percent of plastic strain. These results are closely related to changes in mobility of glide dislocations in ZnS due to light illumination. DFT calculations show that glide dislocations in ZnS have smaller band gaps and excess electrostatic charges, so that extra electrons and holes excited by light illumination can be favorably trapped at the dislocation cores. Such carrier trapping at the dislocation cores also accompany atomic reconstruction, which may be closely related to the decreased dislocation mobilities under light illumination, namely hardening of ZnS, which was suggested by experiment. It is also found that the plastically deformed ZnS single crystals exhibit specific photoluminescence and its intensities tend to increase with the glide dislocation densities. This also suggests interactions of the glide dislocations with exited carriers. It can be said, therefore, that glide dislocations in ZnS can induce not only the distinctive mechanical properties but also the specific functional properties.

9:00 AM

(ICACC-S10-002-2023) Light scattering prediction approaches for transparent ceramics

S. Hribalova*[1]; W. Pabst[1]

1. University of Chemistry and Technology, Prague, Department of Glass and Ceramics, Czechia

Transparent ceramics have been employed in many applications, the most classic being impact-resistant windows and lasers. Despite the long history of the development of high-quality transparent ceramics, the main challenge in preparation remains the same – minimizing the light scattering at the wavelength of interest, which is detrimental to high transparency and good optical properties. In ceramics, there are typically two types of light scattering sources: residual porosity or impurities and randomly oriented birefringent grains, which is where the light scattering predictions come into play. In this contribution, several approaches for light scattering predictions will be presented. Furthermore, their advantages and disadvantages will be discussed using a few examples of transparent ceramic systems such as (i) pyrochlore transparent ceramics, i.e. ceramics with the general composition $A_2B_2O_7$ and pyrochlore structure, which are promising candidates for use as scintillator hosts and luminophores. And (ii) perovskite transparent ceramics, e.g. potassium sodium niobate (KNN – $K_{0.5}Na_{0.5}NbO_3$), mostly known as a potential lead-free alternative to the lead-containing (but still most widely used) so-called PZT piezoelectric ceramics. However, KNN in its transparent form can be used in electro-optic applications, such as optical integrated circuits, optical resonators, etc.

9:20 AM

(ICACC-S10-003-2023) High-throughput extrapolation of 0 K calculated phase stability to relevant temperatures

M. to Baben*[1]; C. Aras[1]

1. GTT-Technologies, Germany

The measurement of thermochemical properties for ceramic systems is extremely challenging because of -usually- slow kinetics and high temperatures needed. Ab initio calculations at 0 K are a very popular additional source of data. In the last decade, this has led to the development of huge repositories with calculated data such as materialsproject.org or oqmd.org, containing data for many hundreds of thousands of compounds. Here, it will be shown how the 0 K phase stability data from the complete databases materialsproject.org and oqmd.org can be extrapolated to relevant temperatures. This is done by ML models for Cp(T) and S(T) trained on high-quality Calphad data taken from the FactSage databases. In addition, enthalpy of formation consistency modifications are used to make the enthalpy of formation consistent to existing Calphad databases. The models have lead to the development of aiMP and aiOQ databases (www.gtt-technologies.de). Application of aiMP and aiOQ for the calculation of phase diagrams are displayed.

9:40 AM

(ICACC-S10-004-2023) Pt Alloy/Oxide Catalysts for Suppressing H_2O_2 Formation in Polymer Electrolyte Fuel Cell Anodes by First-Principles Calculations

R. Kano*[1]; Y. Asano[1]; Q. Chen[2]; Y. Ootani[1]; N. Ozawa[2]; M. Kubo[1]

1. IMR Tohoku University, Japan
2. Tohoku University, New Industry Creation Hatchery Center, Japan

In polymer electrolyte fuel cells (PEFC), OH radicals generated from H_2O_2 decompose the polymer electrolyte membrane, leading to decrease in the PEFC life. Here, H_2O_2 is formed on the Pt catalyst when oxygen passes from the cathode to anode. Experimentally, Co alloying and combination of oxide suppress H_2O_2 formation. For the design of a highly-durable catalyst, the effects of alloying and oxide support on H_2O_2 formation should be revealed. In this study, we investigated H_2O_2 formation on a composite of PtCo alloy nanoparticles and oxides by first-principles calculation. To reveal the effect of Co content on H_2O_2 formation, the activation energies for HO_2 and H_2O_2 formation from a O_2 molecules and H atoms on $Pt_{32}Co_6$ and $Pt_{36}Co_2$ are calculated. The activation energies for HO_2 formation on the $Pt_{36}Co_2$ and $Pt_{32}Co_6$ clusters were 5.98 and 18.20 kcal/mol, respectively. Moreover, those for H_2O_2 formation on the $Pt_{36}Co_2$ and $Pt_{32}Co_6$ clusters were 7.13 and 5.59 kcal/mol, respectively. The activation energy for HO_2 formation on the $Pt_{32}Co_6$ cluster is higher. We suggest that the increase in Co content is effective in inhibiting H_2O_2 formation. The effects of the oxide support will be discussed at the conference. These contribute to proposing guidelines for Pt based catalysts that suppress H_2O_2 formation.

Modeling of Ceramics and Composites II

Room: Coquina Salon A (North Tower)
Session Chair: Katsuyuki Matsunaga, Nagoya University

10:20 AM

(ICACC-S10-005-2023) Random Walks for image-based simulations of heat or mass transfer in evolving porous media: Applications to Ceramic-Matrix Composites & Ceramic Foams (Invited)

G. L. Vignoles*[1]

1. University Bordeaux, LCTS - Lab for ThermStructural Composites, France

Simulation problems linked to the fabrication, use and degradation of Ceramic-Matrix Composites and ceramic foams involves a precise knowledge of effective heat and mass transfer properties of porous media at the fiber scale and the fabric scale for CMC and cell scale for foams. When dealing with complex porous architectures, predictive tools have to be able to handle large 3D images, including the capability to modify them through infiltration or ablation phenomena. This presentation will describe a class of methods developed to fulfil these requirements. Monte-Carlo/Random Walks have the interesting property of simulating diffusive, ballistic or mixed-mode transfer phenomena in a straightforward way. Moreover, they require a very small amount of extra memory in addition to image storage, thus enabling simulations in very large images. Coupled to an efficient interface discretization scheme, the Simplified Marching Cube, they are very efficient. Example of applications to simulations of Chemical Vapor Infiltration, featuring rarefied gas transfer, chemical deposition and porous medium densification, and to simulations of conducto-radiative heat transfer in various media (fibers, foams) will be given, in order to illustrate the versatility and performance of this class of methods.

10:50 AM

(ICACC-S10-006-2023) Flowsheet simulation applied for porcelain tile manufacturing optimization and sustainability

C. Lourenco Alves*[1]; V. Skorych[1]; A. De Noni Jr.[2]; D. Hotza[2]; S. Y. Gómez González[2]; S. Heinrich[1]

1. Hamburg University of Technology, Institute of Solids Process Engineering and Particle Technology, Germany
2. Federal University of Santa Catarina (UFSC), Department of Chemical Engineering (EQA), Brazil

Porcelain tile manufacturing is currenlty guided by insights, material characterization as well as trial and error approaches that are time-consuming and expensive. However, numerical simulations have become valuable tools to describe and analyze production processes. Thereby, the determination of optimal processing parameters from simulations of the whole process chain allows at the same time the optimization of product quality and substantial savings. In this contribution, a simulation approach is presented that has been developed for the complete process chain of porcelain tiles aiming the sustainability and optimization of it. For numerical investigations of processes with connection of production steps by material and energy streams, flowsheet calculations have been already applied successfully. This concept has been proved to be suitable for simulation of porcelain tile manufacturing and forecast outcomes during the process chain. Thereby, a set of semi-empirical models of the different equipment and processing steps has been developed and implemented into an open-source framework Dyssol. The coupling between Dyssol and Matlab allowed to build a first optimization approach. The obtained results confirm that the integration of these simulation possibilities the determination of optimal process parameters and first steps into a complete “digital” manufacturing plant.

11:10 AM

(ICACC-S10-007-2023) Unveiled the origin of structural instability of LixNi1/3Co1/3Mn1/3O2 and LixNi0.8Co0.1Mn0.1O2 cathode materials upon delithiation/lithiation

L. Kuo*[1]; O. Guillon[2]; P. Kaghazchi[3]

1. Ming Chi University of Technology, Chemical Engineering, Taiwan
2. Forschungszentrum Juelich, IEK-1, Germany
3. Forschungszentrum Juelich, Germany

In this work, we combined electrostatic and density functional theory (DFT) calculations to determine atomistic and electronic structures of $Li_xNi_{1/3}Co_{1/3}Mn_{1/3}O_2$ (NCM111) and Ni-rich $Li_xNi_{0.8}Co_{0.1}Mn_{0.1}O_2$ (NCM811) cathode materials. The reason behind the larger lattice parameters changes and structural phase transitions on NCM811 compared to NCM111 are explained. The O-stacking induced phase transition which has not been well understood is shown to be driven by electrostatic forces. It is also found that the reason of hexagonal to monoclinic (H–M) phase transition for Ni-rich NCM811 is due to the collective Jahn-Teller (J–T) distortion. We indicated that the Li-O attractions, charges on transition metals (TMs) and O anions, J–T distortion and electrostatic interactions dominate the structural changes of NCM materials.

11:30 AM

(ICACC-S10-008-2023) Data-driven discovery of computationally complex ceramics for extreme environments

S. Bavdekar*[2]; G. Subhash[1]; R. G. Hennig[2]

1. University of Florida, Mechanical and Aerospace Engineering, USA
2. University of Florida, Materials Science and Engineering, USA

We present a high-throughput strategy to design compositionally complex ceramics (C^3) for extreme environments by establishing design rules that permit accelerated discovery. The first phase – i.e., the ‘exploitation’ phase – focuses on well-understood, design spaces (e.g., Si-C-N, Al-O-N systems), by identifying new stoichiometries and structures. Evolutionary structure searches coupled to density-functional theory (DFT) calculations are applied to predict the ground state and metastable structures within a given system. These searches aim to not only find structures with low energies (i.e., on or close to the convex hull), but also maximize the targeted property (in this case, hardness). Hence, the algorithm serves to both, generate and screen data within the given system to produce structures with the desired stability and properties. The data obtained throughout these structure searches is exploited in a machine-learning model that is trained on the fly and can accelerate the structure prediction and provide an accurate and efficient surrogate model of the energy (and hardness) landscape. Through this framework, we aim to develop chemical design rules to answer the fundamental question of how chemical additions to the structure affect its hardness and stability.

11:50 AM

(ICACC-S10-009-2023) Evaluation of Macrocharacteristics of Materials Based on Highly Boron Compounds by Ab-initio Research at Atomic Level

E. Kartuzov*[1]; V. Kartuzov[1]; L. Ovsiannikova[1]; N. Rozhenko[1]

1. IPMS NASU, Ukraine

Methods to evaluate a performance of characteristics of highly boron compound materials based on the results of ab initio calculations of their structural components are proposed. Establishment of important geometric and energy characteristics of structural components of boron-containing compounds from the first principles was realized with the use of Gaussian and GAMESS application software, which are also used as a means of computer simulation of formation of structure of these compounds. The results of simulation of structure formation of highly boron compounds obtained from the first principles are presented and values of atomization energy and volume of self-organized clusters - fragments of structure of these materials are calculated in terms of DFT. It is shown that the size of substitution atom of C, O, Si, Al, Mg in materials based on icosahedral boron has a determining value for the adhesion energy (stability) of clusters B_{12}. The effect on macrocharacteristics of solid solutions based on zirconium diboride of C and / or Mo substitution atoms has been studied. The developed approaches to determine a bulk compressibility and hardness by the values of atomization energy and the volume of material's structural cell were in a good agreement with experimental data when calculating the fragments of structure up to 50 atoms.

12:10 PM

(ICACC-S10-010-2023) New approaches to computer design of ultrahigh-temperature ceramics based on refractory compounds

V. Kartuzov*[1]; O. Vasiliev[1]; V. Bekenev[1]; V. Muratov[1]; D. Vedel[1]; E. Kartuzov[1]

1. IPMS NASU, Ukraine

In addition to presenting a new methodology for the computer design of new promising materials specifically for ultrahigh-temperature ceramics, calculations using DFT and cluster expansion show the ideal behavior of the corresponding solid solutions and the likelihood of a sudden increase in the level of mechanical properties with varying concentrations of controlling components. Thermodynamic analysis of promising synthesis reactions demonstrates the possibility of obtaining such systems. Trial samples of the prototypes of the studied materials obtained under hot pressing conditions confirm the formation of a solid solution and the multiphase nature of the product. The determination of the hardness of these samples gave interesting dependences.

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

Recycling and Reuse Processes

Room: Ballroom 1-2 (South Tower)
Session Chair: Sungwook Mhin, Korea Institute of Industrial Technology

9:00 AM

(ICACC-S11-013-2023) Innovative Recycling Technology for Used Li-ion Batteries using Li Separation Method by Ionic Conductor: LiSMIC (Invited)

T. Hoshino*[1]; K. Morita[1]; T. Matsumoto[1]

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

The EV market is expanding rapidly toward 2050 carbon neutrality, though it is expected that it will be difficult to secure sufficient Li for lithium-ion battery (LIB) by around 2030. Recycling technology for used LIB is needed to be established for the stable securement of Li. Then, such an ultra-high purity Li recovery technology required for battery raw materials must meet the business profitability. We evaluated the recycling cost of used LIB in Lithium Separation Method by Ionic Conductor (LiSMIC), which is a low-cost, ultrapure (99.99 %) Li recovery method that we have developed. For the evaluation, we have developed a new Li recovery device that can stack 20 plates of ionic conductors, operate continuously for several weeks, and search for the optimum conditions for Li recovery. Black powder (battery ash), obtained by roasting used LIB, was immersed in water, and 50 L of the LIB leachate was used as the stock solution. Then, Li recovery test under the optimal condition was carried out using a new ion conductor that had been subjected to surface treatment, which accelerates the Li recovery by adsorbing Li in the solution. It was clarified that the manufacturing cost of $LiOH(H_2O)$, which is one of the raw materials for LIB, is less than half the import price (1,287 yen / kg).

9:30 AM

(ICACC-S11-014-2023) Preparation of carbon-free lime from unused resources by using the ethical process (Invited)

M. Tafu*[1]; T. Fukumura[2]

1. National Institute of Technology, Toyama College, Japan
2. National Institute of Technology, Ichinoseki College, Japan

Calcium, as one of the common elements, is the base element of various functional materials. Calcium materials derive from calcium salts made by the calcination of limestone. Limestone consists of calcium carbonate and releases half the weight of carbon dioxide from the calcination process. In this study, we have attempted to develop carbon-emission-free lime production from various unused resources containing calcium. We applied the mechanochemical method that obtained various compounds from colliding the raw materials, ball, and mill wall. We investigated a suitable production process using gypsum from waste plasterboard or limestone pellet from the water softener and sodium hydroxide. The experimental results showed that lime powder was successfully obtained from the mechanochemical process from gypsum and calcium carbonate. We also characterized carbon emissions from each process and estimated the ethical character of this method. In this presentation, we introduce the recent achievements of our research. (This research is supported by Environment Research and Technology Development Fund FY2022 (JPMMEERF20222M02), Ministry of the Environment, Japan.)

Sustainable Energy Concepts and Applications

Room: Ballroom 1-2 (South Tower)
Session Chair: Chisung Ahn, Korea Institute of Industrial Technology

10:20 AM

(ICACC-S11-015-2023) Flexible nanogenerator with co-additive, $ZnSnO_3$/surface-modified carbon nanotubes, for high-power energy harvesting and pulse sensing (Invited)

S. Mhin*[2]; H. Han[3]; K. Kim[1]

1. Korea Institute of Industrial Technology, Republic of Korea
2. Kyonggi University, Republic of Korea
3. Konkuk University, Republic of Korea

Wireless and wearable electronic devices for the Internet of Things (IoT) technology have rapidly developed and is gradually integrating into our daily life. Recent technological progress on energy harvesting system is promising to fully replace the battery-driven wireless devices and thus, achieving self-powered devices for IoT. Energy harvesting is eco-friendly technology to generate electricity from ubiquitous mechanical energy via the triboelectric, piezoelectric, pyroelectric, and electromagnetic conversion. Among them, piezoelectric nanogenerator (PENG) is considered as a promising power source due to their high energy efficiency, reliability and cost effectiveness. Herein, we introduce a peapod-inspired co-additive in which $ZnSnO_3$ anchored on surface-modified carbon nanotubes (SMC), enhancing the piezoelectricity of poly(vinylidene fluoride-co-trifluoroethylene)-based (P(VDF-TrFE)-based) nanofibers. Experiments show high output power ((97.5 V and 1.16 μA) as well as imperceptible pulse detection even in posterior tibial arteries. This work gives a scientific and technical understanding to not only the formation of co-additive via pulsed laser ablation, but also the development of polymer-based PENG with enhanced triboelectric performance for use in energy harvesting and sensing.

10:50 AM

(ICACC-S11-016-2023) Application of Manganese Dioxide in Aqueous Zinc-ion Secondary Batteries (Invited)

S. Lee[2]; M. Kim[2]; T. Kim[3]; S. Mhin[2]; J. Kim*[1]

1. Daegu Mechatronics & Materials Institute, Republic of Korea
2. Kyonggi University, Republic of Korea
3. Korea Institute of Industrial Technology, Republic of Korea

Recent technological advance on rechargeable Zn-ion batteries have shown promising applicability as an advanced energy storage system to replace conventional Li-ion batteries. In order to enable its commercial value, it is essential to make highly-rechargeable Zn-ion batteries with improved charge-discharge cycles and performance. In this study, the effect of manganese dioxide prepared by different processing route on the performance of the Zn-ion battery are investigated. In the aspect of processing routes, wet chemical method and solid state reaction were used to prepare nanosized MnO_2 and microsized MnO_2, respectively. Also, nanosized MnO_2/CNT was also prepared using wet chemical method. Materials characterization of the prepared MnO_2 were investigated. Also, different types of MnO_2 were examined using full-cell analysis in CR2032 cells composed of Zn foil as an anode and MnO_2 as a cathode, and specific capacity and cycle performance of the full cell are evaluated. The presented study suggests the guidelines for selection of suitable cathode materials in Zn-ion rechargeable batteries.

11:20 AM

(ICACC-S11-017-2023) The enhancement of power generation through pyroelectric bodies under the pulse power electric field

S. Buddhika Amila Kumara*[1]; T. Nakayama[1]

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

This study aims to improve pyroelectric power generation by using waste heat energy as a renewable energy source in combination with pyroelectric nanogenerators and an external pulse electric field. Under optimal conditions, the maximum power can be accumulated by applying different pulses of the external electric field at temperature variations on the surface of the pyroelectric body. A newly developed experimental setup based on the modified Diode and two Switches circuit is used to measure and calculate the power generation of the pyroelectric body. The power generation of the Standard fuji ceramic C9 sample was evaluated in a continuous temperature variation environment under a low and high, temperature difference of 20°C. The temperature variation frequency was 0.05Hz and the pulse electric field was applied when the temperature rose. The pulse width of the electric field 10, 20, and 50ms pulses were applied to the sample under two pulses and multi-pulse conditions. And the amplitude of each pulse was 500, 1000, and 1500V/mm. Finally, the maximum power generation condition was evaluated by comparing two pulses and multiple pulse trains. The experimental setup can be further upgraded to apply a nano pulse stream with high voltages condition to the pyroelectric body. This could be accomplished without any dielectric breakdown to generate more electrical energy.

S12: On the Design of Nanolaminated Ternary Transition Metal Carbides/Nitrides (MAX Phases) and Borides (MAB Phases), Solid Solutions Thereof, and 2D Counterparts (MXenes, MBenes)

Design of Novel Compositions and Manufacturing Methods II

Room: Ballroom 3 (South Tower)

Session Chairs: Anne Joulain, Institut PPRIME; Helmut Riedl, TU Wien

8:30 AM

(ICACC-S12-008-2023) Magnetocaloric properties of MAB phases (Invited)

D. Cakir*[1]; Y. Loh[1]

1. University of North Dakota, Physics and Astrophysics, USA

MAB phases provide a fertile playground to study the emerging magnetic properties and behaviors with respect to the chemical composition and crystal structure. Also, the discovery of substantial, near-room temperature magnetocaloric properties of Fe_2AlB_2 with a Curie temperature of 270-320 K has triggered a tremendously growing interest in the magnetic properties of MAB phases. In this talk, we investigated the magnetocaloric properties of stable MAB phases and elucidate their potential in magnetocaloric applications using density functional theory calculations, Monte Carlo simulations, and the Heisenberg model.

