The American Ceramic Society

48th International Conference & Exposition on Advanced Ceramics and Composites

ABSTRACT BOOK

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Introduction

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How to Use the Abstract Book

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

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Table of Contents

Plenary Session

13th Global Young Investigator Forum

Special Focused Session on Diversity Entrepreneurship and Commercialization

S2 Advanced Ceramic Coatings for Structural/Environmental & Functional Applications

S4 Protective Ceramics-Fundamental Challenges and New Developments

S13 Development & Applications of Adv Ceramics & Composites for Nuclear Fission/Fusion Energy Sys

S15 8th International Symposium on Additive Manufacturing and 3-D Printing Technologies

S17 Advanced Ceramic Materials and Processing for Photonics and Energy

S18 Ultra-High Temperature Ceramics

S19 Molecular-level Processing and Chemical Engineering of Functional Materials

S1 Mechanical Behavior and Performance of Ceramics & Composites

S3 21th Intl Symp on Solid Oxide Cells Materials Science & Technology

S5 Next-Generation Bioceramics and Biocomposites

S6 Advanced Materials and Technologies for Rechargeable Energy Storage

S8 18th Intl Symp on APMT for Structural & Multifunctional Materials & Systems

S15 8th International Symposium on Additive Manufacturing and 3-D Printing Technologies

S3 21th Intl Symp on Solid Oxide Cells Materials Science & Technology

S6 Advanced Materials and Technologies for Rechargeable Energy Storage

13th Global Young Investigator Forum

S13 Development & Applications of Adv Ceramics & Composites for Nuclear Fission/Fusion Energy Sys

S19 Molecular-level Processing and Chemical Engineering of Functional Materials

S8 18th Intl Symp on APMT for Structural & Multifunctional Materials & Systems

S5 Next-Generation Bioceramics and Biocomposites

13th Global Young Investigator Forum

S13 Development & Applications of Adv Ceramics & Composites for Nuclear Fission/Fusion Energy Sys

S15 8th International Symposium on Additive Manufacturing and 3-D Printing Technologies

S16 Geopolymers Inorganic Polymers and Sustainable Construction Materials

S17 Advanced Ceramic Materials and Processing for Photonics and Energy

S18 Ultra-High Temperature Ceramics

S19 Molecular-level Processing and Chemical Engineering of Functional Materials

S1 Mechanical Behavior and Performance of Ceramics & Composites

S3 21th Intl Symp on Solid Oxide Cells Materials Science & Technology

S5 Next-Generation Bioceramics and Biocomposites

S6 Advanced Materials and Technologies for Rechargeable Energy Storage

S8 18th Intl Symp on APMT for Structural & Multifunctional Materials & Systems

S2 Advanced Ceramic Coatings for Structural/Environmental & Functional Applications

S11 Advanced Materials and Innovative Processing Ideas for Production Root Technologies

S8 18th Intl Symp on APMT for Structural & Multifunctional Materials & Systems

S15 8th International Symposium on Additive Manufacturing and 3-D Printing Technologies

S6 Advanced Materials and Technologies for Rechargeable Energy Storage

13th Global Young Investigator Forum

S2 Advanced Ceramic Coatings for Structural/Environmental & Functional Applications

S11 Advanced Materials and Innovative Processing Ideas for Production Root Technologies

S13 Development & Applications of Adv Ceramics & Composites for Nuclear Fission/Fusion Energy Sys

S18 Ultra-High Temperature Ceramics

S19 Molecular-level Processing and Chemical Engineering of Functional Materials

S5 Next-Generation Bioceramics and Biocomposites

S8 18th Intl Symp on APMT for Structural & Multifunctional Materials & Systems

S16 Geopolymers Inorganic Polymers and Sustainable Construction Materials

S8 18th Intl Symp on APMT for Structural & Multifunctional Materials & Systems

13th Global Young Investigator Forum

S2 Advanced Ceramic Coatings for Structural/Environmental & Functional Applications