9:00 AM

(ICACC-S12-009-2023) Si containing transition metal diborides (TM-Si-$B_{2\pm z}$) – Advanced ternary compounds for high temperature oxidative environments

L. Zauner[2]; T. Glechner[2]; A. Bahr[2]; A. Hirle[2]; C. Fuger[2]; R. Hahn[2]; T. Wojcik[2]; J. Ramm[3]; O. Hunold[3]; P. Polcik[4]; H. Riedl*[1]

1. TU Wien, Institute of Materials Science and Technology, Austria
2. Christian Doppler Laboratory for Surface Engineering of high-performance Components, TU Wien, Austria
3. Oerlikon Surface Solutions AG, Liechtenstein
4. Plansee Composite Materials GmbH, Germany

In the exploration of novel MAB phases owning unusual and unique properties, hexagonal transition metal diborides often constitute the basis for developments of these atomically layered arrangements – obtaining in the simplest form at least 2-dimensional boron hexagon sheets. With this contribution, we want to introduce ternary Si containing transition metal diborides (TM = Cr, Hf) as promising compounds for extreme conditions. In contrast to classical 2D solids but also well-known SiC alloyed bulk ceramics, we directly synthesized TM-Si-$B_{2\pm z}$ solid solutions by physical vapor deposition (PVD). Based on the limited kinetics during growth all TM-Si-$B_{2\pm z}$ coatings investigated were stabilized in the AlB_2-type structure. Above well-defined Si contents Cr-Si-$B_{2\pm z}$ and Hf-Si-$B_{2\pm z}$ exhibit strongly retarded oxidation kinetics with k_p values being below 10^{-11} $kg^2m^{-4}s^{-1}$ at 1100 °C. Long term oxidation tests at 1200 °C confirmed the protective ability, i.e. the initially 2.60 μm thin $Hf_{0.20}Si_{0.23}B_{0.57}$ exhibits an oxide scale thickness of only 1.5 μm after 60 h. Along with a broad set of high-resolution characterization techniques describing the phase stability (HR-TEM, APT, SIMS) as well as the fracture behaviour by micromechanical testing, the study suggests these ternary diborides as alternatives for mechanically and oxidative harsh environments.

9:20 AM

(ICACC-S12-010-2023) Analyzing the effects of external deformation constraint on the mechanical response of MAX phase crystals

M. Dujovic*[1]; M. Radovic[2]; A. Srivastava[3]; T. Ouisse[4]

1. Texas A&M University, Materials Science and Engineering (MSEN), USA
2. Texas A&M University, Materials Science & Engineering, USA
3. Texas A&M University, USA
4. Grenoble INP, France

A family of atomically layered ternary carbides and nitrides commonly referred to as MAX phases exhibit properties that combine some of the best attributes of both metals and ceramics. Their atomically layered crystal structure facilitates several deformation and failure mechanisms that include crystallographic slip, cleavage, buckling of layers, and kinking. Recently, several attempts have been made to characterize these materials' single-crystal level mechanical response using compression tests of micropillar specimens milled from individual grains. These studies have shown that the crystallographic slip in MAX phases not only depends on the resolved shear stresses on the active (basal) slip systems but also on the stresses normal to the basal planes. Building on these recent works, here we carry out a series of unconventional small-scale compression tests on single crystal specimens of a MAX phase to directly analyze the effects of imposed external constraint on crystallographic slip, cleavage and kinking. We will present the results correlating the effects of imposed external constraint and the crystallographic orientation of the single crystal specimens on the overall mechanical response of MAX phases.

Design of Novel Compositions and Manufacturing Methods III

Room: Ballroom 3 (South Tower)
Session Chairs: Sophia Tsipas, Universidad Carlos III de Madrid; Ankit Srivastava, Texas A&M University

10:20 AM

(ICACC-S12-012-2023) Nano and Sub-Nano Scales Manipulation of 2D Materials (Invited)

M. Naguib*[1]

1. Tulane University, Physics and Engineering Physics, USA

MXenes constitute a large family of 2D transition metal carbides/nitrides with a composition of $M_{n+1}X_nT_x$ (M is an early transition metal, X is C or N, n =1-4, and T_x stands for surface terminations). Their excellent electrical conductivity and capability to host ions make them ideal candidates as electrode materials for electrochemical energy storage. While intercalation plays an important role in the applications of MXenes since it is the mechanism for storing ions in it, less is known about the effect of pre-intercalation on the electrochemical performance of MXenes. We will discuss few examples where pre-intercalation introduced a significant change in the electrochemical performance of MXenes. Manipulating MXenes multilayers using pre-intercalations resulted in an ultra-high areal capacitance of up to 5.7 F/cm^2, which is a record number for MXenes. We found that pre-intercalated MXene with an interlayer spacing of $\approx$ 2.2 nm has a capacitance of 257 F/g -an order of magnitude higher than pristine MXene- over a voltage window of > 3V in neat EMIMTFSI electrolyte, leading to a high-energy density comparable to batteries without compromising their high-power density. Other atomic scale manipulation strategies for MXenes, for both electrochemical energy storage and hydrogen evolution reaction, will be discussed. In addition to MXenes, recent discovery of 2D transition metal carbo-chalcogenides (TMCC) will be presented.

10:50 AM

(ICACC-S12-013-2023) Expanding the Large Family of Ordered Double-Transition Metal 2D MXenes (Invited)

B. Anasori*[1]

1. Indiana University – Purdue University, Mechanical and Energy Engineering, USA

Since the first report of ordered double-transition metal carbides MXenes in 2015, they have become one of the largest subfamilies of MXenes. In the out-of-plane ordered double-transition metal MXenes, one or two atomic planes of a transition metal (e.g., Ti, Sc) are sandwiched between two planes of another transition metal (e.g., Mo) in the M_3C_2 or M_4C_3 structures, such as Mo_2TiC_2, Mo_2ScC_2 and $Mo_2Ti_2C_3$. In these MXenes, electrochemical, mechanical, optical, and electrical properties can be tuned by controlling the transition metals' ordered chemistry. In this talk, we present the new developments in these ordered structures by presenting the first tungsten-containing M_3C_2 MXene, W_2TiC_2, and a Mo-Nb-containing M_4C_3 MXene, $Mo_2Nb_2C_3$. W_2TiC_2 is the first MXene that was synthesized from a layered non-MAX precursor with no A-element. We will discuss the exact atomic ordering and the possibility of partial intermixing in some of the ordered atomic planes and the importance of these intermixing of atoms in the successful synthesis of MXenes and their resulting properties.

11:20 AM

(ICACC-S12-014-2023) Radiation-induced swelling in MAX phase ceramics for nuclear applications (Invited)

K. Lambrinou*[1]; N. Goossens[2]; B. Tunca[2]; T. Lapauw[2]; K. Van Loo[2]; S. Huang[2]; J. Hinks[1]; P. Persson[3]; J. Vleugels[2]

1. University of Huddersfield, School of Computing and Engineering, United Kingdom
2. KU Leuven, Department of Materials Engineering, Belgium
3. Linköping University, Department of Physics, Chemistry and Biology, Sweden

Nanolaminated MAX phases are described by the $M_{n+1}AX_n$ stoichiometry, where M is an early transition metal, A is an A-group element, X is C or N, and n = 1-3. Their hexagonal crystal structure leads to anisotropic swelling under irradiation, whereupon the unit cell expands along the c-axis and contracts along the a-axis. Phase-impure MAX phase ceramics are also susceptible to failure by radiation-induced differential swelling. This work addresses the swelling behaviour of MAX phases, mainly for fuel cladding applications. The evolution of radiation swelling in ternary (Zr_2AlC, Ti_2AlC, Cr_2AlC, etc.) and solid solution MAX phases was monitored in-situ in the TEM by means of helium implantation (6 keV He^+, 10^{14} ions/cm^{2}s) up to 10 dpa, in the 350-800°C range. The two main ways to control radiation swelling in the MAX phases are strong texture and high phase purity. The latter can be achieved in compositionally tailored double solid solutions that are produced with metal hydride raw powders. As opposed to pure metals, the milling of metal hydrides into fine particles does not lead to excessive oxidation, since the hydrides decompose into their respective metals, producing in situ powders with highly reactive surfaces and simultaneously releasing H_2 that prevents oxidation. The use of early transition metal hydrides enabled the synthesis of "impossible" to produce MAX phases, e.g., in the (Zr,Hf)-Al-C system.

S14: Crystalline Materials for Electrical, Optical and Medical Applications

Battery / Piezoelectric Material

Room: Flagler A (South Tower)
Session Chairs: Kenji Toda, Niigata University; Nerine Cherepy, Lawrence Livermore National Lab

10:10 AM

(ICACC-S14-001-2023) Lead-free 3D Piezoelectric Transition-Metal Tantalate for Ubiquitous Sub-kHz Sensing and Classification of Dynamic Vibrations

R. Mitra*[1]; U. Manju[1]

1. CSIR-Institute of Minerals and Materials Technology, Materials Chemistry Department, India

In this work, we discuss our findings on $ZnTa_2O_6$ (ZTO) – a transition metal tantalate ceramic, predicted to be a novel lead-free piezoelectric candidate and its first experimental evidence. Single-phase polycrystalline sample having orthorhombic Pbcn symmetry with non-centrosymmetric tri-α-PbO_2 structure was formed, possessing a mean crystallite size of ~40 nm. The crystallization of ZTO was observed at ~691 °C with a melting enthalpy, ΔH_m = ~301.7 J/g. Force-driven dielectric constant (ε_r) was found to be 7.05 with an ultra-low loss tangent value of $1x10^{-4}$ at 0.45 N dynamic force. From PE hysteresis experiments, a piezoelectric charge coefficient (d_{33}) value of 20 pC/N and an energy storage efficiency of 43% at 5 kV/cm were determined. Temperature-dependent Van der Pauw-Hall tests, which were predicated on first-order kinetics and produced anisotropic resistivity behaviour, revealed the existence of electron trapping centres. Further fabrication of a prototype sensor enabled it to measure the rotational frequency of a DC motor using Fast Fourier Transform (FFT) as well as detect dynamic vibrations ensuing from various weight loadings. ZTO synergistically combines all of these attributes to create a promising ferroic material with high-temperature stability that has novel advantages in MEMS, electronics, sensing, and energy harvesting.

10:30 AM

(ICACC-S14-002-2023) Highly Flexible PMN-0.3PT/PDMS Based Piezoelectric Polymer Composites for Energy Harvesting Application

A. Kumar*[1]; A. Roy[1]

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Here we report the design and fabrication of high-energy-density piezoelectric -polymer composite for low-frequency mechanical energy harvesting applications. Matrix (polydimethylsiloxane polymer) and reinforcement (PMN-0.3PT ceramic) are identified based on the figure-of-merit analysis. Room temperature powder x-ray diffraction and electron backscatter diffraction analyses predicted co-existing monoclinic (Cm and Pm) phases with a plausible Cm → Pm transformation mechanism. Flexible piezoelectric-polymer composite v_r-PMN-0.3PT/PDMS with different reinforcement content, v_r = 0.00-0.50 was fabricated. The effective elasto-electric properties estimated from experiments were compared with the finite element model predictions. A new figure-of-merit was proposed for quantitative estimation of mechanical flexibility of the composite samples. Energy harvesting under dynamic mechanical loading shows frequency and amplitude dependence of the applied loading. It was concluded reinforcement orientation is critical for higher output energy density.

10:50 AM

(ICACC-S14-003-2023) Synthesis of Cathode Materials using Water-Assisted Solid-State Reaction method for LIBs (Invited)

K. Toda*[1]

1. Niigata University, Japan

Recently, we have developed a new low temperature synthesis method, Water-Assisted Solid-State Reaction (WASSR) method. Since this reaction proceeds at low-temperature (from room temperature to 500 K), nano-particles can be obtained by the WASSR method. Additionally, this reaction is based on the solid-state reaction completes by a simple process, which is just addition of the amount of water to the mixing each raw materials and leaving the wet slurry for several hours. A nano-sized particle technique of the cathode materials is effective to solve the problem of slow charge-discharge because a diffusion path way of Li-ion in the crystal lattice becomes shorter. Therefore, in this work, we challenge to synthesize cathode materials using the WASSR method, and investigated the reaction mechanism, crystal structure and fundamental electric properties of the obtained cathode materials. Acknowledgements This work was supported by JSPS KAKENHI Grant Number 22H01769.

11:20 AM

(ICACC-S14-004-2023) High Performance Aurivillius Phase Calcium Bismuth Niobate for High-Temperature Piezoelectric Applications

C. Wang[1]; Q. Wang*[1]

1. Shandong University, School of Physics, State Key Laboratory of Crystal Materials, China

High-temperature (HT) piezoelectric materials are required for piezoelectric sensors in the nuclear power plants, aerospace, and energy industries. Calcium bismuth niobate ($CaBi_2Nb_2O_9$, CBN) is considered to be one of the most promising HT piezoelectric materials owing to its high Curie temperature T_c of ~940 °C, however, its low electrical resistance and poor piezoelectric properties at elevated temperatures limit its HT applications. In this work, we report the significantly enhanced dc electrical resistivity and piezoelectric performance of CBN ceramics. The donor substitutions of rare-earth elements (REE) ions for Ca^{2+} ions reduce the oxygen vacancy concentrations and increase the band-gap energy, which is responsible for the enhancement of dc electric resistivity. The temperature-dependent dc conduction properties reveal that the conduction is dominated by the thermally activated oxygen vacancies in the low-temperature (LT) region (200-350 °C) and by the intrinsic conduction in the HT region (350-650 °C). By compositions adjusting, it is found that the d_{33} value can be promoted to >16 pC/N, while the T_c remains ~900 °C. The d_{33} value can be further promoted to higher than 30 pC/N by spark plasma sintering (SPS). The results indicate that the CBN ceramics are promising materials for HT piezoelectric sensor applications.

11:40 AM

(ICACC-S14-005-2023) Simultaneously achieving high energy storage density and energy efficiency in $BaTiO_3$-$Bi(Mg_{1/2}Sn_{1/2})O_3$ ceramics

Q. Wang*[1]; X. Zhao[2]; C. Wang[1]

1. Shandong University, School of Physics, State Key Laboratory of Crystal Materials, China
2. Shandong University, Center for Optics Research and Engineering (CORE), Key Laboratory of Laser and Infrared System of Ministry of Education, China

The increasing demand for high performance energy storage devices with fast discharge speed and high power density have heightened the attentions of dielectric capacitors. Despite many explorative investigations, the recoverable energy storage density W_{rec}, the energy efficiency η and their thermal stabilities are barely satisfactory simultaneously. In this work, the 0.86$BaTiO_3$-0.14$Bi(Mg_{1/2}Sn_{1/2})O_3$ (BT-14BMS) relaxor ferroelectric ceramics have been synthesized by conventional solid-state reaction method. A large W_{rec} of 3.61 J/cm^3 and a high η of 92.8% are achieved at a high electric field of 370 kV/cm in BT-14BMS ceramics, with a fast discharge speed ($t_{0.9}$ of 127 ns), showing great advantages over the reported BT-based dielectric ceramics. Moreover, the energy storage properties of BT-14BMS ceramics exhibit good temperature and frequency stabilities in the range of 20 - 160 °C and 0.01 - 100 Hz, respectively. The remarkable energy storage performance achieved in BT-14BMS relaxor ferroelectric ceramics are attributed to the strong relaxor behavior and the enhanced dielectric breakdown characteristics, which is confirmed by the dielectric, ferroelectric and electric properties. These results indicate that BT-14BMS relaxor ferroelectric ceramics have promising potential for advanced pulsed power applications.

S16: Geopolymers, Inorganic Polymers and Sustainable Construction Materials

3D Printing of Geopolymers I

Room: Coquina Salon C (North Tower)
Session Chair: Sylvie Rossignol, Laboratoire SPCTS

8:30 AM

(ICACC-S16-029-2023) Composition and synthesis protocol as tools to tailor the textural properties of 3D printed geopolymeric components (Invited)

M. D'Agostini[1]; M. Muracchioli[1]; P. Colombo[1]; G. Franchin*[1]

1. University of Padova, Industrial Engineering, Italy

The mesoporous nature of geopolymers, together with their low cost and facile synthesis route, make them attractive for applications in adsorption processes. Several studies have been conducted in order to assess the feasibility of using geopolymer-based adsorbents for water purification and gas separation, as well as employing them as catalyst supports, where appropriate noble or transition metal species can be easily introduced through ion exchange. Slurry composition and synthesis parameters have a strong influence on the textural properties of geopolymers to be exploited for such functional applications, yet little research has been conducted in order to identify clear trends. Here, the results of a systematic study of multiple compositional parameters will be presented and discussed, including the choice of alkali cation and of SiO_2/Al_2O_3 and H_2O/Al_2O_3 molar ratios. The effect of the curing temperature and time on the material texture and its stability will also be addressed. An informed selection of all parameters allows fabricating components with high specific surface area (SSA) and pore size distribution tailored to the targeted application, and can promote bulk crystallization of the 3D printed slurries into zeolite monoliths. The performance of such components in key applications such as CO_2 uptake will be compared to the state-of-the-art.

9:00 AM

(ICACC-S16-030-2023) 3D printing of Martian/Lunar Regolith Simulant-based Geopolymer Composites (Invited)

P. He*[1]; S. Ma[1]

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

Geopolymers (GPs) have emerged as promising building blocks for Martian/Lunar base construction, yet their inherent brittleness and relatively high density hinder practical application and technological implementation. Implanting biomimetic inspiration from long-term evolution into GPs provides an alternative approach to endow the skeleton with fascinating properties such as lightweight, high strength and high toughness. Here, we first develop fiber-reinforced Martian/Lunar regolith simulant-based GP inks that exhibits excellent rheological properties and rapid solid-to-liquid transition, enabling large-scale production of GP composites with customizable biomimetic patterns and controllable fiber alignment. Furthermore, we reveal the biomimetic pattern-dependent mechanical properties and fracture mechanisms by combining in-situ compression experiments with finite element analysis. The results showed that the designed geopolyer strcutrues should very distinct fracture tolerance, and the ability to precisely control biomimetic patterns not only offers a new degree of freedom for fine-tuning their mechanical properties, but also opens up new avenues for designing damage-tolerant materials toward outer space bases.

9:30 AM

(ICACC-S16-031-2023) Effect of Temperature on Rheology during Early Stage Geopolymerization (Invited)

A. S. Brandvold*[1]; W. M. Kriven[2]

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

Metakaolin geopolymers have gained much interest as a potential alternative to Ordinary Portland Cement as a 3D printable material. It is well known that an increase in temperature can catalyze the geopolymerization reaction in geopolymer pastes, but little is known how the manipulation of temperature completely influences the rheological properties. We investigate the influence of temperature on the viscosity of potassium geopolymer pastes through constant shearing at various temperatures and shear rates. It is found that the use of high temperature will result in an initial drop in viscosity as the paste heats up and subsequent rapid increase in viscosity as polymerization starts to occur. At low temperatures, there is a substantial increase in viscosity and a slow decrease as temperature equilibration occurs and heating due to mechanical shearing. The shear rate is found to delay the start of polymerization if held at a high enough rate. Overall, the influence of temperature on rheological properties can be used to manipulate the geopolymer viscosity to optimize printing conditions.

3D Printing of Geopolymers II

Room: Coquina Salon C (North Tower)
Session Chair: Ange-Therese Akono, Northwestern University

10:20 AM

(ICACC-S16-032-2023) Influence of raw materials on the 3D printing behaviour of geopolymer slurries without organic additives

P. Scanferla*[1]; W. N'Cho[1]; J. Jouin[1]; S. Rossignol[2]

1. University of Limoges, IRCER, France
2. Laboratoire SPCTS, France

Geopolymers are inorganic materials obtained through the reaction of an aluminosilicate in an alkaline solution. Among all the raw materials that can be used as aluminosilicate source to make geopolymers, metakaolin is one of the most used, thanks to its large availability and sustainability. The use of different type of metakaolin, and their combinations, leads to changing the material's production properties. The aim of this work is to investigate several ceramic objects obtained via additive manufacturing processes, especially extrusion and direct ink printing (DIW). The study is focused on the possibility to produce objects starting from any metakaolin modifying the printing parameters, without introducing any organic additive. Geopolymers used in this work are obtained from a potassium silicate solution ([K] = 5 mol/l) and various types of metakaolin, which differ from Si/Al ratio and contaminants presence. Several formulations were investigated and preliminary tests on printability are encouraging. Results show the influence on the printability of the different metakaolins. In fact, varying the raw materials leads to have diverse liquid to solid ratios that influence the overall viscous behaviour of the paste.

10:40 AM

(ICACC-S16-033-2023) Composition dependence of water loss rate and near-surface microstructure of open air-cured metakaolin geopolymers for 3D printing

D. Samuel*[1]; W. M. Kriven[1]

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

To facilitate 3D printing of geopolymers in dry environments, it would be beneficial to design a mixture or coating that retains sufficient water within the geopolymer for reaction until it has completely hardened. Large differences in water retention of metakaolin-based geopolymer, as evaluated by dehydration rate in open air, have been found, due to changes in the initial water content and alkali cation. To better understand these effects, the pore size distribution and microstructure were characterized near the surface of geopolymers synthesized with 7 to 13 moles of H2O initial water content and both sodium and potassium as alkali cations. The properties of thin geopolymer samples cured in open conditions and in closed, high-humidity conditions were compared in order to identify changes due to surface dehydration. Comparisons were also made between thin geopolymers and sections taken from the interiors of large samples to characterize microstructural differences between the surface layer and bulk of geopolymers.

11:00 AM

(ICACC-S16-034-2023) Sustainability of additive manufacturing: Case studies of solid wastes and circular economy (Invited)

H. A. Colorado L.*[1]

1. Universidad de Antioquia, Colombia

This investigation presents research in additive manufacturing of construction materials towards circular economy solutions, and also case studies from Colombia using solid wastes. Several technologies are analyzed and also some examples are presented with complementary characterization techniques such as mechanical, physical, and microscopy techniques.

Porous Geopolymers I

Room: Coquina Salon C (North Tower)
Session Chair: Enrico Bernardo, University of Padova

11:30 AM

(ICACC-S16-035-2023) Formulation and mechanical properties of porous extruded geopolymers (Invited)

C. Zoude*[2]; L. Gremillard[2]; É. Prud'homme[2]; K. Johannes[1]

1. INSA Lyon, Centre for Energy and Thermal Sciences of Lyon, France
2. INSA Lyon, Materials, Engineering and Science, France

Thermochemical energy storage seems promising to meet new energy challenges in our society. However, the hygroscopic salts used by these systems must be associated with a host material as porous and resistant as possible. Geopolymers are good candidates due to their affordability, eco-friendly character, their good mechanical properties and thermal resistance. To optimise their porosity, we have chosen to design them by Direct Ink Writing. Different additives were mixed to an initial geopolymer ink formulation to achieve the desired properties. Initially, to make their rheology adequate for the printing process (shear thinning behaviour with high enough moduli), polyethylene glycol was selected. And in a second step, in order to increase their porosity to store more salt, we added aluminium powder which creates porosities by releasing dihydrogen. As a result, structures have been successfully printed and show good fidelity to the model at two different scales. The mechanical properties of printed filaments have been tested and show very different results depending on the drying conditions (temperature and humidity), formulation and the choice of protocol for paste preparation.

Emerging Materials and Sustainable Manufacturing Technologies in a Global Landscape: Symposium in Honor of Dr. Tatsuki Ohji

Tatsuki Ohji Honorary Symposium VI

Room: Coquina Salon D (North Tower)
Session Chairs: Katalin Balazsi, Centre for Energy Research HAS; Tohru Sekino, Osaka University

1:30 PM

(ICACC-HS-035-2023) 3D Printed Fiber-reinforced Highly Ductile Composite Scaffolds for Bone Tissue Engineering (Invited)

J. Chen[1]; S. Chen[1]; M. Wang*[1]

1. The University of Hong Kong, Department of Mechanical Engineering, Hong Kong

Bone tissue defects caused by injury or surgical resection of bone tumor deteriorate the integrity and functionality of bone. Tissue engineering provides an attractive approach for treating body tissue damage/loss. In scaffold-based bone tissue engineering, polymeric scaffolds are normal used but owing to the low strength of polymers and high porosity of scaffolds, they have poor mechanical properties. In this study, fiber-reinforced composite scaffolds were fabricated by direct ink drawing 3D printing for bone tissue engineering. Thermoplastic polyurethane nanofibers were electrospun first. They were then homogenized and evenly distributed in poly (D,L- lactide-co-trimethylene carbonate) (PLATMC) to form inks for 3D printing. The fiber-incorporated inks exhibited shear-thinning behavior and hence showed good printability. 3D printed composite scaffolds showed better shape fidelity than pure PLATMC scaffolds. SEM examinations revealed that nanofibers were unidirectionally distributed in printed struts, which contributed to the enhancement of mechanical properties of scaffolds with Young's modulus and tensile strength of composite scaffolds being 2.5 and 3.6 times, respectively, of those of PLATMC scaffolds, while the ultimate elongation of composite scaffolds was over 600%. Osteoblastic cells seated on composite scaffolds proliferated well, indicating good cytocompatibility of scaffolds.

2:00 PM

(ICACC-HS-036-2023) Waste material as source for nanosized bioceramics, bioactive coatings and porous ceramics (Invited)

K. Balazsi*[1]; M. Furko[1]; H. R. Ben Zine[1]; M. H. Kaou[2]; C. Balazsi[3]

1. Centre for Energy Research HAS, Thin Film Physics, Hungary
2. Óbuda University, Doctoral School on Materials Sciences and Technologies, Hungary
3. ELKH Centre for Energy Research, Hungary

The utilization of powder technology to modify the properties of different waste materials to develop the novel bioceramics is the focus of many scientific studies these days. The increasing demand for implantable devices, growing demand for biomaterial market. In this presentation the preparation of calcium phosphate thin bioactive coating, nanosized powders or sintered materials and porous calcium based ceramics prepared by powder technology will be shown. Hydroxyapatite is a widely used bioceramic in implantology considering its high bioactivity. A bioactive coating on the bioinert ceramic implant's surface (Si3N4) 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 with focus to main trends and fundamental scientific problems.

2:30 PM

(ICACC-HS-037-2023) Environmental barrier coating for SiC$_f$/SiC CMC: Science and Technology (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

Disruptive development of ceramic matrix composites (CMCs), specially the SiC$_f$/SiC CMC, boosted the new era of emerging engine towards higher efficiency and thrust-to-weight ratio. In the state-of-the-art design of aviation engine, environmental barrier coating (EBC) is crucial and protect CMC hot-section components against chemical degradation in extreme combustion environment. Mining advanced EBCs was active with the explorations of available rare earth elements and new phases; but the progress was not always encouraging. Innovation is frequently frustrated by the cautious balance of thermal property and corrosion resistances against hot steam and molten CaO-MgO-Al$_2$O$_3$-SiO$_2$ (CMAS) deposit at high temperatures. This lecture introduces the status and perspectives of high-throughput design of new EBCs and the multilevel construction of advanced coating through plasma spray technology.

3:20 PM

(ICACC-HS-038-2023) Chemical and Structural Tuning of Visible-light Responsible Nano-structured Titania for Photochemical Functions (Invited)

T. Sekino*[1]; H. Park[1]; D. Han[1]; S. Chou[1]; H. Nishida[1]; T. Goto[2]; M. Kakihana[1]; Y. Morimoto[1]

1. Osaka University, SANKEN (The Institute of Scientific and Industrial Research), Japan
2. Osaka University, Institute for Advanced Co-Creation Studies, Japan

Visible-light responsible titania nanotubes (TNTs) could be modified by peroxo functional groups via facile solution chemical routes by hydrogen peroxide treatment of pristine TNT and via one-step bottom-up synthesis from peroxo titanium complex precursor. The introduction of peroxo groups decreased band gap energy of titania down to 2.6 eV that was lower than that of pure TNTs. Further materials tuning could also be possible by cation doping to enhance optical absorption properties. The photocatalytic performance of the modified TNTs was enhanced under the visible light irradiating. The active species were identified by using various radical trap agencies, and it was found that the photo-exited electron played a major role for visible-light driven photocatalytic degradation of organic molecules. Such photo-induced functions might be expected to contribute not only to environmental cleaning but also to application as hygienic components. Detailed synthesis processes, formation mechanisms, physical, chemical and photochemical properties will be discussed.

S1: Mechanical Behavior and Performance of Ceramics & Composites

Processing-Microstructure-Mechanical Properties Correlation II

Room: Ballroom 5 (South Tower)
Session Chairs: Prabhakar Singh, Indian Institute of Technology (BHU); Tetiana Prikhna, Institute for Superhard Materials of the National Academy of Sciences of Ukraine

1:30 PM

(ICACC-S1-051-2023) Correlating the extent of thermal exfoliation of graphene oxide with dispersibility/reinforcing ability of reduced-graphene oxide in glass-ceramics

V. Verma*[1]; L. Gurnani[1]; A. Mukhopadhyay[1]

1. Indian Institute of Technology Bombay, Metallurgical Engineering and Materials Science, India

Thermal reduction of graphene oxide (GO) to reduced graphene oxide (rGO) is a scalable and economically viable method to obtain few-layers graphene, a potential structural reinforcement in ceramic/glass-ceramic matrices. This work highlights the influences of heating rate (during thermal reduction of GO) on the degree of exfoliation and defect-structure of the resulting rGOs, and further explores the abrasive wear behaviour of alumina-borosilicate (ABS) glass-ceramics reinforced with differently exfoliated rGOs. Faster heating (at 10°C/min) of GO to 350°C led to an efficient thermal exfoliation, yielding rGO (R10) with ~400% increase in surface area (w.r.t.GO). Contrarily, slower heating (at 1°C/min) caused a reduction in surface area of rGO (R01) by ~30% (w.r.t. GO), implying unsuccessful exfoliation, despite R01 exhibiting a defect density, L_d~1.22 nm (as determined from Raman spectroscopy) similar to that of R10 (L_d~1.15 nm). Incorporation of 2.5 vol.% R10 in ABS glass-ceramic led to ~33% reduction in abrasive wear rate as compared to unreinforced ABS. In stark contrast, incorporation of 2.5 vol.% of R01 resulted in ~39% increase in wear rate, owing to inefficient exfoliation post thermal reduction, leading to poor dispersibility in, and weaker interface with ABS glass-ceramic matrix.

1:50 PM

(ICACC-S1-052-2023) Shape Memory Ceramics Under Stress

H. Ozcan*[1]; E. Pang[1]; C. A. Schuh[1]

1. Massachusetts Institute of Technology, Department of Materials Science and Engineering, USA

As a new emerging class of shape memory materials, Zirconia (ZrO_2)-based shape memory ceramics offer unique properties that may not be achievable with their metallic counterparts; higher martensitic transformation temperatures with excellent oxidation resistance, higher transformation stresses, and higher work outputs than all currently existing actuators. However, transformation-induced cracking and large hysteresis of polycrystals pose a challenge to fully utilizing these beneficial properties. This talk will review work in our group that has focused on 1) uncovering complex mechanisms behind transformation-induced cracking and strategies to mitigate this effect by carefully tuning the crystallography of the transformation and 2) controlling factors and fundamental mechanisms behind thermal transformation hysteresis. This talk will also extend these topics to the case of transformation under stress. The details of some of the important transformation characteristics – the evolution of transformation temperatures, hysteresis, and transformation strain – will be described in terms of thermodynamics, variant selection, and inherent crystallographic parameters of the martensitic transformation. The introduction of stress-dependence to these topics provides a path towards understanding the potential of shape memory ceramics as solid-state actuators.

2:10 PM

(ICACC-S1-053-2023) Ceramic upside-down composites for electronic applications

N. Kuzmic*[1]; M. Nelo[2]; S. D. Škapin[1]; H. M. Jantunen[2]; M. Spreitzer[1]

1. Jozef Stefan Institute, Advanced Materials Department, Slovenia
2. University of Oulu, Microelectronics Research Unit, Finland

Dielectric ceramics are widely used in various electronic devices. Due to high energy requirement in their large-scale production, tendency to go greener is becoming more apparent. One way to reduce processing temperatures is Room Temperature Fabrication (RTF) of upside-down composites, where high loading of functional ceramic filler and a small amount of binder in the form of a solid and saturated aqueous solution are combined. In our study, a composite system made from strontium titanate $SrTiO_3$ (ST) and lithium molybdate Li_2MoO_4 (LMO) was investigated. As the LMO binder deposits on the surface of ST filler particles during pressing and drying, densification takes place. LMO was later replaced with sodium- and magnesium-based salts, and water-soluble polymers resulting in sufficient binding (relative density ≈ 85 %), relative permittivity (65-130), and dielectric loss tangent values (0.002-0.05) at 1 MHz. RTF processing parameters were optimized to improve the dielectric performance of the composites, which were characterized with microstructural, mechanical, FTIR, and XRD analysis. In addition, the influence of the residual porosity on dielectric and mechanical properties was also studied by mixing mathematical rules and simulations. Its negative effect was partially compensated by impregnation. RTF entails almost infinite possibilities of combining different materials for a large variety of applications.

2:30 PM

(ICACC-S1-056-2023) Processing-Microstructure-Property Characterization of 3D-Printed Metal-Reinforced Polylactic Acid and Acrylonitrile Butadiene Styrene Composites

M. Ranaiefar*[2]; M. Singh[1]; S. Gupta[3]; J. Salem[2]; M. C. Halbig[2]

1. Ohio Aerospace Institute, USA
2. NASA Glenn Research Center, USA
3. University of North Dakota, USA

The expansive utility of polymeric 3D printing technologies and demand for high-performance lightweight structures has prompted the emergence of various metal-reinforced polymer composite filaments. However, detailed characterization of the processing-microstructure-property relationships of these materials is still required to realize their full potential. In this study, both PLA (polylactic acid) and ABS (acrylonitrile butadiene styrene) serve as blueprints for metallic reinforcements (Magnetic Iron, Stainless Steel, Bronze, Copper, Bismuth, Tungsten), and the properties of these polymeric composites are investigated across multiple print variables. Microscopy of material cross-sections is conducted to evaluate the relationship between print parameters, porosity, and the polymer matrix with metal reinforcement. The dependence of tribological behavior on print parameters and particulate additives is also investigated. Additionally, mechanical properties are evaluated through compression testing, demonstrating the potential of variant metal-reinforced PLA and ABS polymer composites for novel 3D printed structures.

3:10 PM

(ICACC-S1-057-2023) Study on Thermal Conductivity and Mechanical Properties for Polyamide-11 Composite Including Alumina Particle

M. Ijiri*[1]; S. Ishida[1]; T. Osada[1]; S. Kobayashi[1]

1. Tokyo Metropolitan University, Japan

Polyamide 11 (PA11) has excellent durability and chemical resistance, but low heat resistance and mechanical strength. In this study, in order to improve heat resistance and mechanical properties, a composite material was prepared using Al_2O_3-particles as a filler. The density of the samples with a content of 40 vol% or more decreased in the samples molded with a manual injection molding machine. In the samples molded by a manual injection molding machine, the Young's modulus and tensile strength of the samples containing more than 40 vol% alumina decreased, but the thermal conductivity increased. The decrease in density and tensile strength was caused by the presence of gaps at the interface between the filler and the resin due to the microstructure. When the injection molding machine was changed to a hydraulic type, the density, Young's modulus and tensile strength of the samples containing more than 40 vol% were improved.

3:30 PM

(ICACC-S1-058-2023) Thermal and Mechanical Analysis of Epoxy Matrix Composites with Abaca and Pineapple Leaf Fiber Reinforcements

R. A. Sirot*[1]; R. E. Alemania[1]; E. d. Magdaluyo[1]

1. University of the Philippines Diliman, Department of Mining, Metallurgical, and Materials Engineering, Philippines

Fiber-reinforced polymer (FRP) composites are continuously being used in various applications because of their superior properties from the polymer matrix and fiber reinforcements. Epoxy resin has been established among the most preferred matrices due to its low cost-to-performance ratio whereas natural fibers are consistently being studied because of their sustainability over the synthetic ones. Abaca fibers and pineapple leaf fibers (PALF) are two of the most utilized natural fibers in the Philippines given their abundance and excellent thermal and mechanical properties. The thermo-mechanical properties of abaca fiber and PALF reinforced epoxy composites fabricated via vacuum resin infusion were evaluated. Dynamic mechanical and thermogravimetric analyses showed that PALF exceeded abaca as reinforcement in terms of thermal properties by having a higher decomposition temperature of 406.29°C compared to the 396.84°C of abaca. The mechanical tests proved that the warp untreated pineapple leaf fiber orientation has higher tensile and shear strength of 68.1 MPa and 39.3 MPa than their weft counterparts having 24.9 MPa and 14.4 MPa, respectively. Acetylation treatment also provided better properties. Further, the research aims to contribute to the utilization of local fibers for research and development, and localization of FRP fabrication from indigenous natural fibers.

S3: 20th International Symposium on Solid Oxide Cells (SOC): Materials, Science and Technology

Degradation

Room: Ponce de Leon (North Tower)
Session Chairs: Marie-Laure Fontaine, SINTEF AS; Tae Ho Shin, Korea Institute of Ceramic Engineering & Technology

1:30 PM

(ICACC-S3-053-2023) Materials Interfacial Stability in Solid Oxide Cells (Invited)

S. Barnett*[1]

1. Northwestern Univ, USA

Solid oxide cell long-term stability is affected by a number of factors, including materials interfacial stability, impurities, and electrode morphological stability. Here the focus is on materials interfacial stability. Some effects, such as reactions between the electrodes and electrolyte (e.g. oxygen-electrode/YSZ reactions) occur at cell firing and/or operating temperature, but do not necessarily result from cell operation. Other effects, such as surface segregation (e.g., Sr segregation on oxygen electrodes), are temperature dependent but can also depend strongly upon cell current/overpotential. Some effects occur only at extremely low or extremely high oxygen partial pressures present during electrolysis operation – these include the destabilization of Ni-YSZ fuel electrodes and fracture at oxygen-electrode/electrolyte interfaces. This talk will review these phenomena and attempt to assess their importance in achieving high performance SOCs with excellent long-term stability.

2:00 PM

(ICACC-S3-054-2023) Pt Current Collectors Artificially Boosting Si-Contaminated Praseodymium Doped Ceria Oxygen Surface Exchange Coefficients (Invited)

Y. Ma[1]; T. E. Burye[1]; J. D. Nicholas*[1]

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

In an attempt to rank the oxygen exchange catalysts used in Solid Oxide Fuel Cells, Solid Oxide Electrolysis Cells, redox-enabled solar concentrators, catalytic converters, and other oxygen-exchange-enabled devices, for the past 90 years the scientific community has used precious metal current collectors to perform a variety of electrically-based chemical oxygen surface exchange coefficient (k_{chem}) measurements. Unfortunately, these attempts have yielded a k_{chem} reproducibility crisis where the k_{chem} values of “identical” materials under “identical” conditions can vary by 4 orders of magnitude, or more. Here, in situ, non-contact, current-collector-optional Curvature Relaxation k_{chem} measurements on $Pr_{0.1}Ce_{0.9}O_{2-x}$ (PCO) thin films show that unpolarized Pt current collectors covering < 3% of the PCO surface can quadruple the 650°C PCO k_{chem}. Similarly, unpolarized Pt current collectors covering the entire PCO surface can boost the 500°C PCO k_{chem} by 2 orders of magnitude. Secondary Ion Mass Spectroscopy analyses suggest these k_{chem} enhancements are related to current-collector-induced reductions in the PCO surface Si concentration and/or Pt diffusion into the PCO. These, and other literature results, suggest that extreme caution must be used when performing Electrochemical Impedance Spectroscopy and/or Electrical Conductivity Relaxation k_{chem} measurements.

2:30 PM

(ICACC-S3-055-2023) Atomically-flat $SrTi_{0.5}Fe_{0.5}O_{3-\delta}$ Model Thin Film – a Case Study of Activity Degradation of an Ideal Perovskite Surface (Invited)

W. Jung*[1]

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

Perovskite oxides with mixed ionic and electronic conductivity are promising electrode materials used in many electrochemical applications such as solid oxide fuel/electrolysis cells, oxygen permeation membranes, and metal-air batteries. However, their surfaces are often not structurally or chemically stable, and the resulting degradation in electrode performance remains a severe problem. This study presents an atomically flat model thin film with an ideal stoichiometric surface. $SrTi_{0.5}Fe_{0.5}O_{3-\delta}$ (STF) is chosen as a model mixed conducting perovskite oxide. We fabricate thin films of STF via PLD on a pretreated single-crystal $SrTiO_3$ substrate, and observe changes in the surface structure, composition, and oxygen exchange kinetics according to temperature and time, with a focus on the surface Sr segregation phenomenon, through angstrom-level analytical techniques (e.g., AFM, APT, LEIS and HR-TEM). Combined with computational calculations, these observations make it possible to answer fundamental questions such as 1) how reactive is an ideal perovskite oxide surface? 2) can we quantify how much Sr is segregated on the surface? 3) what is the quantitative relationship between the degree of Sr segregation and the surface oxygen exchange rate?

3:20 PM

(ICACC-S3-056-2023) Exsolution of phase-separated nanoparticles via trigger effect toward reversible solid oxide cell (Invited)

J. Myung*[1]

1. Incheon National University, Dept. of Materials Science and Engineering, Republic of Korea

The electrocatalytic characteristics of heterostructure nanoparticles have attracted attention in energy and environment devices. Among the various features, exsolved alloy or core–shell nanoparticles display high reactive surface area and strong interaction between metal and substrate oxide. Herein, we report multifunctional heterostructure nanoparticles; Fe/Cu Janus nanoparticle (JN) and verify an exsolution trigger effect using Cu as a seed in a $La_{0.43}Sr_{0.37}Fe_{0.09}Cu_{0.06}Ti_{0.88}O_{3-\delta}$ (Fe75) perovskite substrate. During the exsolution process, the exsolved Cu particles played a key role in triggering the Fe exsolution by contributing to the additional surface energy. These Fe/Cu JNs exhibited enhanced the catalytic activity in the H_2O/H_2 and CO/CO_2 atmosphere. Solid oxide reversible cell with Fe75 showed impressive current density of -1.11 A cm^{-2} at 1.3 V and 900 °C. Our study provides the experiment-based mechanism of the trigger effect of co-exsolution and a multifunctional catalyst using Fe/Cu JNs.

3:50 PM

(ICACC-S3-057-2023) Mitigating nitridation in ammonia fueled SOFCs

H. L. Frandsen*[1]; D. G. Marin[1]; F. Mondi[1]; A. Nemati[1]; H. Nami[1]; C. Goebel[1]; M. Chen[1]

1. Technical University of Denmark, Department of Energy Conversion and Storage, Denmark

The limitation to how much land we can utilize for energy crops will impose limitations on how much biomass we can use for production of carbon-based green fuels. Consequently, ammonia is attracting attention as a probable future fuel. SOFCs systems with very high efficiency are currently being developed, and there is a great chance that these can compete with the highly efficient ammonia fuelled internal combustion engines. 4 stroke engines prototypes for maritime use have already been developed and 2 stroke engines are in the progress. In this talk we show a conceptual design of an ammonia fuelled solid oxide fuel cell system, which will be able to reach a very high efficiency, while also addressing a main concern of nitridation occurring when feeding SOFCs with ammonia. We will show simulations through multiphysics models with the aim to uncover the operational regimes, where nitridation is not a challenge. These will be supplemented with experimental investigations on the same. Finally, we will show how this can be realized with high efficiency in a system configuration.

4:10 PM

(ICACC-S3-058-2023) Electrochemically-Induced Degradation of Solid Oxide Electrolyzer Cells (SOECs)

P. Elahi*[1]

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

Most active current-carrying electrochemical devices, such as fuel/electrolyzer cells or batteries degrade under various operating conditions. The observation of electrochemical device degradation suggests that there may be a common underlying reason which is applicable to all such electrochemical systems. Thermodynamic instability may occur in electrolyzer cells (or fuel cell stacks) during operation. In many studied cases, the degradation in electrolyzer cells manifests as electrode delamination at the electrode/electrolyte interface, and/or complete delamination of the electrolyte occurs by crack nucleation and growth adjacent to the fuel electrode. Degradation in these devices may manifest as an increase in resistance, loss of capacity, or both. The degradation of the active current-carrying systems can be understood using linear non-equilibrium thermodynamics. In this work, we theoretically and experimentally study the degradation of such electrochemical devices, which occur under coupled transport of mobile species. A key conclusion is that the chemical potential of an electrically neutral species within the electrolyte corresponding to the mobile ion may lie outside the range covered by the values at the electrodes (reservoirs), which can lead to transport-induced instability. Specific examples of solid electrolytes in electrolyzer mode will be addressed.

4:30 PM

(ICACC-S3-059-2023) Exploring the Safe Operational Current Density for High Temperature Solid Oxide Electrolyzers

K. Huang[1]; Y. Wen*[1]

1. University of South Carolina, Mechanical Engineering, USA

High temperature water splitting through solid oxide cells (SOCs) under electrolysis mode has fundamental advantage in efficiency and yield over its low temperature counterparts. However, high temperature solid oxide electrolytic cells generally exhibit faster performance degradation than solid oxide fuel cells, thus presenting a major challenge to scale up the technology to the hydrogen market. One leading cause for the degradation is the delamination of oxygen electrode (OE) under high current densities. In this presentation, we show our effort to determine conditions under which OE can safely operate without delamination. We particularly show application of DC-biased electrochemical impedance spectroscopy (EIS) method to a three-electrode symmetrical cell as a means of delineating OE polarization resistance R_P and overpotential h as a function of current density and time under both oxygen evolution reaction (OER) and oxygen reduction reaction (ORR), from which the intrinsic exchange current density (i_o) is extracted using Butler-Volmer equation. We then correlate the obtained i_o with time-to-delamination (TTD) determined by using the ohmic resistance (R_{ohm}) as an indicator of delamination. We finally establish the boundaries for OE to safely operate without delamination under electrolytic operation.

4:50 PM

(ICACC-S3-060-2023) Durability Comparison of Electrolyte Supported Solid Oxide Cells Operated During 10,000 to 50,000 Hours as Steam Electrolyser

J. Schefold*[1]; A. Léon[1]

1. European Institute for Energy Research, EIFER, Germany

Steam electrolysis with solid oxide cells (SOC) shows the highest energy-conversion efficiency among the different electrolysis technologies, and prototype upscaling to the MW range and beyond is currently under way (e.g., H2GIGA project in Germany) for a coupling with renewable energy sources. Such application relies on the availability of cells with appropriate durability under the different operation conditions (constant or intermittent power). In that context, EIFER has performed long-term cell tests with up to 50,000 h duration with electrolyte supported cells using Ni/GDC hydrogen electrodes. Impedance spectroscopy was applied as in-situ analytical method. In general, the tested cells showed a sufficiently low degradation, even for elevated current densities in the 1 A/cm^2 range. This implies a high degree of SOFC/SOEC reversibility. The main contribution to degradation was ohmic, which indicates that the decay of ionic electrolyte conduction is significant, notably for electrolytes of high ionic conductivity (e.g., 6/10Sc1CeSZ electrolytes). A comparison of the tests with different current densities, electrolytes and electrolyte thickness, as well as varying steam conversion will be presented for the assessment of degradation. Moreover, post-test analysis with classical and advanced methods will be included for part of the samples.

5:10 PM

(ICACC-S3-061-2023) Review on Fault Diagnosis and Prognosis of Solid Oxide Fuel Cells

N. Ghulamullah*[1]; Y. Du[2]

1. Kent State University, Mechatronics, USA
2. Kent State University, USA

Solid oxide fuel cells (SOFCs) are a potential candidate to replace the conventional fossil fuel-based energy generations, but SOFC progress is hindered due to its performance durability, reliability and cost. A SOFC system undergoes many degradations during its lifetime which makes it expensive and less reliable than conventional sources. The overall performance degradation in power drawn is reliant on balance of plant components. The malfunctioning of any of the component can cause a devastating loss to expensive SOFC stack as well as can cause serious hazards to the operator. To make the overall system operating in normal conditions, a well-organized monitoring system is required and in case of any anomaly can detect and direct instructions. Fault diagnosis and prognosis are key for the sustainable performance of SOFC. Moreover, prognostics of SOFC are also important for tracking the health of the system while running. This attempt is made here, to sum up, the diverse types of degradations, faults and their diagnosis and prognosis available in the literature. Using real systems for fault diagnosis is awfully expensive and risky so many attempts are made on diagnosis and prognosis using mathematical models. The model suitability for fault diagnosis is discussed. Bibliometric analysis is performed on the available literature and future research areas are suggested.

5:30 PM

(ICACC-S3-068-2023) On the optimal energy integration and lifetime of solid oxide cells: Towards a wholistic multi-scale optimization approach (Invited)

R. Nogueira Nakashima*[1]; S. de Oliveira Junior[2]; H. L. Frandsen[3]

1. Technical University Darmstadt, DTU Energy, Denmark
2. University of São Paulo, Mechanical Engineering, Brazil
3. Technical University of Denmark, Department of Energy Conversion and Storage, Denmark

Solid oxide cells (SOCs) are the technological frontier of energy conversion, allowing a highly efficient interchange between chemical energy and electricity. However, the efficiency of systems employing SOCs significantly depends on the integration of heat. Furthermore, operational strategies and plant design are strongly connected with the overall lifetime of the stacks and cells. Thus, it is necessary an overall holistic optimization connecting operation with local degradations throughout multi-scale modeling. In this work, a linear optimization problem in conjunction with multi-scale modeling is proposed as a method to solve this problem. For instance, details of temperature and composition distribution can be used to pinpoint overpotential losses to different physical phenomena in local points of the stacks. Moreover, the influence of operational parameters and system design in the degradation of stacks can be assessed and optimized in order to maximize the equipment lifetime. A number of different applications of this mathematical technique are presented in order to illustrate the different insights that this state-of-the-art method can provide. In addition, the inclusion of dynamic operation and degradation effects are proposed as the next steps towards a wholistic optimization approach.

S4: Armor Ceramics - Challenges and New Developments

Army Outreach: Funding Opportunities at the Army Research Office

Room: Coquina Salon B (North Tower)
Session Chair: Jerry LaSalvia, DEVCOM Army Research Laboratory

1:30 PM

(ICACC-S4-019-2023) Extramural basic research funding opportunities at DEVCOM ARL Army Research Office (Invited)

M. P. Bakas*[1]; C. Varanasi[1]

1. U.S. Army Research Lab DEVCOM, USA

In this joint presentation by Dr. Pani Varanasi and Dr. Mike Bakas, the speakers will initially discuss new competency based structure of the Army Research Laboratory. They will outline the goals and programs of the Science of Extreme Materials Competency, and discuss opportunities to conduct extramural research supporting the competencies under the Army Research Office's Programs. Topics that are particularly of interest to early career researchers such as how to make initial contact and engage ARO program managers and how to submit white papers/proposals will also be discussed.

Dynamic Response and Failure of Ceramics and Glasses I

Room: Coquina Salon B (North Tower)
Session Chair: Jerry LaSalvia, DEVCOM Army Research Laboratory

2:00 PM

(ICACC-S4-020-2023) Static and Dynamic Compression Strength of Advanced Ceramics

J. Swab*[1]; J. J. Pittari[1]; C. Meredith[1]

1. Army Research Laboratory, USA

Many of the modeling and simulation packages that predict the impact performance of ceramic armor systems include compression strength of the advanced ceramic component as one of the input parameters. As a result, it is imperative that the compression strength of ceramics being considered for this application be properly measured. Cuboids and cylinders are the most common specimens used to determine compression strength but they typically generate misleading low strength values due to axial cracking that initiates at the specimen/loading platen interface and leads to failure. In the late 1980s a dumbbell-shaped specimen was designed to minimize the stresses at this interface and promote fracture in the gauge section. Recently a scaled-down version of this dumbbell specimen has successfully been used to generate compression strength data for a wide range of ceramics under quasi-static (10^{-2}/s) and dynamic (10^{2}/s) strain rates. This includes monolithic ceramics and cermets as well as a heterogenous B_4C-SiC material fabricated by additive manufacturing. The results clearly show that the dumbbell specimen is the most appropriate specimen geometry for generating compression strength of these brittle materials over this strain rate regime. It significantly reduces failure due to axial splitting and yields strength values that are consistently higher than those generated with cuboids or cylinders.

2:20 PM

(ICACC-S4-021-2023) Dynamic Knoop Indentation of Ceramics using a Miniature Kolsky Bar Method

D. T. Casem*[1]; E. Retzlaff[4]; J. Swab[2]; M. Mello[3]

1. CCDC Army Research Laboratory, USA
2. Army Research Laboratory, USA
3. CalTech, USA
4. US Naval Academy, USA

A novel dynamic indentation method is used to conduct Knoop indentation on ceramics. The method is a modified Split Hopkinson Pressure Bar. Loading times are in the range of 8-15 us and the load range spans 5 – 40 N. Three important features of this method are (1) the ability to obtain low loads (< 20 N), (2) reliable recovery of the sample after a single loading cycle, and (3) high temporal resolution sufficient for the short loading durations. To achieve these goals, miniature bars (diameters as small as 1.2 mm) are used, along with optical instrumentation with Michelson-type displacement interferometers. Loads at the lowest end of the range (~ 5 N) use a novel phase-multiplied normal displacement interferometer (NDI) with doubled measurement sensitivity. The method is applied to Si3N4, Al2O3, TiB2, and B4C.

Dynamic Response and Failure of Ceramics and Glasses II

Room: Coquina Salon B (North Tower)
Session Chair: Daniel Casem, CCDC Army Research Laboratory

3:20 PM

(ICACC-S4-022-2023) Dynamic Failure of Acrylic Glass and Glass Ceramic under Multiaxial Loading (Invited)

L. Zhang*[1]; D. Townsend[1]

1. University of Oxford, Department of Engineering Science, United Kingdom

Studying the dynamic failure of glass and ceramic will guide the impact-resistant design of aircraft windshield and armour systems. Firstly, with acrylic glass as a model material, the multiaxial loading is applied by using the thin metallic ring and the Hopkinson bar technique. The critical strain rate is determined, at which the acrylic shows brittle fragmentation. The critical multiaxial stress state is identified to achieve the brittle-to-ductile transition of acrylic. The high speed photography shows the melting nanofiber during the brittle-to-ductile transition in real time. The failure mode is identified as adiabatic shear failure. Next, with Macor (Corning Inc.) as a model ceramic, the multiaxial loading technique is extended to ceramic. The dynamic fracture of Macor is also investigated by using the Brazilian disk technique. Under carefully controlled laboratory condition, the brittle-to ductile-transition of ceramic and the corresponding fine particles in the shear zone are reported. Thirdly, an effort is made to investigate the dynamic shear compressive response of Macor, by using the cone specimen and inclined specimen. The equivalent stress at the failure initiation and the shear stress are nonlinearly related. The high speed images show that the crack originates from the minimum cross-section of the cone specimen and the obtuse angle corner of the inclined specimens.

3:50 PM

(ICACC-S4-023-2023) Computer modeling of impact of hybrid protective panel with a shock absorber layer made from aluminum foam granules and discrete reinforcing elements

E. Kartuzov*[1]; V. Kartuzov[1]; O. Mikhailov[1]; A. Mikhailov[1]

1. IPMS NASU, Ukraine

An impact of a hybrid protective panel made from compact ceramic and metal layers and a shock-absorbing layer of foamed aluminum granules and discrete reinforcing elements, is investigated by computer simulation. Particles of metals and ceramics were used as reinforcing elements. A behavior of compact ceramic and metal layers was studied by the finite element method. Modeling of the impact of shock-absorbing layer was carried out by a combined method that involves continual and discrete approaches. The material's response was determined at two scale levels. Deformation of each particle was studied in terms of continuum approach and the finite element method. The dynamic response of protective panel of shape and initial porosity of particles involved in shock-absorbing layer, a type of packing and friction conditions among those, physical properties of reinforcing elements, as well as a number of other parameters has been studied. Computer modeling showed that the value of initial porosity of foamed aluminum granules has a significant influence on dynamic response of the protective panel. A decrease in porosity brings to a decrease in penetration depth of a projectile. As the initial porosity increases, the effect on bottom plate of protective panel decreases, yet it is necessary to use a shock-absorbing layer of bigger thickness.

S6: Advanced Materials and Technologies for Rechargeable Energy Storage

Sodium Batteries, Potassium Batteries, Magnesium Batteries and Calcium Batteries I

Room: Coquina Salon E (North Tower)
Session Chairs: Moulay Tahar Sougrati, CNRS ICGM; Sergio Brutti, Università di Roma La Sapienza

1:30 PM

(ICACC-S6-043-2023) New Approaches and Materials for Na-ion Batteries (Invited)

M. Fichtner*[1]

1. Helmholtz Institute Ulm, Germany

Na ion batteries have prospects as sustainable solutions for electrochemical energy storage. The use of Na+ rather than Li+ ions as shuttle open possibilities to replace expensive and partially rare or critical transition metals such as Cu, Co, and Ni by other, abundant and non-toxic elements. Moreover, the (predicted) lower cost, the high efficiency and the favorable low temperature behavior offer additional, commercial and technical advantages. First batteries have been commercialized by various companies, however, their properties are still not satisfactory, mostly because the combination of high storage capacity and cycle life needs to be improved. The talk will deal with a new approach to stabilize layered cathode materials based on Mg and Mn oxides and on a novel class of inorganic binders which are ionically conducting, have excellent thermal properties and binder capabilities and can be used in water-based processes.

2:00 PM

(ICACC-S6-044-2023) Facile Wet-Processes of $MgMn_2O_4$ Spinel Nanoparticles Synthesis for Magnesium Rechargeable Batteries (Invited)

H. Kobayashi*[1]

1. Tohoku University, Institute of Multidisciplinary Research for Advanced Materials, Japan

High energy and high density rechargeable batteries are indispensable for widespread portable electronic devices and the spreading of electric vehicles. Mg rechargeable batteries (MRBs) have gained much attention as promising alternatives to Li-ion batteries due to the high natural abundance, high volumetric energy density, and no dendrite formation of the Mg metal anode. The prototype MRBs using sulfide cathodes operate low terminal voltages, therefore development of oxide-based high-voltage cathodes is promptly required. Among various oxide cathode candidates, spinel oxides have a high redox potential and a relatively high ion-diffusivity. Especially in the common electrochemical window of electrolytes, $MgMn_2O_4$ spinel can exhibit a high theoretical capacity of 540 mAh g^{-1} using both Mn^{3+}/Mn^{4+} and Mn^{3+}/Mn^{2+} redox reactions (λ-MnO_2 formation and rock-salt $Mg_2Mn_2O_4$ formation reactions, respectively). One of the problems in oxide-based cathode materials is very low rate capability at cathodes due to much slower diffusion of Mg^{2+} ions in solids than Li^+ ions. To solve this, reducing particle size of cathode material is effective to improve the rate capability. Herein, we report ultraporous and ultrasmall cubic spinel $MnMn_2O_4$ prepared by various wet-processes, enabling room temperature MRB operation.

2:30 PM

(ICACC-S6-045-2023) From bulk alloys to surface coatings: Developing performing magnesium negative electrodes (Invited)

C. Pechberty[1]; L. Stievano*[1]; R. Berthelot[1]

1. Université de Montpellier, Institut Charles Gernardt Montpellier, France

The surface passivation of magnesium metal anodes with conventional battery electrolytes is one the main hurdles hampering the development of performing high-energy magnesium batteries. Several strategies have been proposed to contrast this drawback, mainly by designing of complex, corrosive and/or difficult-to-handle electrolytes which are not adapted for large-scale production. An alternative strategy allowing the use of conventional electrolyte is the development of alloy anodes. This approach, however, strongly reduces the stored energy of the battery, making it less competitive compared to other technologies such as Li-ion batteries. If the alloy anode is reduced to a thin surface layer on top of the magnesium electrode, however, such a loss is energy does not occur. In this work, we present the surface protection of Mg metal anodes by alloy coatings prepared from liquid metals. Such alloy interphases hinder Mg metal passivation and enable stable plating/stripping of Mg with conventional $Mg(TFSI)_2$/DME electrolytes. Such an alloy-based surface protection opens the door to the use of electrolytes previously considered as non-compatible with magnesium metal, thus paving the way to the application of magnesium metal electrodes in practical magnesium batteries.

Sodium Batteries, Potassium Batteries, Magnesium Batteries and Calcium Batteries II

Room: Coquina Salon E (North Tower)
Session Chairs: Shih-kang Lin, National Cheng Kung University; Hiroaki Kobayashi, Tohoku University

3:20 PM

(ICACC-S6-046-2023) Solid state NMR characterization of $Na_3V_2(PO_4)_2F_{3-y}O_y$ positive electrode materials for Na-ion batteries (Invited)

D. Carlier*[1]

1. CNRS, University Bordeaux, France

In the scope of finding new positive electrode materials for Li-ion and Na-ion batteries, the deep understanding of the link between their structure, electronic structure and electrochemical behavior is crucial. To that extent, Magic Angle Spinning Nuclear Magnetic Resonance (MAS-NMR) appeared to be a key tool, as for paramagnetic materials, it allows to probe both, the local structure and the local electronic structure thanks to the hyperfine interaction. Using ^{23}Na and ^{31}P MAS-NMR we studied the $Na_3V_2(PO_4)_2F_{3-y}O_y$ series, which are promising materials for positive electrode application Na-ion batteries. We showed that the end member $Na_3V_2(PO_4)_2F_3$ exhibits some O-defects leading to the formation of V^{4+} ions locally. NMR combined with DFT calculations, allows to characterize the very specific electronic configuration around V^{4+} ions forming a vanadyl-type bond with the O-defect, that affects the electrochemical cycling performances. Na^+ ions deintercalation mechanism was also investigated by ^{23}Na and ^{31}P MAS NMR for two different systems: $Na_xV_2(PO_4)_2F_3$ and $Na_xV_2(PO_4)_2F_2O$. Finally we investigated the thermal stability of a series of $Na_xV^{3+}_{2-y}V^{4+}_y(PO_4)_2F_{3-y}O_y$ ($0 \leq y \leq 2$) materials in order to learn more about the influence of F^- for O^{2-} substitution and the state of charge. NMR studies were crucial to understand the degradation mechanisms.

3:50 PM

(ICACC-S6-047-2023) Impact of Li and Ni doping on P2-Na_xMnO_2 cathodes for Sodium-Ion Batteries

N. Yaqoob*[1]; P. Kaghazchi[2]

1. Forschungszentrum Juelich, Material Synthesis and Manufacturing Processes (IEK-1), Germany
2. Forschungszentrum Juelich, Germany

Oxygen-redox-based cathode materials for sodium-ion batteries have attracted significant attention in recent years owing to the possibility of delivering additional capacity in the high-voltage region. However, P2-type Na_xMnO_2, an oxygen-redox cathode material, suffers from fast capacity fading and structural instability during the charging and discharging process. In this work, a combination of Coulomb energy analysis and DFT calculations was used to study the influence of Li and Ni doping on the stability and redox mechanism of P2-Na_xMnO_2 cathodes. We characterized the redox mechanism by computing the magnetic moments and spin density differences. We found that the co-doping of Li and Ni in P2-Na_xMnO_2 improves structural stability by reducing Li migration from transition metal to Na layers. Moreover, our study demonstrates that Ni suppresses the O redox, but due to the higher structural stability and Ni-redox activity, $Na_{0.75}[Li_{0.15}Ni_{0.15}Mn_{0.7}]O_2$ has a better performance in comparison to $Na_{0.67}[Li_{0.22}Mn_{0.78}]O_2$.

4:10 PM

(ICACC-S6-048-2023) Optimized electric-energy storage in $BiFeO_3$-$BaTiO_3$ ceramics via microstructure tailoring

R. H. Montecillo*[1]; C. Chen[2]; Y. Lee[3]; P. Chen[1]; C. Tu[4]

1. Ming Chi University of Technology, Innovative Technology of Biomedical Engineering and Medical Devices, Taiwan
2. Hwa Hsia University of Technology, Department of Mechanical Engineering, Taiwan
3. Ming Chi University of Technology, Department of Mechanical Engineering, Taiwan
4. Fu Jen Catholic University, Department of Physics, Taiwan

This study demonstrates the high energy-storage performance using 0.1 wt.% MnO_2–added $0.7(Bi_{1-x}Sm_xFeO_3)$-$0.3(BaTiO_3)$ ($x = 0$-0.3) ceramics through tailoring microstructures and polar order. Sequentially substitution-driven structural transitions were identified from a co-occurrence of nonpolar pseudo-cubic Pm-3m and ferroelectric rhombohedral R3c symmetries to antipolar orthorhombic Pbam and nonpolar orthorhombic Pnma symmetries as Sm substitution increases. Recoverable energy densities (W_{rec}) of ~4.5 J/cm^3 and ~4.1 J/cm^3 with efficiencies (η) of ~62.1% and 78.1% can be achieved respectively for x = 0.15 and 0.2 at a field of 220 kV/cm. The improved energy storage can be associated with microstructure modification and complex grain matrix, consisting of grain boundaries, nanoclusterstructures (or core-shell structures), and polar nano-regions. The nanocluster structure may act as a barrier to suppress polar order and to enhance dielectric breakdown strength. This work provides an efficient route to utilize binary $BiFeO_3$-$BaTiO_3$ ceramics for electrical energy storage.

S7: 17th International Symposium on Functional Nanomaterials and Thin Films for Sustainable Energy Harvesting, Environmental, and Health Applications

Nanomaterials for Thermoelectrics, Photocatalysis, Electrocatalysis

Room: Coquina Salon G (North Tower)
Session Chair: Sanjay Mathur, University of Cologne

1:30 PM

(ICACC-S7-009-2023) Green Chemical Syntheses of High-Efficiency Nanostructured Bi_2Te_3 and its Hybrids (Invited)

M. S. Toprak*[1]

1. KTH Royal Institute of Technology, Dept. of Applied Physics, Sweden

There is an increasing interest to develop hybrid/composite materials in the field of thermoelectrics (TEs). A pre-requisite for these efforts to succeed is to have a set of well-defined, large-scale and highly reproducible quality n- and p-type TE materials. Fabrication methods of materials influence their morphology and microstructure, which in turn strongly impacts the transport properties. We report on nanostructured bismuth telluride (Bi_2Te_3) using rapid bottom-up syntheses methods. The resultant materials' characteristics are compared in terms of microstructure and transport properties. The TE transport properties of these nanostructured Bi_2Te_3 are measured on compacted pellets, where the figure of merit, reaching unity were obtained for both the hydrothermal and polyol synthesized samples. The developed two rapid syntheses methods enable large scale production of TE nano powders with promising performance and with different surface chemistry, which are pre-requisites for the future composite TE materials and device developments. The synthesized TE nanoparticles were then used to fabricate hybrid films, using a common polymer as poly (methylmethacrylate) as the matrix into which the NPs are dispersed. Thick films were developed and evaluated of these formulations, revealing promising power factor values. Recent achievement in this arena are summarized in this talk.

2:00 PM

(ICACC-S7-010-2023) Performance Analysis of Phase Change Materials and Thermoelectric Module Integrated Concentrated Photovoltaic System (Invited)

A. Yusuf[1]; S. Ballikaya*[2]

1. Istanbul University, Elec. Engineering Dept., Turkey
2. Istanbul University-Cerrahpasa, Engineering Science, Turkey

Phase change materials (PCMs) are continuously being used to enhance the performance of concentrated photovoltaic (CPV) systems, and the performance can further be improved by incorporating a thermoelectric (TE) module into the system. This study analysed and compared the performances of standalone CPV, CPV-TE, CPV-PCM, CPV-TE-PCM, and CPV-PCM-TE systems. Among the five systems, the CPV operating in the CPV-PCM-TE system has the highest performance, this is as a result of the enhancement of the cooling performance by both the PCM and the TE and due to the low thermal resistance of the PCM which is in direct contact with the PV. Similarly, the TE couple operating in the CPV-TE-PCM system has the highest performance due to the large temperature gradient, which is a result of the absorption of high thermal energy from the PV and cooling enhancement by the PCM. Moreover, the CPV-TE-PCM system has the overall highest energy efficiency, this energy efficiency is 2.4% higher than that of the CPV-PCM-TE system. This shows that different configurations of the hybrid-cooled CPV systems can result in different output performances. Finally, the energy efficiency of the CPV-PCM-TE model with a cross-fin configuration of the PCM container is 0.025% higher than that of the model with a triangular-fin configuration.

Nanomaterials for Energy Conversion and Storage and Catalysis

Room: Coquina Salon G (North Tower)
Session Chair: Sedat Ballikaya, Istanbul University

3:00 PM

(ICACC-S7-011-2023) Solution grown Silicon Nanowires in High Mass Loadings from 3D Electrode Substrates for High Performance Lithium ion Batteries (Invited)

K. M. Ryan*[1]; s. Imtiaz[1]

1. University of Limerick, Chemical Sciences, Ireland

Silicon nanowires (Si NWs) have great promise as an anode material for lithium ion batteries (LIBs) due to their very high specific capacity. Achieving adequate mass loadings for binder free Si NWs have been restricted by low surface area, mechanically unstable and poorly conductive current collectors (CC), as well as complicated and expensive fabrication routes. Herein, we report a tunable mass loading and dense Si NW growth on a highly conductive, flexible, fire-resistant and mechanically robust 3D interwoven stainless steel fiber cloth (SSFC) using a simple glassware setup. The 3D SSFC CC facilitates dense growth of Si NWs where its open structure allows a buffer space for expansion/contraction during Li cycling. The Si NWs@SSFC anode displays a stable performance for 500 cycles with an average Coulombic efficiency of more than 99.5 %. Galvanostatic cycling of the Si NWs@SSFC anode with a mass loading of 1.32 mg.cm $^{-2}$ achieves a stable areal capacity of 2 mAh cm $^{-2}$ at 0.2C after 200 cycles

3:30 PM

(ICACC-S7-012-2023) New cathode materials for Li-S batteries (Invited)

C. Zhang[1]; A. Cabot*[1]

1. Catalonia Institute for Energy Research, Spain

Lithium-sulfur batteries (LSBs) have emerged as an exciting alternative to Li-ion batteries. However, the practical application of LSBs requires overcoming several challenges, including the electrical insulating character of sulfur and lithium sulfides, the severe volumetric variation during charge/discharge processes, the diffusion of soluble lithium polysulfides (LiPS) intermediates, and the slow redox kinetics of the LiPS conversion reaction. Several strategies have been developed to improve the performance of LSBs. In terms of materials, one effective approach is to host sulfur at the cathode in carbon-based materials with high conductivity. Polar materials are also used to strongly bind LiPS and efficiently confine them within the cathode, achieving notable improvements in the cycling stability. In terms of structure, porous nanomaterials have been demonstrated advantageous in LSBs, to mitigate the detrimental effect of the volume expansion and provide physical confinement for LiPS. Besides, the use of electrocatalysts to enhance LiPS redox kinetics has been demonstrated effective to improve LSB performance. In this talk, I will present our recent advances in the design and engineering of promising sulfur hosts, using materials with high electrical conductivity, significant polarity to ensure a strong polysulfide affinity, highly porous nanostructures and having high catalytic activity toward sulfide redox reactions.

4:00 PM

(ICACC-S7-013-2023) Nanostructured lead-free piezoelectric materials for energy harvesting and piezo-catalytic applications (Invited)

T. Fischer*[1]; A. Verma[1]; A. Ichangi[1]; S. Mathur[1]

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

Piezoelectric materials find widespread applications as ultrasonic transducers, piezo-actuators as well as capacitors, with lead zirconate titanate $Pb[Zr_xTi_{1-x}]O_3$ (PZT) and its derivatives being the most used materials classes for these applications. Lead-free alternatives with similar performance like potassium sodium niobate $K_xNa_{1-x}NbO_3$ (KNN) are emerging and widely investigated to replace lead containing piezomaterials in the near future. We report on the fabrication of KNN materials in both thin-film and fiber geometry using metal alkoxide precursors in sol-gel processing and by electrospinning. While fibers enable alternate geometries for materials integration, KNN thin-films were investigated as support for piezo-electrocatalytic nitrogen activation for room-temperature nitrogen production. The fabrication and performance of both KNN fibers and thin-films will be evaluated and their application as catalyst support for dinitrogen activation will be discussed.

4:30 PM

(ICACC-S7-014-2023) Ammonia synthesis based on piezo-assisted materials

B. Witulski*[1]

1. University of Cologne, Institute of inorganic chemistry, Germany

Although new innovations for sustainable chemical synthesis have made great development recently, finding technically workable answers to surpass fossil fuels remains difficult. A viable route to a green fuel in this context is electrochemical synthesis of ammonia (NH3) because of its high energy content (18.8 MJ/kg), cheap storage costs, and zero carbon footprint. Through the Haber-Bosch process, nitrogen fixation has been carried out for more than a century. This process requires high pressure (20 – 40 MPa) and temperatures (400 – 600°C). The industrial NH_3 synthesis requires 3-5% of the global natural gas production and consumes 1% of the global energy, which underlines the need of alternative NH_3 synthesis routes. Through mechanical vibrations piezoelectric materials effect the electrochemical processes accelerates the transfer of electrons from catalyst to molecular absorbates thereby improving the sluggish nitrogen reduction reaction (NRR) kinetics. The created transient dipoles in surface-absorbed non-polar N_2 molecule increases the N_2 coverage on the electrocatalyst surface. The feasibility of ($Na_{0.5}K_{0.5}NbO_3$) KNN deposited on stainless steel and on a titanium electrodes via the sol-gel route using alkoxide precursor will be discussed with respect to piezo-enhanced nitrogen fixation. By applying piezo-actuation the ammonia production could be enhanced by more than four times in comparison without any piezo actuation.

S9: Porous Ceramics: Novel Developments and Applications

Innovative Processing of Porous Ceramics

Room: Coquina Salon H (North Tower)
Session Chairs: Paolo Colombo, University of Padova; Farid Akhtar, Lulea University of Technology

1:30 PM

(ICACC-S9-010-2023) Porous ceramics from capillary suspensions - unique proprties & applications (Invited)

N. Willenbacher*[1]

1. Karlsruhe Institute of Technology (KIT)IT, MVM, Germany

Capillary suspensions are ternary solid/liquid/liquid systems in which a strong particle network controlled by capillary forces is formed by the addition of small amounts of a second immiscible liquid to a binary suspension. Such systems are excellent as pre-cursor for the fabrication of strong ceramic materials with high open porosity (> 50%) and pore sizes in the range 0.5 to 50 μm. Specific structural features of corresponding sintered ceramics will be discussed. High shape accuracy, reduced shrinkage and crack formation compared to ceramics made from conventional slurries is achieved. Outstanding specific mechanical strength (2 MPa at 75% porosity) is enabled via deposition of nanoparticles at the particle contact regions. Their distinct flow behavior (high yield stress, pronounced shear thinning) makes capillary suspensions ideally suited for extrusion but also for direct ink writing (DIW), strut diameters D down to 70 μm and span ratios L/D = 15 (were L is the length of an undeformed, free-standing filament) are accessible. Ceramic slurries for strongly accelerated investment casting have been developed based on the capillary suspension concept. Highly porous discs with distinct pore size distribution for soil water potential sensing, ceramic filters with complex shape, and porous electro-ceramic structures with giant functional properties made from capillary suspensions will also be presented.

2:00 PM

(ICACC-S9-011-2023) 3D thermal energy storage materials based on highly porous patterned printed scaffolds (Invited)

M. Belmonte*[1]; I. Díaz-Herrezuelo[1]; L. Moreno-Sanabria[1]; M. I. Osendi[1]; P. Miranzo[1]

1. Institute of Ceramics and Glass, CSIC, Spain

The thermal energy is one of the most appealing energy sources to reduce the greenhouse gas emissions and the dependence on fossil energy resources. Thermal energy storage (TES) arises as an environmental friendly technology to mitigate those issues and, in this way, phase change materials (PCM) are considered the most efficient method of storing thermal energy. However, one of the main drawbacks of PCM is the liquid leakage, which considerably reduces the storage efficiency. To overcome this problem, a novel approach is presented, which is based on the development of three-dimensional (3D) engineered TES structures (3DTES) able to efficiently storage and release thermal energy to be potentially employed in concentrated solar power plants. 3DTES are formed by highly porous 3D printed low-cost clay supports that are infiltrated with a PCM based on molten salts. Clay supports are additive manufactured by robocasting and the cellular pattern of the skeleton is modified to promote distinct porosities and, hence, PCM infiltration degree. 3DTES are lightweight, mechanically robust and avoid the molten salt leakage. They present good enthalpy of fusion, excellent thermal stability, and high thermal energy storage efficiency and thermal conductivity, which is indicative of a great charging-discharging ability. 3DTES open new opportunities to manufacture affordable materials for energy applications

2:30 PM

(ICACC-S9-012-2023) Direct Ink Writing of porous geopolymers for thermochemical energy storage

C. Zoude*[1]; L. Gremillard[1]; É. Prud'homme[1]; K. Johannes[2]

1. INSA Lyon, Materials, Engineering and Science, France
2. INSA Lyon, Centre for Energy and Thermal Sciences of Lyon, France

Due to the depletion of fossil resources and to the rising ecological awareness, energy storage has become a major issue in our society. Thermochemical storage, based on hygroscopic salts, seems promising to meet these new challenges. However, in order to optimize the thermal properties of these systems, it is necessary to associate the salt with a host material. For our application, this host material must be affordable, resistant and as porous as possible. To meet these requirements, we have chosen to design porous geopolymers. A first level of porosity is controlled by the shaping technique: Direct Ink Writing. It is particularly relevant for our application since it allows to obtain the desired controlled porosity between filaments. A second finer level of porosity is obtained by printing a geopolymer foam. This foam is realised by direct foaming thanks to the addition of aluminium powder, which, in contact with the basic medium, releases dihydrogen, generating porosities in the paste. The impacts of the amount of powder, the foaming protocol, the printing process as well as the influence of different surfactants on the porosity rate and the connectivity of the pores are studied. For now, structures have been successfully printed and show good fidelity to the model at two different scales.

3:10 PM

(ICACC-S9-013-2023) Crack-Free Macroporous Structures by 3D Printing combined with Colloidal Assembly

B. Winhard[1]; L. Grassi Maragno[1]; A. Gómez Gómez[1]; J. Katz[1]; K. P. Furlan*[1]

1. Hamburg University of Technology, Institute of Advanced Ceramics, Integrated Materials Systems group, Germany

High-resolution 3D printing is often synonym to long printing times as well as costly equipment. We hereby demonstrate that the combination as an additive manufacturing process, direct writing, with colloidal assembly of low viscosity suspensions enables fast printing times while reaching high-resolution in terms of 3D structural features. We demonstrate that by using a "comb-strategy" for 3D printing of micron-sized particles' suspensions, uncontrolled contact line slipping is avoided and upon optimization of the printing parameters, printing of crack-free cm-sized homogeneous colloidal coatings is possible. Those are then transformed into ceramic macroporous structures with remarkable broadband reflection in the near infrared by using atomic layer deposition. Our work highlights the advantages of combining additive manufacturing with colloidal assembly (AMCA) for novel applications of colloidal-based printed structures.

3:30 PM

(ICACC-S9-014-2023) Lithography-based ceramic manufacturing of isotropic and functionally graded porous alumina ceramics

M. Schwentenwein*[1]; J. Schlacher[2]; R. Bermejo[3]; S. Nohut[1]

1. Lithoz GmbH, Austria
2. Montanuniversitaet Leoben, Austria
3. Montanuniversitaet Leoben, Institut fuer Struktur- und Funktionskeramik, Austria

Additive manufacturing (AM) allows the production of customized porous ceramic components with complex geometries for applications such as membranes, filters, thermal and acoustic insulation catalysis, or biostructures. In this study, porous alumina samples have been produced by using different concentrations of poly(methyl methacrylate) as pore-forming agent (PFA) in a photocurable slurry via lithography-based ceramic manufacturing (LCM). The mechanical properties and microstructural features were determined as a function of PFA concentration. Furthermore, the printed layer thickness, PFA particle size, and sintering temperature were varied to assess their influence on the resulting material properties. The results showed that the counterbalance effect between porosity and mechanical properties can be optimized by design of material composition and processing parameters according to the end application. Furthermore, multi-material LCM printing was evaluated in combination with the aforementioned PFA concept to create functionally graded ceramics with porosity gradients in different orientations.

3:50 PM

(ICACC-S9-015-2023) A hybrid robocasting process for ceramics with hierarchical porosity

L. Tabard[1]; V. Garnier[1]; É. Prud'homme[1]; S. Meille[1]; J. Adrien[1]; Y. Jorand[1]; E. Courtial[2]; K. Johannes[3]; L. Gremillard*[1]

1. INSA, Materials, Engineering and Science, France
2. 3D.FAB, France
3. CETHIL, INSA, France

We present a hybrid additive manufacturing process combining direct-ink writing (robocasting) and starch-consolidation method, applied to the fabrication of highly porous Ce-stabilized zirconia scaffolds. This process enables a precise control of the volume and size of the porosity. It hierarchization is controlled by three independent parameters: i – the numerical design of the pieces controls the macroscopic porosity (from 100 μm to 2 mm); ii – addition of starches as pore formers enables to control a micronic porosity (from 2 μm to 70 μm); iii – a partial sintering steps enables to preserve residual, sub-micronic porosity with a characteristic size of 200 nm. A hierarchization over 4 decades of length scales and up to three families of pores is thus obtained. The porosity fraction as high as 85% and the precise control of its size distribution are above those usually obtained in ceramics scaffolds processed with robocasting. The obtained compressive strengths depend both on the porous fraction and pores shape. Moreover, they are closely related to those of individual filaments produced by the same process. Thus, additive manufacturing combined with more conventional porous ceramics fabrication techniques show a big step forward for both increasing and tailoring the porosity in order to produce complex porous structures. An application of such material for thermochemical energy storage will be shown.

4:10 PM

(ICACC-S9-016-2023) Direct Extrusion-based 3D-Printing of Lunar Soil Simulant

D. T. Desai*[1]; A. Kumar[1]; K. Viswanathan[1]

1. Indian Institute of Science, Mechanical Engineering, India

We describe here a direct extrusion 3D printer for printing structures from extra-terrestrial soil simulants using a piston-based extruder. Tool paths are generated by defining a continuous extrusion nodal path with a uniform flow rate using customized user-generated G-code routines. Printing of simple geometries like beam, cylinder, and 2D-uniform diamond and hexagonal frame structures is demonstrated using a slurry composed of lunar soil simulant (LSS) variant LSS-ISAC-1 (avg. particle size ~ 90μm) mixed with naturally occurring polymeric adhesive (guar gum) and DI water as solvent. Parts were printed using a 2 mm diameter nozzle by optimizing print speed, nozzle height, inter-layer drying time, and build temperature to ensure shape retention post-printing. The final green parts were dried in a hot air oven (50°C) for 48hrs, followed by sandpaper polishing. The strengths of the samples were evaluated using compression and flexure tests and were found to be comparable to that of bio-consolidated structures. These results show the potential for using extrusion-based 3D printing in remote extra-terrestrial environments to obtain load-bearing structures.

S10: Modeling and Design of Ceramics and Composites

Modeling of Ceramics and Composites III

Room: Coquina Foyer (North Tower)
Session Chair: Sota Terasaka, Japan Fine Ceramics Center

1:30 PM

(ICACC-S10-011-2023) Four parameter scalar continuum damage models for ceramic matrix composites with significant in plane ply anisotropy (Invited)

C. P. Przybyla*[1]; A. Debarre[2]; J. Maire[2]; E. Baranger[3]; F. Laurin[2]

1. Air Force Research Laboratory, Materials and Manufacturing Directorate, USA
2. Office National d'Etudes et de Recherches Aérospatiales (ONERA), France
3. ENS Paris-Saclay, France

Current models for non-linear response prediction of ceramic matrix composites (CMCs) to support the design, certification and sustainment of components are limited. Here a formulation for a two-dimensional (2D) continuum damage model derived via a thermodynamics-based approach based called openDM is introduced. The model is formulated with four scalar damage parameters that account for damage as a result of matrix cracking in the fiber and transverse direction as well as matrix damage due to shear in the ±45° directions. The applicability of these models are considered for a balanced 2D woven based CMC and a 2D unidirectional ply based CMC with significant anisotropy between the fiber and transverse directions. The process of model calibration, verification and validation is discussed for both materials that were considered. Implemented was done using a nonlinear classical laminate theory and the code is generally applicable for the analysis of any arbitrary stacking sequence.

2:00 PM

(ICACC-S10-012-2023) Examination of Discontinuous Changes in Grain Boundary Velocity Induced by Grain Boundary Transformations

C. Marvel*[1]; B. Zalatan[2]; H. Zhou[2]; C. Riedel[3]; B. Chen[2]; M. Harmer[3]

1. Louisiana State University, Mechanical and Industrial Engineering, USA
2. Lehigh University, Computer Science and Engineering, USA
3. Lehigh University, Materials Science and Engineering, USA

A growing literature based on theory, simulation and experiment has inferred that grain boundaries undergo first-order transitions, resulting in discontinuous changes of grain boundary properties, particularly mobility. However, upon instant changes in mobility, it is unclear if there is a proportional increase in grain boundary velocity, which more directly drives microstructure evolution. Here, 3D Monte Carlo Potts grain growth models were completed and grain boundary velocities were tracked in the presence of stochastic grain boundary transitions that increased grain boundary mobility 100-fold. Grain boundary curvature was measured using the innie/outie methodology and grain boundary velocity was quantified by tracking net voxel swapping. Discontinuous changes in grain boundary velocity were assessed by comparing grain boundary curvature, mobility, and velocity time series. It was observed that, while grain boundary mobility underwent a 100-fold increase, the grain boundary velocity, on average, only increased by a factor of 80, thus suggesting that additional mechanisms (e.g. triple junction drag) were also emergent in the simulations. Interestingly, 85% of grain boundaries were inversely correlated to curvature, leaving 15% of grain boundaries anomalously migrating against curvature. More analysis and interpretation of the computational results will be discussed.

2:20 PM

(ICACC-S10-013-2023) Simplified analysis of delamination propagation at the free edge of cfrp composite fan blade

S. Kajihara*[1]; R. Higuchi[1]; T. Yokozeki[1]; T. Aoki[1]

1. University of Tokyo, Japan

This study investigates the energy release rate of the tip region of a composite fan blade during vibration due to bird strike impact. For simplicity, the energy release rate is obtained considering a beam model, where only the tip region of the blade is modeled. First, a dynamic finite element analysis is performed with impulse loading applied to the beam model with both edges free. The dynamic energy release rate is affected by the displacement distribution due to inertial forces, and it is confirmed to be equal to the static energy release rate obtained from the static analysis and Virtual Crack Closure Technique (VCCT) with the displacement distribution specified from time to time. Then, the proposed static analysis is performed using the out-of-plane displacement history at the tip obtained from bird strike simulations. The curvature distribution near the initial crack has a significant effect on the energy release rate, suggesting that delamination tends to propagate from the trailing edge, where the curvature value is larger.

2:40 PM

(ICACC-S10-014-2023) Effect of porosity on ablation resistance of C/C composites

M. Fradin*[1]; G. L. Vignoles[2]; G. Couégnat[2]; F. Rebillat[3]; K. Haras[4]; C. Grégis[4]

1. ArianeGroup SAS / LCTS, France
2. LCTS - CNRS, France
3. University Bordeaux, Laboratory of thermostuctural composites, France
4. ArianeGroup SAS, France

C/C composites used for rocket nozzles and divergents are often processed by the gas-phase route, which has the advantage of yielding an excellent carbon matrix but the drawback of leaving the material with an appreciable amount of residual porosity. This structural feature has an important impact on the recession rate of the material when exposed to ablation. In this work, a joint experimental and modeling analysis has been carried out in order to improve the understanding on this effect. Oxidation experiments associated to post-test X-ray CMT analyzes allowed characterizing not only the recession rate but also the depth of the affected zones in the material. The model then helps interpreting the results and also can be used as a design tool. A sensitivity study produced information on the most crucial parameters impacting the ablation behavior.

Modeling of Ceramics and Composites IV

Room: Coquina Foyer (North Tower)
Session Chairs: Jingyang Wang, Shenyang National Laboratory for Materials Science, Institute of Metal Research, Chinese Academy of Sciences; Matthew Guziewski, US Army Research Laboratory

3:30 PM

(ICACC-S10-015-2023) Simulation study on microstructural changes of ceramic powder by sintering (Invited)

S. Terasaka*[1]; H. Nomura[1]; T. Kimura[1]; H. Matsubara[2]

1. Japan Fine Ceramics Center, Japan
2. Tohoku University, Graduate School of Environmental Studies, Japan

Ceramic products are developed and manufactured by a sintering process. The process is optimized through repeated trial and error in the field, which requires time and cost for development. The introduction of simulation is important to achieve high efficiency in the optimization of process conditions. In this study, solid phase sintering and grain growth of ceramics were investigated by simulation and experiment. The microstructural changes by sintering of alumina and barium titanate, which are representative ceramic materials, were analyzed by simulation using the Monte Carlo method and compared with experiments. The simulations reproduced the experimental sintering and grain growth behavior by setting appropriate simulation parameters corresponding to alumina and barium titanate respectively. The simulations indicate that the microstructure changes through the interaction of sintering and grain growth can be calculated.

4:00 PM

(ICACC-S10-016-2023) Hierarchical Design Optimization of Ceramic Composite Laminates (Invited)

M. C. Guziewski*[1]; K. D. Behler[2]; N. Ku[3]; T. W. Moore[4]; J. LaSalvia[5]; J. Pelz[6]; T. Shoulders[7]; J. Swab[1]

1. US Army Research Laboratory, USA
2. DEVCOM-Army Research Lab, Ceramics and Transparent Materials Branch, USA
3. DEVCOM - Army Research Laboratory, Ceramics and Transparent Materials Branch, USA
4. DEVCOM-Army Research Lab, SURVICE Engineering, USA
5. DEVCOM Army Research Laboratory, USA
6. US Army Research Laboratory, DEVCOM, USA
7. CCDC Army Research Laboratory, USA

Heterogeneous structuring of ceramic composites has the potential to achieve properties and performances superior to their constituent materials, however they also come with a unique set of challenges. Among these are residual stresses that arise during processing due to the mismatch in thermal expansion and elastic properties of the constituent materials. While this residual stress is among the primary reasons for the potential for better properties and improved performance, it can also reduce them or even cause failure during processing if not properly controlled. Thus, in an effort to control residual stress and improve the strength of ceramic composite laminates, a computational framework has been developed that implements classic micromechanics and plate theory to predict the stress state as a function of volume loading and inclusion size within a layer, laminate design, and applied stress. Due to the analytic nature of these formulations, it is then possible to determine the optimal design of the laminate for such properties as fracture toughness and flexural strength at minimal computational costs through the use of a genetic algorithm. This approach is applied to silicon carbide-boron carbide composites, where it is compared to both residual stress characterization and mechanical testing, and shown to consistently predict performance.

4:30 PM

(ICACC-S10-017-2023) Prediction of filled-hole tensile strength of CFRP laminates with various ply thicknesses (Invited)

R. Aoki*[1]; R. Higuchi[1]; T. Yokozeki[1]; K. Aoki[3]; S. Uchiyama[3]; T. Ogasawara[2]

1. The University of Tokyo, Department of Aeronautics and Astronautics, Japan
2. Tokyo University of Agriculture and Technology, Japan
3. SUBARU CORPORATION, Japan

In this study, damage simulation on filled hole tensile (FHT) tests was conducted on CFRP laminates with several ply thicknesses to predict the FHT strengths. In the damage analysis, the continuum damage mechanics model and the cohesive zone model were combined with finite element analysis for modeling the damage modes in composite laminates. The continuum damage mechanics model takes into account the effect of ply thickness on damage progress. The fastener restraint in the filled hole tensile test was modeled in finite element analysis to simulate the Filled Hole Tension test. The prediction results of FHT strengths of composite laminates with several ply thicknesses were compared and the effects of ply thickness and laminate layups on the FHT strengths were discussed. In addition, the open hole tensile strength of CFRP laminates with several ply thicknesses was predicted and compared with the FHT strength. The effects of fastener restraint properties and tightening torque on FHT strength in the FHT test were also numerically investigated.

5:00 PM

(ICACC-S10-018-2023) The Anomalous Hardness in the Transition Metal Carbides and Nitrides

C. R. Weinberger*[1]; H. R. Brumblay[2]; G. Thompson[3]

1. Colorado State University, Department of Mechanical Engineering, USA
2. Colorado State University, School of Advanced Materials Discovery, USA
3. Thompson, Metallurgical & Materials Engineering, USA

Transition metal carbides and nitrides are well known UHTCs that exhibit both extreme melting temperatures and high hardness. The mechanical properties of these materials are dependent on the carbon/nitrogen content as well as the transition metal. The hardness of the group IVB carbides decrease monotonically with loss of carbon while some of the group VB carbides show a peak in hardness around the $MeC_{0.88}$ composition, a phenomenon known as the anomalous hardness. This peak in hardness and its variation with carbon content has be argued to exist due to bonding effects, dislocation pinning effects due to carbon, and microstructural effects such as domain size, amongst many others. However, to date there is no scientifically accepted cause of this behavior. In this talk, we revisit the evidence and hypotheses of the anomalous hardness and provide new insight to this debate by examining the role of microstructure and bonding in controlling hardness. We use density functional theory calculations to examine the change in elastic constants and demonstrate that this cannot rationalize the change in hardness. However, calculations of the resistance to slip, via generalized stacking fault energy curves, provide new surprising insight into the origins of hardness and slip in these materials which have the potential explain the origins of the anomalous hardness.

5:20 PM

(ICACC-S10-019-2023) Surfing boundary conditions to characterize effective fracture-toughness of ceramic composites

Z. Hossain*[1]

1. University of Delaware, USA

Despite century-long successes in the field of fracture mechanics, the understanding of toughness-structure correlation remains elusive, regardless of the scale of the structure. For over a century now it has been possible to describe effective properties (such as effective stiffness) that are characterized by variational principles. Nonetheless, it remains a challenge to establish a notion of effective toughness that is governed by the criticality of the material. To design new ceramic-based armor materials, it is essential to develop robust characterization tools that can describe effective fracture toughness as a function of the nano-microstructural attributes of the composite. Under the conventional uniform remote loading at the macroscopic boundary, initial cracks grow in an unstable manner due to wild spatial variations in the singularity of the elastic fields across the nano-microstructure. In this talk, we will present a new boundary condition called the 'surfing boundary condition' that enables the determination of effective fracture toughness as a function of the nano-microstructures at multiple scales. It also makes it possible to explore the evolution of crack-path in complex heterogeneous media. The approach reveals the fundamental design insights for devising brittle composites possessing controllable anisotropic and asymmetric fracture toughness.

5:40 PM

(ICACC-S10-020-2023) Numerical and Experimental Ballistic Performance Investigation of Fiber Reinforced Polymer Composites

A. M. Punongbayan*[1]; R. A. Sirot[1]; E. d. Magdaluyo[1]

1. University of the Philippines Diliman, Department of Mining, Metallurgical, and Materials Engineering, Philippines

Natural fibers such as pineapple leaf fibers are now being utilized in fabricating composite products for ballistic applications because of their mechanical properties as well as their abundance and sustainability in terms of disposal. The assessment of ballistic performance of fiber-reinforced epoxy matrix composite can be done through numerical and experimental methods. In this study, numerical simulation via Ansys was first done to determine the ideal number of layers for each component in different layer combinations to minimize cost since actual testing is expensive. To validate the numerical simulation, three composite plate samples of varying amount of fabric layers: Sample A – 3 carbon: 1 aramid, Sample B – 2 carbon: 2 aramid and Sample C – 2 carbon: 1 aramid: 1 pineapple were fabricated via vacuum-assisted resin infusion. The actual ballistic penetration test was then performed on a shooting range where the simulated and actual results matched. All samples were penetrated by 5.56 M193 caliber (National Institute of Justice, NIJ Level III). Meanwhile, the 357 Magnum (NIJ Level IIIA) and 9 MM (NIJ Level II) calibers were not able to penetrate the composite plates. Moreover, the results of the ballistic tests for the fiber reinforced polymer composites containing natural fiber (Sample C) is comparable to the samples with purely synthetic fibers (Sample A and Sample B).

6:00 PM

(ICACC-S10-021-2023) First principles investigation of surface stability and water adsorption behavior of Lu_2SiO_5

M. Liu*[1]; J. Wang[2]; J. Wang[3]

1. Institute of Metal Research, Chinese Academy and Sciences, Advanced Ceramics and Composites Division, China
2. Institute of Metal Research, High-performance Ceramic Division, China
3. Shenyang National Laboratory for Materials Science, Institute of Metal Research, Chinese Academy of Sciences, High-performance Ceramics Division, China

Rare earth (RE) monosilicates are used as the top coat of environmental barrier coating (EBC) systems due to their excellent environmental durability. The interaction between water vapor and RE monosilicates plays a critical role in the application of EBC. However, the reaction mechanism between water and RE monosilicates is complicated and remains poorly understood at the atomic level. In this work, in order to explore the interaction between surface of RE monosilicate and water molecular, the surface stability and water adsorption behavior of Lu_2SiO_5 were investigated by first principles calculations. Based on the structure features of Lu_2SiO_5, 4 kinds of low index surfaces, including 13 kinds of terminations, were considered. The surface stability was determined as (1 0 -1) > (001) > (110) > (100) from the surface energies. Then the adsorption sites and energies of water molecule on (1 0 -1) and (100) surface were predicted. Two kinds of adsorption mechanisms were found. Water molecule remains intact after adsorbed on the (1 0 -1) surface with a relatively low adsorption energy. Whereas the absorbed water molecule will dissociate into a H atom and a -OH group on the (100) surface with higher adsorption energy. Electronic structure analysis shows that the chemical environments of under-coordinated O atoms on surface play key role on the water adsorption mechanism.

S12: On the Design of Nanolaminated Ternary Transition Metal Carbides/ Nitrides (MAX Phases) and Borides (MAB Phases), Solid Solutions Thereof, and 2D Counterparts (MXenes, MBenes)

Design of Novel Compositions and Manufacturing Methods IV

Room: Ballroom 3 (South Tower)
Session Chairs: Surojit Gupta, University of North Dakota; Christopher Shuck, Drexel University; Deniz Cakir, University of North Dakota

1:30 PM

(ICACC-S12-015-2023) Scalable synthesis and purification of high-yield monolayer MXene dispersions in different media (Invited)

N. Goossens*[2]; K. Lambrinou[1]; J. Vleugels[2]

1. University of Huddersfield, School of Computing and Engineering, United Kingdom
2. KULeuven, Materials Engineering, Belgium

MXenes are electrically conductive nanometric 2D sheet-like transition metal carbides, resulting from the chemical exfoliation of 3D nanolaminated $M_{n+1}AX_n$ (MAX) phases. The precursor MAX phases are described by the $M_{n+1}AX_n$ general stoichiometry, where M is an early transition metal, A is an A-group element, X is C or N, and n = 1-3. Chemical exfoliation is achieved by the selective etching of the A-layers in the MAX phases, typically consisting of aluminum (Al). Their surface is terminated with functional groups (e.g., -F/-Cl/-OH) and becomes tunable by adapting the synthesis method, allowing MXenes to be tailored towards the envisaged application. MXenes are sparking an extreme interest as active functional 2D materials in electrochemistry, energy storage, catalysis, as well as for diverse thermoelectric and biomaterial applications. This study addresses the scalable synthesis of high-purity monolayer MXenes using a modified LiF/HCl etching route, characterizes their surface and morphology, and quantifies the amount of undesirable residual species. Moreover, this work identifies the relationship between the adopted synthesis route and the resulting impurities in the MXene products, also proposing a new methodology for purification and high-throughput MXene extraction that aims at improving the stability, quality, and yield of MXene dispersions in various aqueous and organic media.

2:00 PM

(ICACC-S12-016-2023) Synthesis, Characterization, Properties, First Principles Calculations, and X-ray Photoelectron Spectroscopy of Bulk Mn_5SiB_2 and Fe_5SiB_2

T. A. Elmeligy*[1]; S. Kota[3]; V. Natu[1]; H. Lind[2]; J. Palisaitis[2]; P. Persson[2]; J. Rosen[4]; M. Barsoum[5]

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2. Department of Physics, Chemistry and Biology (IFM), Sweden
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5. Drexel University, Materials Science and Engineering, USA

We synthesize fully dense, bulk, predominantly single-phase, polycrystalline samples of layered ternary transition metal borides Mn_5SiB_2 and Fe_5SiB_2 by reactively hot-pressing of Mn, Fe, FeB, Si, and B powders. The atomic structures resolved by high-resolution scanning transmission electron microscopy revealed high-crystal quality. Elongated striped defects, confined below the nanometer in width, were observed. The selected area electron diffraction further accentuates the high-crystal quality by discrete pattern spots, consistent with a tetragonal crystal structure along the [001] zone axis. With Vickers hardness values of 12.1±0.4 GPa, and 12.7±0.1 GPa, for Mn_5SiB_2 and Fe_5SiB_2 respectively, these borides are relatively soft. The room temperature electrical resistivities at 1.5±0.1 and 1.2±0.1 μΩ.m, for Mn_5SiB_2 and Fe_5SiB_2, respectively. The binding energies of the Mn, Fe and Si measured by X-ray photoelectron spectroscopy bolster the idea that the bonds are metallic. Density functional theory (DFT) calculations confirm that the ground states of both compounds are ferromagnetic as observed experimentally. DFT predictions show that the density of states at the Fermi level are dominated by the d-orbitals of the transition metals. Neither material was readily machinable with conventional tooling, but with electro-discharge machining (EDM).

2:20 PM

(ICACC-S12-018-2023) Synthesis of New Carbonitride MXenes and Their Electrochemical Behavior

A. Tabassum*[1]; K. Liang[1]; C. E. Shuck[2]; K. Prenger[1]; A. Majed[1]; C. Dun[4]; J. Urban[4]; Y. Gogotsi[3]; M. Naguib[1]

1. Tulane University, Physics and Engineering Physics, USA
2. Drexel University, Materials Science and Engineering, USA
3. Drexel University, USA
4. The Molecular Foundry, LawrenceBerkeley National Laboratory, USA

MXene family has been growing rapidly after the discovery of first MXene, $Ti_3C_2T_x$, in 2011 among which most of them are carbides with very few nitrides and carbonitrides. Even though Ti_3CNT_x exhibited unique properties and behavior compared to its carbide counterpart in applications including energy storage, electromagnetic shielding, optics, sensing etc. it had been the only carbonitride MXene for more than 10 years until recently we reported on $Ti_2C_{0.5}N_{0.5}T_x$. This is, in part, because of the challenging synthesis due to lower stability of nitride MXenes compared to carbides. Herein, we report on the synthesis and applications of titanium carbonitride MXenes series from three different titanium carbonitride MAX phases- 211, 312 & 413. By tuning the synthesis conditions using potassium fluoride and hydrochloric acid, we realized $Ti_2C_{0.5}N_{0.5}T_x$ MXene from $Ti_2AlC_{0.5}N_{0.5}$. When tested as electrode material for sodium ion battery, $Ti_2C_{0.5}N_{0.5}T_x$ exhibited the highest specific capacity among all reported multilayer MXene electrodes. We will discuss the performance of Ti_3CNT_x as electrocatalyst for hydrogen evolution reaction with an onset potential of 56 mV which is significantly lower than that of $Ti_3C_2T_x$. Finally, we will present our new findings on the synthesis of $Ti_4C_{(3-y)}N_yT_x$ (y = 2.2, 2.4, 2.6 and 2.8) MXenes along with their potential application in electrochemical energy storage.

S14: Crystalline Materials for Electrical, Optical and Medical Applications

Scintillator

Room: Flagler A (South Tower)
Session Chair: Kenji Toda, Niigata University

1:30 PM

(ICACC-S14-006-2023) Fabrication and characteristics of a rare-earth ion doped calcium fluoride transparent ceramics for dosimetric applications (Invited)

N. Kawano*[1]; D. Nakauchi[2]; G. Okada[3]; N. Kawaguchi[2]; T. Yanagida[2]

1. Akita University, Japan
2. Nara Institute of Science and Technology, Japan
3. Kanazawa Institute of Technology, Japan

Dosimeters using phosphors have gained considerable attention with an aim to estimate radiation dose. For the dosimeters, a calcium fluoride with a rare-earth ion is a famous material owing to its low effective atomic number and high sensitivity. In this study, calcium fluoride transparent ceramics with an impurity (Dy^{3+} or Tm^{3+}) were fabricated by spark plasma sintering, and their scintillation and dosimeter properties were evaluated for radiation dosimetry. A Tm-doped calcium fluoride transparent ceramic showed emission peaks originating from self-trapped excitons and the 4f-4f transition of Tm^{3+} under X-ray. Further, the ceramic showed thermally-stimulated luminescence (TSL), and linear dose response was confirmed from the 0.01 mGy to 1000 mGy. A Dy-doped calcium fluoride transparent ceramic exhibited scintillation peaks originating from self-trapped excitons and the 4f-4f transition of Dy^{3+}. Further, the ceramic showed TSL with the lowest detectable dose of 0.01 mGy. Moreover, the ceramic was found to show optically-stimulated luminescence originating from the 4f-4f transition of Dy^{3+} when excited by 590 nm light.

2:00 PM

(ICACC-S14-007-2023) Transparent Ceramic Scintillators (Invited)

N. Cherepy*[1]; R. Osborne[1]; J. Smith[1]; S. O'Neal[1]; D. Schneberk[1]; Z. M. Seeley[1]; C. McNamee[1]; S. A. Payne[1]

1. Lawrence Livermore National Laboratory, USA

Transparent ceramic oxides offer excellent ruggedness and environmental stability for long service life even when deployed in harsh environments or high irradiation conditions. Spectroscopic Radiation Portal Monitors, Radioisotope Identification Detectors and X-ray Imaging Screens need scintillator detector materials that are not fragile and do not degrade in humid air or typical ambient temperatures. The Cerium-doped Gadolinium Yttrium Gallium Aluminum Garnet, or GYGAG(Ce), polycrystalline transparent ceramic scintillator offers a density of 5.9 g/cm^3 and Z_{eff} of 48, along with a light yield of 50,000 Photons/MeV and a principal decay time of 120 ns, making it a promising candidate for the next generation of gamma spectrometers. PMT or SiPM readout of GYGAG(Ce) ceramics in sizes of 2 in^3 can achieve energy resolution of R(662 keV) 5-6%. The transparent ceramic scintillator Europium-doped Gadolinium Lutetium Bixbyite, or GLO(Eu), offers 9.1 g/cm^3, Z_{eff} of 68, a light yield of 55,000 Photons/MeV and a principal decay time of 1 ms. It can be formed into large transparent sheets needed for a variety of MeV X-ray imaging applications.

2:30 PM

(ICACC-S14-008-2023) Additive manufactured high-aspect-ratio pixelated transparent ceramic scintillator for x-ray imaging (Invited)

J. Smith*[1]; R. Osborne[1]; T. Yee[1]; B. Moran[1]; Z. M. Seeley[1]; N. Cherepy[1]; S. A. Payne[1]

1. Lawrence Livermore National Laboratory, USA

Current x-ray imaging capabilities are dominated by thin phosphors on standard TFT arrays. The GLO (Europium-doped Gadolinium Lutetium Oxide) transparent ceramic scintillator has been shown to improve high energy x-ray imaging capabilities. A pixelated GLO array with a high aspect ratio would increase the interaction length while maintaining spatial resolution, and therefore further improve diagnostic capabilities. Additive manufacturing approaches including direct ink write, and stereolithography combined with traditional ceramic consolidation approaches are being studied to produce these pixelated arrays. Pixelated GLO arrays with an aspect ratio near 10:1 have been fabricated via direct ink write printing and studies are underway for direct SLA printing of pixelated ceramic arrays.

Optical Material

Room: Flagler A (South Tower)
Session Chair: Nerine Cherepy, Lawrence Livermore National Lab

3:20 PM

(ICACC-S14-009-2023) Development of transparent SrF_2 ceramics for laser gain applications

T. Rudzik*[1]; Z. M. Seeley[3]; N. Cherepy[2]; S. A. Payne[1]

1. Lawrence Livermore National Laboratory, USA
2. Lawrence Livermore National Lab, Chemistry and Materials Science, USA
3. Lawrence Livermore National Lab, Chemical Sciences Division, USA

The performance of high-power lasers is limited by the properties of the current glass and single crystal laser gain media in terms of the maximum pulse power, the damage threshold, and the required time between shots resulting from heat dissipation limitations. SrF_2 ceramic has been identified as a potential host material which could improve all these factors and therefore enable more energetic lasers and more compact architectures. Of primary importance to the success of SrF_2 ceramic in high-energy laser applications is optical scatter minimization, which has proven especially difficult in halides due to their strong affinity for oxide/hydroxide defect species. Although methods have been found to produce single crystal halides without such defects, multiple factors complicate polycrystalline ceramics fabrication. This work reports the current progress in fabrication of SrF_2 ceramics with sufficient optical quality for the intended laser applications, including powder feedstock studies, powder purification tests, and sintering investigations using both pressureless and pressure-assisted sintering as well as isostatic pressing. We have made vast improvements over initial tests and have achieved optical scatter of ~2%/cm. Problematic trace impurities present in many feedstocks were identified and fabrication optimization for low scatter is ongoing.

3:40 PM

(ICACC-S14-010-2023) Contamination and Laser Damage Morphology on Fused Silica Polished by Magnetic Field-Assisted Finishing

J. T. Long*[1]; Y. Tsunezuka[3]; A. Uemura[3]; D. Funayama[3]; T. Kamimura[3]; R. Yasukuni[3]; H. Yamaguchi[2]

1. University of Florida, Materials Science and Engineering, USA
2. University of Florida, Mechanical and Aerospace Engineering, USA
3. Osaka Institute of Technology, Japan

Surface contamination and surface/subsurface damage have a well-documented negative effect on the laser-induced-damage threshold (LIDT) of transparent optics. The presence of contamination and damage layers on the surface of polished fused silica contributes to a large reduction in LIDT at ultraviolet wavelengths. Magnetic field-assisted finishing (MAF) with iron particle tools has previously been applied to fused silica and shown to improve LIDT by up to 45%. However, the possibility of iron contamination due to the iron particle tool has not been explored. This presentation will examine the laser damage morphology on the surface of fused silica samples finished with either iron particles or commercial polishing pads. The diameter of the damaged area was used to compare the extent of contamination on the surface of each sample. The results show no significant difference in the diameter of damaged areas between surfaces polished with different tool types. This indicates that both the iron particle and commercial pad tools are removing existing contamination with similar effectiveness.

4:00 PM

(ICACC-S14-011-2023) Fabrication of Laser Transparent Ceramics via Additive Manufacturing

R. Osborne*[1]; T. J. Wineger[1]; T. Yee[1]; Z. M. Seeley[1]; I. R. Phillips[1]; T. Rudzik[1]; J. Smith[1]; N. Cherepy[1]; R. M. Gaume[2]; M. Dubinskiy[3]; S. A. Payne[1]

1. Lawrence Livermore National Laboratory, USA
2. University of Central Florida, CREOL, USA
3. US Army Research Laboratory, USA

Transparent ceramic laser media offer many advantages over single crystals, particularly the ability to create spatially controlled laser ion doping profiles to enhance thermal management and beam quality. Additive manufacturing techniques may be used to produce ceramic green bodies that are then consolidated via sintering and hot isostatic pressing into laser materials with optimized doping profiles. The direct ink write (DIW) technique involves extruding a shear thinning viscous "ink" of ceramic nanoparticles through a nozzle onto a substrate while moving the nozzle to achieve the desired shape. Two or more inks of different compositions can be used to create specific doping profiles. Resolution in the hundreds of microns can be achieved with this method, enabling the production of laser rods with endcaps, core-clad, or gradient profiles. Material jetting, in which nanoparticle suspensions are deposited onto a powder bed in small droplets, can produce thin, planar doping profiles with resolution in the tens of microns. These techniques open many possibilities in the design of advanced laser materials for improved performance, as demonstrated with various dopant ions and profiles in YAG.

4:20 PM

(ICACC-S14-012-2023) Optical properties and laser performance of Nd:Y_2O_3 ceramics with fine-grained microstructure

H. Kim*[1]; H. Oh[1]; Y. Park[1]; J. Ko[1]; J. Lee[1]; H. Ma[1]

1. Korea Institute of Materials Science, Republic of Korea

Transparent neodymium doped Y_2O_3 ceramics is adopted for use in high power solid-state lasers due to their superior thermal characteristics compared to YAG ceramics. To fabricate highly transparent Y_2O_3 ceramics, the microstructural evolution was investigated in this study via hot-pressing followed by a HIP treatment while controlling the composition of the sintering additive and hot-pressing temperature. Without a sintering additive, the grain growth was vigorous as hot-pressing temperature increased and led to coarsening-dominated sintering trajectories with various residual pore sizes. Densification increased with the composition of the Zr and La co-doped additive, leading to a fine-grained microstructure (~0.94 μm) after the HIP treatment. Thus, the prepared Nd:Y_2O_3 ceramics presented optical and laser properties comparable to those of conventional Nd:Y_2O_3 ceramics.

4:40 PM

(ICACC-S14-013-2023) Fabrication of Rugged Transparent Ceramics

J. Gild*[1]; A. Floyd[1]; B. Sadowski[2]; T. Zhou[3]; W. Kim[1]; S. Bayya[1]; J. Sanghera[1]

1. Naval Research Laboratory, Optical Science Division, USA
2. Jacobs, USA
3. University Research Foundation, USA

Recent progress on the procurement, purification, and sintering of a variety of high temperature transparent ceramics is presented. There are numerous applications operating in very harsh environments that require rugged windows. Oxide materials such as Y_2O_3 and non-oxides such as cubic β-SiC are prime candidates for these applications due to high mechanical strength, good transmission range, and isotropic structure. To fabricate a high transparency window, phase and chemically pure powder precursors must be obtained. Higher purity β-SiC powders are becoming commercially available and multiple sources are analyzed, purified, and sintered to optimize transmission. Both oxide and non-oxide materials are sintered via spark plasma sintering (SPS) and the optical and physical properties are discussed.

5:00 PM

(ICACC-S14-014-2023) Relationship Between Grain Size and Grain Boundary Energy of Yttria Nanoceramics

N. M. O'Shea*[1]; J. Mason[1]; R. Castro[1]

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

Controlling the microstructure of highly-dense yttria (Y2O3) ceramics is key to their utilization in applications like laser optics since cubic yttria is transparent at full density. Sintering to high densities is more readily achieved when starting with nanoparticles, though the reduced grain size increases the volume fraction of grain boundaries in the material. Since grain boundary energies and their effects on the material's properties are still not well understood, the increased specific grain boundary area may result in properties that differ from those of conventional materials. In this study, yttria nanoparticles of various grain sizes were synthesized by reverse-strike coprecipitation and sintered to full or nearly full density via high-pressure spark plasma sintering (HP-SPS). X-ray diffraction, electron microscopy, and nanoindentation were employed to characterize microstructures. Differential scanning calorimetry was used to investigate grain boundary energy in relation to grain size. Additionally, molecular dynamics simulations were performed using LAMMPS to explore the behavior of dopants in the material, especially the segregation of dopants to grain boundaries, and their impact on the overall energy of the system. The results of these simulations will be used to select candidates for improving the properties of the sintered yttria nanoparticles through doping.

S16: Geopolymers, Inorganic Polymers and Sustainable Construction Materials

Porous Geopolymers II

Room: Coquina Salon C (North Tower)
Session Chair: Enrico Bernardo, University of Padova

1:30 PM

(ICACC-S16-036-2023) Porous geopolymer composite filter media for the removal arsenic from ground water (Invited)

K. Qureshi*[1]

1. Mehran University of engineering and Technology Jamshoro, Chemical Engineering, Pakistan

Water is one of the essential elements of life on earth, the number of physical, chemical, and biological processes including filtration, reverse osmosis, degasification, sedimentation, flocculation, precipitation and adsorption. Adsorption is an important process the most commonly employed due to high efficiency, simple procedure and cost effectiveness. In this study the porous geopolymer was developed using GP+airation method and fixed in packed bed column by to investigate arsenic removal efficiency. Porosity was determined by pycnometer, X-ray Powder diffraction (XRD) were performed by D8-Bruker and SEM were analyzed by JEOL 6060LV. the column parameter, time, initial concentration of water, inlet water flow has been optimized.

2:00 PM

(ICACC-S16-037-2023) Development of porous geopolmer by using different methods for the removal arsenic from synthetic waste water (Invited)

A. Qadeer*[1]

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

Water is one of the basic needs of human survival and development, the world water resources are contaminated due to natural and anthropogenic activities. The number of physical, chemical, and biological processes are employed for pollutants removal from water and wastewater. Adsorption is the most widely utilized

process due to high efficiency, simple procedure and cost effectiveness. In this study the porous geopoloymer was produced by using different method (i) GP+aeration (ii)) GP+Canola oil (iii) GP+dimethyldiethoxysilane (iv) GP+Graphene Oxide to investigate removal efficiency. Porosity was determined by pycnometer, X-ray Powder diffraction (XRD) were performed by D8-Bruker and SEM were analyzed by JEOL 6060LV. The arsenic removal capabilities of various adsorbent filter media were carried out using packed bed column laboratory scale continuous flow experiments.

2:30 PM

(ICACC-S16-038-2023) Preparation of alkali-activated foams by using peracetic acid as a novel blowing agent (Invited)

M. A. Bhuyan*[1]; C. Kurtulus[2]; A. Heponiemi[3]; T. Luukkonen[1]

1. University of Oulu, Fibre and Particle Engineering Research Unit, Finland
2. Afyon Kocatepe University, Department of Materials Science and Engineering, Turkey
3. University of Oulu, Research Unit of Sustainable Chemistry, Finland

Alkali-activated foams (AAFs) are commonly prepared by using the direct foaming method, in which blowing agents such as H_2O_2, metallic Al, elemental Si, sodium perborate, or sodium hypochlorite are used to generate porosity. At high pH, these blowing agents are decomposed and produce gas, which is trapped inside the material to generate a pore structure. As a result, typically a large volume increase is observed, which is a practical problem since cutting the expanded parts of the foams is often required for their applications. To overcome this problem, peracetic acid (PAA) was investigated in this study as a novel blowing agent for AAFs prepared from metakaolin (MK) or blast furnace slag (BFS). The benefit of using PAA in comparison to H_2O_2 is lower stability of the O-O bond. Additionally, the use of PAA in AAFs provides acetate ions, which contribute to the enhanced dissolution of precursors through the chelation with cations and decreased surface tension of the gas-liquid interfaces creating open porosity. Setting time, porosity, mechanical strength, reaction kinetics, chemical composition, and volume increase of the foams prepared by PAA and H_2O_2 were studied and compared. The reduction in volume increase for PAA compared to H_2O_2 for both MK and BFS was 92-94% and 72-80%, respectively. Thus, this study reports the potentiality of using PAA for preparing foams with lower volume increases.

Friday, January 27, 2023

S3: 20th International Symposium on Solid Oxide Cells (SOC): Materials, Science and Technology

Interconnects and Coatings

Room: Ponce de Leon (North Tower)
Session Chairs: John Hardy, Pacific Northwest National Laboratory; Mihails Kusnezoff, Fraunhofer IKTS

8:30 AM

(ICACC-S3-062-2023) Protective-conducting ceramic coatings for SOFC steel interconnects (Invited)

T. Brylewski*[1]; L. Mazur[1]; S. Molin[2]; A. Gil[1]

1. AGH University of Science and Technology, Faculty of Materials Science and Ceramics, Poland
2. Gdansk University of Technology, Laboratory of Functional Materials, Faculty of Electronics, Telecommunications and Informatics, Poland

In the presented study, the surface of the Crofer 22 APU ferritic steel was modified with Gd_2O_3 nanoparticles and coated with a contact layer based on spinel or a spinel/perovskite composite. The objective was to determine if such modifications improve oxidation resistance and area-specific resistance and protect the cathode material. The tested samples were oxidized for 7000 h in air at 1073 K. Oxidation products were characterized using XRD, SEM-EDS, and confocal Raman imaging. The effect of gadolinium segregation at grain boundaries in Cr_2O_3 was evaluated using S/TEM-EDS. Area specific-resistance was measured and fuel cell tests investigating the electrochemical performance and Cr contamination of the electrodes were also performed. The dual modification proved to be more advantageous than either single modification. Tests comparing the performance of fuel cells before and after 250 h of aging in humidified hydrogen at 1073 K and involving an interconnect made of the dual-modified material showed that the electrochemical parameters of the aged cells were nearly identical to those of the non-aged reference electrode. In addition, the proposed modification was effective at mitigating Cr poisoning and protecting the electrode material. Acknowledgements: This project was financed by the National Science Centre, Poland, project number 2021/41/B/ST8/02187 (T. Brylewski).

9:00 AM

(ICACC-S3-063-2023) Electrophoretic deposition of ceramic coatings: From the spinel in-situ modification to the multilayer approach

F. Smeacetto*[1]; E. Zanchi[1]; H. Javed[4]; S. Molin[2]; A. R. Boccaccini[3]

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. University of Erlangen-Nuremberg, Institute of Biomaterials, Germany
4. Sunfire, Germany

Electrophoretic deposition is a viable and effective method for the co-deposition of spinel precursors and for designing in-situ modified ceramic coatings with improved functional properties. A systematic microstructural, thermo-mechanical and electrical characterization of simultaneous Fe-Cu doped MnCo spinel coatings processed by electrophoretic co-deposition on Crofer 22 APU is here reported and discussed. The innovative approach for the electrophoretic deposition of three spinel precursors is designed, conceived and optimised, to outline time- and energy-saving spinel modification routes as well as unravel the effect of mutual addition of Cu and Fe on the thermo-mechanical and corrosion properties of in-situ modified spinels. A multilayer system, processing a ceria inner layer is also electrophoretically deposited on both a 441 and Crofer22APU interconnect and tested at different atmospheres over time, to assess the effect of cerium in limiting Cr outward diffusion.

9:20 AM

(ICACC-S3-064-2023) Wet powder spraying derived spinel protective coatings for solid oxide cell interconnects

M. Wolff*[1]; C. Lenser[1]; N. H. Menzler[1]

1. Forschungszentrum Jülich GmbH, IEK-1, Germany

Cr diffusion and Cr_2O_3 scale formation are widely regarded as one major degradation issue for Cr-containing steel interconnects in solid oxide cells (SOC). Although the application of $(Mn,Co)_3O_4$ spinel by atmospheric plasma spraying (APS) has proven as an effective method to improve Cr retention, high processing costs still limit the potential of this approach. In this study, we evaluated wet powder spraying as a fast, scalable and cost-effective alternative to apply protective coatings of $Mn_{1.0}Co_{1.9}Fe_{0.1}0_4$ (MCF) on Crofer 22H. Deposition of homogeneous coatings have been shown to depend on a complex interplay of surface pre-treatment, slurry composition, wet powder spray process parameters and post-sintering conditions. Defect-free MCF layers with high adhesion strength have been successfully applied by gradual fine-tuning of deposition and sintering parameters. According to microstructural analysis, protective layers remained stable mechanically and chemically with improved Cr retention after thermal treatment for 1000 h at 800 °C.

Overall, wet powder spraying shows great potential as a highly efficient and economically viable technique for the application of gas-tight ceramic protective layers in the micrometer range.

9:40 AM

(ICACC-S3-065-2023) Ag-Based Brazes with Built-In Aluminum Getters to Produce Dense, Strong, and Low-Resistance Electrical Contacts on Al_2O_3-Protected Stainless Steel

G. Hu*[1]; J. D. Nicholas[1]

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

Alloying elements are commonly used to improve the oxidation resistance of stainless steel alloys. Specifically, chromium and aluminum form protective oxide surface scales that block oxygen transport to the underlying metal. Unfortunately for Solid Oxide Fuel Cells and other high-temperature, oxygen-exchange-enabled electrochemical devices, chromium can form volatile high temperature phases that can poison the oxygen exchange catalysts used in these devices. Similarly, aluminum can create sapphire scales that 1) block the electron transport needed in some applications and 2) make it difficult to braze to Al-containing stainless steel. Here, porous particle interlayers made from aluminum getters are shown to 1) facilitate the wetting, spreading, and adhesion of molten silver on various substrates, and 2) oppose the formation of sapphire surface scales. Al- and/or Cr- containing stainless steel joints brazed with this technique showed dense microstructures, high fracture strength, and low electrical resistance (in both the as-produced and long-term, air-annealed states).

10:20 AM

(ICACC-S3-066-2023) Application of Alumina Forming Stainless Steel on SOFC Stack for Automotive Use (Invited)

M. Suehiro*[1]; K. Ichihara[1]; T. Shiomi[1]; M. Abdul Jabbar[1]; P. Singh[2]; N. Dale[1]; Y. Furuya[1]

1. Nissan, USA
2. University of Connecticut, Materials Science and engineering, USA

To utilize SOFC power generator system as range extender in electric vehicles, it is important to increase volumetric power density of SOFC stack due to limited space availability in vehicles when compared to stationary needs. Based on previous research, utilization of AFA (alumina forming alloy) as base material for SOFC stack components facilitated the reduction of stack volume. In addition to the power density improvement requirements, AFA could prevent chemical degradation under harsh operating condition of typical SOFCs. Although AFA forms alumina layer on the surface, such layer protects the steel from degradation, however, the insulating alumina layer interfere with the electrical contacts, resulting in increased ASR (area specific resistance) in SOFC stack. Thus, to ensure high and reliable SOFC stack performance, AFA interconnect components must provide sufficient electrical conductance to achieve the required lower ASR. In our study, firstly, SOFC repeating unit structure has been constructed with special AFA interconnect parts and a metal supported cell. Secondly, diffusion bonding has been introduced to join AFA interconnect components. In this part, metallurgical and physical approaches have been considered to optimize insert materials and surface treatment for the efficient diffusion bonding. Finally, very low ASR has confirmed between AFA samples under SOFC operating test conditions.

10:50 AM

(ICACC-S3-067-2023) Chromium poisoning mitigation coatings for stainless steel interconnects and balance of plant protection in solid oxide electrolysis systems

S. Ibanez*[1]; E. Dogdibegovic[2]; A. Wallace[1]; D. Kopechek[1]; G. Arkenberg[2]; J. Funk[1]; P. Singh[3]; D. Ding[4]; M. Tucker[5]; S. Swartz[1]

1. Nexceris LLC, Fuel Cells and Electrolyzers, USA
2. Nexceris, USA
3. University of Connecticut, Materials Science and Engineering, USA
4. Idaho National Lab, Hydrogen and Electrochemistry, USA
5. Lawrence Berkeley National Laboratory, USA

Protective coatings are commonly applied to stainless steel interconnects and balance of plant components for solid oxide cell stacks. Such coatings reduce oxidation and Cr evaporation rates, leading to improved degradation rate and stack lifetime. In this work, two protective coatings are evaluated. ChromLok™ MCO-based composition $(Mn,Co)_3O_4$ is applied to Crofer 22 APU stainless steel. The MCO coating is found to decrease the stainless-steel oxidation rate by about one order of magnitude, and decrease the Cr evaporation rate by fourfold. The coating is demonstrated on a stack tested in solid oxide electrolysis mode (SOEC), which displays stable operation for 700 h. Another protective coating, $SrMnO_3$ (SMO), has proven effective for balance of plant components. The coating is either applied to ceramic monoliths or coated directly to the metal components using a bi-layer approach (AlumiLok™/SMO). The bi-layer coating is used in combination with MCO for air-side protection in single-cell SOEC testing. The AlumiLok™/SMO coating is deposited on tubing and manifold cavities, whereas the MCO coating is applied to the manifold surface, interconnect and current collector. The results show that the addition of balance of plant protection improved performance stability by 40%, when compared to SOEC test with only MCO coated interconnect.

S7: 17th International Symposium on Functional Nanomaterials and Thin Films for Sustainable Energy Harvesting, Environmental, and Health Applications

Nanotoxicity, Bio-imaging, Drug-delivery and Tissue Engineering with Tailored Nano-bioconjugates

Room: Coquina Salon G (North Tower)
Session Chair: Andreu Cabot, Catalonia Institute for Energy Research

9:00 AM

(ICACC-S7-015-2023) Multimodal Contrast Agents for Complementary XFCT-MRI Bioimaging (Invited)

G. Saladino[1]; B. Brodin[1]; H. M. Hertz[1]; M. S. Toprak*[1]

1. KTH Royal Institute of Technology, Dept. of Applied Physics, Sweden

Multimodal contrast agents in biomedical imaging enable the collection of more comprehensive diagnostic information. In the present work, we designed ruthenium-decorated superparamagnetic iron oxide nanoparticles (NPs) as the contrast agents for both magnetic resonance imaging (MRI) and X-ray fluorescence computed tomography (XFCT). The NPs are synthesized via one-pot polyol hot injection route, in diethylene glycol. In vivo preclinical studies demonstrate the possibility of correlative bioimaging with these contrast agents. The complementarity allows the accurate localization provided by the high contrast for the soft tissues in MRI combined with the elemental selectivity of XFCT, leading to NP detection with high specificity and resolution. We envision that this multimodal imaging could find future applications for early tumor diagnosis, improved long-term treatment monitoring, and enhanced radiotherapy planning.

9:30 AM

(ICACC-S7-016-2023) Light emitting porous silicon microparticles as optimized carriers for anticancer agents and immunologic enhancers (Invited)

A. Sambugaro[1]; E. Chisté[1]; C. Nardon[3]; M. Donini[2]; S. Dusi[2]; M. Scarpa[4]; N. Daldosso*[1]

1. University of Verona, Italy
2. University of Verona, Medicine - General Pathology, Italy
3. University of Verona, Biotechnology, Italy
4. University of Trento, Physics, Italy

The interesting properties of functionalized porous silicon microparticles, such as visible photoluminescence, large surface to volume ratio and easily-tunable surface to be functionalized, the fact that it is biodegradable, biocompatible and does not induce any immunogenicity nor toxicity, allow it to be an ideal carrier for theranostics purposes. We report herein about the experimental studies of porous silicon microparticles as a delivery system for anticancer agents (e.g. doxorubicin, docetaxel) and immunologic adjuvants, such as Pam3CSK4, a toll-like receptor (TLR) 1 and 2 agonist used as an immunologic adjuvant for immunotherapy. We performed preliminary in vitro tests of loading and release of the different drugs and, and the interaction between loaded pSi and dendritic cells (DCs) was studied by live-cell confocal microscopy. The immunologic response was evaluated by ELISA-assay showing a promising enhancement effect resulting from the synergic interaction between porous silicon microparticles and Pam3CSK4. Cytotoxicity tests on human non-small cell lung carcinoma were performed by comparing the effects of different dose release, showing a more efficient delivery with respect to anticancer drugs themselves.

Functional Coatings and Innovative Thin Film Techniques

Room: Coquina Salon G (North Tower)
Session Chair: Muhammet Toprak, KTH Royal Institute of Technology

10:20 AM

(ICACC-S7-017-2023) Electrostatic jet deflection to improve printing speed and resolution of electrohydrodynamic jet printing (Invited)

A. Cabot*[1]

1. Catalonia Institute for Energy Research, Spain

Electrohydrodynamic (EHD) jetting uniquely allows generating submicrometer jets that can reach speeds above 1 m s^{-1}, but such jets cannot be precisely collected by too slow mechanical stages. In this talk, I will present our progress in the control of the jet trajectory in EHD jetting technologies through a voltage applied to electrodes located around the jet. This method allows to continuously adjust the jet trajectory with lateral accelerations up to 10^6 m s^{-2}. Through electrostatically deflecting the jet, 3D objects with submicrometer features can be printed by stacking material layers on top of each other at layer-by-layer frequencies as high as 2000 Hz. The fast jet speed and large layer-by-layer frequencies achieved translate into printing speeds up to 0.5 m s^{-1} in-plane and 0.4 mm s^{-1} in the vertical direction, three to four orders of magnitude faster than techniques providing equivalent feature sizes.

10:50 AM

(ICACC-S7-018-2023) Cross-sectional Profiling of Photocarrier Mobility in Solar Cells via Nongeminate Recombination and Charge Extraction

N. B. Stocek[1]; G. Fanchini*[1]

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

The ability to spatially resolve the carrier mobility profile along the cross section of micrometer-thin solar cells is vital both for fundamental studies in photovoltaics and as a quality control for reproducibly obtaining high conversion efficiencies in commercial solar cell modules. Presently, no technique capable of such an endeavor is available to the best of our knowledge. Here, we introduce a novel method capable of profiling the carrier mobility along the z-axis in thin-film photovoltaics. Our setup is based on the integration of photogenerated charge extraction by linearly increasing voltage (p-CELIV) with a scanning confocal optical microscope (SCOM) towards a cross-sectional sensitive p-CELIV (cs-p-CELIV) system. As geminate recombination of excess carriers is the most frequent radiative pathway for electrons and holes in solar cells at low power density of illumination, while nongeminate recombination dominates at high power, enhanced nongeminaterecombinationoccursattheSCOMfocalplane.Thus,thecs-p-CELIV signal provides enhanced information on the mobility of all of the cross-sectional layers, except for the focal plane. The mobility profiles thus obtained correlate well with well-known depletion layers effects, and the hydrogen content content profile measured independently in hydrogenated amorphous silicon (a-Si:H) solar cells.

11:10 AM

(ICACC-S7-019-2023) Thermal and electrical conductivity properties controlled by manipulation of defect using ion implantation in ScN coatings

R. Burcea[1]; J. Barbot[1]; P. Renault[1]; D. Eyidi[1]; T. Girardeau[1]; M. Marteau[1]; F. Giovannelli[3]; A. Zenji[2]; J. Rampoux[2]; S. Dilhaire[2]; P. Eklund[4]; A. le Febvrier*[4]

1. University of Poitiers, Institut Pprime, France
2. Université de Bordeaux, Laboratoire LOMA, France
3. Université de Tours, Laboratoire GREMAN, France
4. Linkoping University, Dept. of Physics, Chemistry, and Biology, Sweden

The improvement of thermoelectric properties of ScN coatings was performed by injecting and manipulate defects by ion implantation of electrically active (Mg^+) and electrically non-active (Ar^+) ion. ScN has shown great potential for thermoelectric application but exhibit moderate thermal conductivity around 10-12 $Wm^{-1}K^{-1}$. Structural (XRD, TEM), optical, electrical and thermal properties were investigated with temperature. Generated defects using the two ions yielded to distinct types and natures of defect whom stability was limited in temperature at 400 K for the case of Mg^+ implantation, while stable up to 800 K for the Ar^+ implantation. In both situations, the electrical and thermal properties were altered with higher Seebeck coefficients, reduced electrical and thermal conductivities (12 to 3 $Wm^{-1}K^{-1}$) revealing the influence of defects in increasing phonon scattering, charge carrier concentration and mobility. The use of temperature, during implantation or after implantation, helps in controlling defects, thus controlling the type of defect generated or recovering partially physical properties of the coatings. We demonstrated the capability of a controlled introduction of defects, or defect engineering as a strategy for electrical / thermal properties management for the development of thermoelectric materials.

11:30 AM

(ICACC-S7-020-2023) Sol-gel spin-coated zinc oxide thin film as an efficient counter electrode in electrochromic devices

H. Ajitha Haridasan*[1]

1. FunGlass – Centre for Functional and Surface Functionalized Glass, Coating, Slovakia

Electrochromism is the modulation of optical properties caused by the redox process of an electrochromic (EC) material, which involves the intercalation/deintercalation process of ions in the lattice. EC materials primarily need to have a rapid and reversible redox characteristic. However, the appropriate choice of a complimentary material which undergoes the opposite electrochemical process to the redox process of the EC material is also one of the crucial factors. In this study, we demonstrate an electrochromic device with WO_3 thin film as the electrochromic material for the working electrode (WE) and ZnO thin film as a counter electrode (CE) fabricated on FTO glass substrates by a simple sol-gel spin coating method. We employed graphite-coated FTO as the CE for a comparative analysis of the ECDs. XRD studies indicate the WO_3 and ZnO films are well crystallized in monoclinic and wurtzite structures with crystallite sizes of 38.5 and 42.2 nm, respectively. The SEM image confirms the formation of porous and nanorod-like structures of WO_3 and ZnO, respectively. Furthermore, a transmittance modulation of 6.5 % was observed for graphite-CE-based ECD and enhanced optical contrast for ZnO-CE-based ECD (12.4%). The chronoamperometry results show that the current drawn for the coloring is higher in the case of graphite-CE-based ECD (6.9 mA.s) than that for ZnO-CE-based ECD (5.6 mA.s).

11:50 AM

(ICACC-S7-021-2023) Laser crystallization of thermochromic VO_2 thin films obtained by an environmentally friendly sol gel approach (Invited)

M. Basso[1]; E. Colusso[1]; C. Carraro[2]; E. Napolitani[2]; A. Martucci*[1]

1. University of Padova, Industrial Engineering, Italy
2. University of Padova, Physics, Italy

The peculiar optical properties of Vanadium dioxide, which can be controlled in function of the temperature, offer remarkable advantages for the thermal regulation of buildings: the employment of smart windows, coated with VO_2 thin films, allow a passive regulation of the thermal exchange. The open questions for VO_2 solution-based approaches are related to the safety issues and to the crystallization conditions. The toxicity of several vanadium precursors for the human health and for the environment underlines the need of alternatives. The variety of oxidation states of vanadium imposes a careful control of the atmosphere and the temperatures for its crystallization into VO_2. Here, a green sol-gel synthesis based on vanadium acetylacetone was employed for the deposition of VO_2 thin films on several types of substrates. The conventional furnace annealing was compared to the photonic curing of an excimer KrF laser. A systematic study of the different crystallization conditions was carried to evaluate the influence of the energy density and the number of pulses on the final properties. The comparison between the two annealing systems was done in terms of morphology, crystallinity, thickness and optical constants with several characterizations. The thermochromicity of the VO_2 was evaluated in function of the temperature with transmittance and reflectance measurements.

S9: Porous Ceramics: Novel Developments and Applications

Porous Ceramics for Functional Applications

Room: Coquina Salon H (North Tower)
Session Chairs: Tobias Fey, Friedrich-Alexander University Erlangen-Nürnberg; Tatsuki Ohji, National Institute of Advanced Industrial Science and Technology (AIST)

8:30 AM

(ICACC-S9-018-2023) Porous ceramics – why it's important to identify the representative volume-of-interest

T. Fey*[1]

1. Friedrich-Alexander University Erlangen-Nürnberg, Department Material Science and Engineering, Germany

Porous ceramic foams are used in a wide field of applications as catalyst support structures, lightweight materials, filtration, energy or acoustic adsorption or energy storage materials. Beside this, porous ceramic scaffold for bone replacement are a promising material in future to ensure a faster medical healing. In addition to determining the pore morphology, surface, connectivity and shape, the physical properties (thermal, mechanical, acoustic) are of crucial relevance for the applications mentioned. There is a difference here compared to dense ceramic materials, as the structural as well as physical properties are significantly shaped via the pores and porosity. Therefore, it is essential to determine the minimum volume, which allows a generally valid prediction for the entire structure. This so-called representative volume-of-interest (REVOI) can be determined by a combination of image evaluation based on μCT images and FE simulations in combination with physical measurements. Using examples of Al_2O_3 replica foams, porous Al_2O_3 ceramics via sacrificial templates and 3D printed structures, the principle and necessity of the representative volume is explained.

8:50 AM

(ICACC-S9-020-2023) Tracking Shape Memory Transformations in Porous Zirconia via Raman Spectroscopy

L. Quinn*[1]; K. Faber[1]

1. California Institute of Technology, USA

Through a diffusionless martensitic transformation between the monoclinic and tetragonal phases, ceria-stabilized zirconia exhibits shape memory and superelastic effects. However, a volume change that accompanies this transformation can cause strain mismatches at grain boundaries and ultimately cracking in bulk systems. To circumvent this premature failure, columnar, honeycomb-like pores can be incorporated into bulk-scale zirconia systems through freeze casting. This increases the samples' surface-to-volume ratio such that the martensitic transformation can be accommodated. In this work, porous ceria-stabilized zirconia ceramics are exposed to mechanical compression and thermal treatment to exhibit the full shape memory effect. While X-ray diffraction is commonly used to quantitatively track the extent of the transformation, Raman spectroscopy is a valuable tool with which to supplement XRD studies of these transformations. Here, Raman spectroscopy is used to provide a small-scale view of the shape memory transformation as it progresses in ceria-stabilized zirconia. Through this technique, we explore the advantages and challenges of studying shape memory zirconia in polycrystalline, bulk-scale zirconia.

9:10 AM

(ICACC-S9-021-2023) Sustainable Water Treatment via Ceramic Sorbent to Fertilizer Technology (Invited)

A. Apblett*[1]; C. Kelley[1]; P. Kitzel[1]

1. Oklahoma State University, USA

Eutrophication and hazardous algal blooms constitute serious environmental problems that affect waters throughout the world with severe impacts on human and animal health, and high economic costs. One of the key causes of these problems is plant nutrient enrichment that occurs when nutrients from agriculture, wastewater treatment plants, septic tanks, and urban water runoff enter waterways. We will report a sustainable technology that can decrease the incidence and severity of these problems by capturing plant nutrients using a sorbent that can subsequently be employed as a time release fertilizer. The sorbent is a ceramic fabricated from two inexpensive non-toxic earth abundant metal oxides that provides the capability of adsorbing nitrate, ammonia and phosphate. The material possesses excellent capacity for sorption of the highly difficult to capture nitrate ions due to a molecular imprinting process. Nutrient-loaded sorbent granules were applied as a slow-release fertilizer to effectively grow plants (radishes and marigolds) thus demonstrating the recovery and reuse of essential plant nutrients. Thus, a highly economical, scalable, and net cost reducing solution was developed for treatment of wastewater and contaminated natural waters. This novel technology will also curtail the need for synthesis of fertilizers and the attendant high environmental costs of phosphate production and nitrogen fixation.

9:40 AM

(ICACC-S9-022-2023) Novel glass-based sorbents for the stabilization of liquid nuclear waste

D. C. Lago*[2]; J. Kraxner[2]; E. Bernardo[1]

1. University of Padova, Department of Industrial Engineering, Italy
2. Centre for Functional and Surface Functionalized Glass, Italy

The aim of this work was to assess different novel glass-based sorbents for the immobilization of liquid nuclear waste surrogates. These materials were prepared through alkaline activation of a pharmaceutical boroaluminosilicate glass (BSG) and by stereolithography of yttrium alumino-silicate glass particles (YAS) suspended in acrylate resin added with silicone polymer (H44). The BSG was shaped in form of cubic castings, while YAS was printed in form of gyroid scaffolds. Highly porous structures were stabilized by the gelation of suspensions and curing at 40 °C (BSG-NaOH/KOH), or cross-linking, debinding and viscous flow sintering at 950 °C (YAS-H44). Simulated nuclear waste removal was carried out by immersion of porous structures in copper and neodymium aqueous solutions, respectively, followed by a final sintering step. The homogeneity of the incorporated waste and its distribution into the glasses were characterized through SEM/EDS analysis. The designed experimental procedure led to the development of novel metal ion sorbents structurally resistant to aqueous corrosion.

10:20 AM

(ICACC-S9-023-2023) Glyphosate adsorption performances of polymer-derived SiC/C aerogels

A. Zambotti*[1]; A. Bruni[1]; G. D. Soraru[1]; L. Rivoira[2]; M. Castiglioni[2]; E. Cagno[2]; B. Onida[3]; M. C. Bruzzoniti[2]

1. University of Trento, Industrial Engineering, Italy
2. University of Turin, Chemistry, Italy
3. Polytechnic of Turin, Applied Science and Technology, Italy

In the recent years glyphosate, a systemic herbicide, has come to a worldwide attention due to its possible health-related issues that are still far from being clarified. In the framework of water purification, a growing attention has been drawn to potential sorbents for its targeted removal, highlighting active carbons as good and relatively cheap candidates, thanks to their high specific surface area and microporosity. Despite their retention ability, the surface chemistry of active carbons is not optimal for the adsorption of glyphosate, which has an ionic characteristic that complicates its removal from aqueous solutions. In this work we propose polymer-derived ceramic aerogels as candidates for the removal of glyphosate from water. Polymer-derived ceramic aerogels are synthesized through a route that allows tuning their microstructure, i.e. porosity and specific surface area and, by selecting the polymeric precursors, their chemistry. Here, we present a highly porous nanocrystalline SiC/C aerogel with a glyphosate retention capacity exceeding 95%. Adsorption kinetics and isotherms were studied using pseudo-first and -second order and Elovich equations, and Langmuir, Freundlich and Temkin isotherm models were adopted. This work was supported by the Italian Ministry of Education, University and Research within the program PRIN-2017 "Nanostructured Porous Ceramics for Environmental and Energy Applications".

10:40 AM

(ICACC-S9-024-2023) Calcium/strontium chloride impregnated zeolite A and X granules as optimized ammonia sorbents

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

1. Lulea University of Technology, Division of Materials Science, Sweden

A stable, reliable ammonia carrier, therefore, is of desire for expanding the potential ammonia applications, such as clean fuel, and reductant in a selective catalytic reduction (SCR) system. Calcium chloride ($CaCl_2$) impregnated zeolite A and strontium chloride ($SrCl_2$) impregnated zeolite A and X composite granules were evaluated as ammonia sorbents for automotive selective catalytic reduction systems. The $SrCl_2$-impregnated zeolite A granules showed a 14% increase in ammonia uptake capacity (8.39 mmol g^{-1}) compared to zeolite A granules (7.38 mmol g^{-1}). Furthermore, composite granules showed 243% faster kinetics of ammonia sorption (0.24 mmol g^{-1} min^{-1}) compared to the $SrCl_2$ (0.07 mmol g^{-1} min^{-1}) in the first 20 min. The composite $CaCl_2/SrCl_2$ impregnated zeolite A granules combined the advantages of the zeolites and $CaCl_2/SrCl_2$, where the rapid physisorption from zeolites can reduce the ammonia loading and release time, and chemisorption from the $CaCl_2/SrCl_2$ which offers abundant ammonia capacity. Moreover, by optimizing the content of $SrCl_2$ loading, the composite granules maintained the granular form with a crushing load of 17 N per granule after ammonia sorption-desorption cycles. Such structural-stable composite sorbents offer an opportunity for fast ammonia loading/release in automotive selective catalytic reduction systems.

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*** Denotes Presenter**

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