S13 Development & Applications of Adv Ceramics & Composites for Nuclear Fission/Fusion Energy Sys

S15 8th International Symposium on Additive Manufacturing and 3-D Printing Technologies

S15 8th International Symposium on Additive Manufacturing and 3-D Printing Technologies

S6 Advanced Materials and Technologies for Rechargeable Energy Storage

S8 18th Intl Symp on APMT for Structural & Multifunctional Materials & Systems

13th Global Young Investigator Forum

S11 Advanced Materials and Innovative Processing Ideas for Production Root Technologies

S13 Development & Applications of Adv Ceramics & Composites for Nuclear Fission/Fusion Energy Sys

S19 Molecular-level Processing and Chemical Engineering of Functional Materials

S16 Geopolymers Inorganic Polymers and Sustainable Construction Materials

S2 Advanced Ceramic Coatings for Structural/Environmental & Functional Applications

S13 Development & Applications of Adv Ceramics & Composites for Nuclear Fission/Fusion Energy Sys

S15 8th International Symposium on Additive Manufacturing and 3-D Printing Technologies

S16 Geopolymers Inorganic Polymers and Sustainable Construction Materials

S17 Advanced Ceramic Materials and Processing for Photonics and Energy

S18 Ultra-High Temperature Ceramics

S19 Molecular-level Processing and Chemical Engineering of Functional Materials

S1 Mechanical Behavior and Performance of Ceramics & Composites

S3 21th Intl Symp on Solid Oxide Cells Materials Science & Technology

S6 Advanced Materials and Technologies for Rechargeable Energy Storage

S7 18th Intl Symp on Functional Nanomaterials & Thin Films for Sustainable Energy Harvesting

S8 18th Intl Symp on APMT for Structural & Multifunctional Materials & Systems

S9 Porous Ceramics Novel Developments and Applications

S11 Advanced Materials and Innovative Processing Ideas for Production Root Technologies

S16 Geopolymers Inorganic Polymers and Sustainable Construction Materials

S2 Advanced Ceramic Coatings for Structural/Environmental & Functional Applications

S15 8th International Symposium on Additive Manufacturing and 3-D Printing Technologies

S6 Advanced Materials and Technologies for Rechargeable Energy Storage

S11 Advanced Materials and Innovative Processing Ideas for Production Root Technologies

S13 Development & Applications of Adv Ceramics & Composites for Nuclear Fission/Fusion Energy Sys

S16 Geopolymers Inorganic Polymers and Sustainable Construction Materials

S19 Molecular-level Processing and Chemical Engineering of Functional Materials

S8 18th Intl Symp on APMT for Structural & Multifunctional Materials & Systems

FS1 Bioinspiration/Green Processing & Related Technologies of Advanced Materials

FS3 Nanostructures and Low-Dimensional Materials for Chemical Sensors

S2 Advanced Ceramic Coatings for Structural/Environmental & Functional Applications

S10 Modeling and Design of Ceramics and Composites

S12 Design and Applications of Nanolaminated Ternary Transition Metal Carbides/Nitrides and Borides, Their solid solutions and 2D Counterparts

S13 Development & Applications of Adv Ceramics & Composites for Nuclear Fission/Fusion Energy Sys

S15 8th International Symposium on Additive Manufacturing and 3-D Printing Technologies

S16 Geopolymers Inorganic Polymers and Sustainable Construction Materials

S18 Ultra-High Temperature Ceramics

S1 Mechanical Behavior and Performance of Ceramics & Composites

S3 21th Intl Symp on Solid Oxide Cells Materials Science & Technology

S6 Advanced Materials and Technologies for Rechargeable Energy Storage

S7 18th Intl Symp on Functional Nanomaterials & Thin Films for Sustainable Energy Harvesting

S8 18th Intl Symp on APMT for Structural & Multifunctional Materials & Systems

S9 Porous Ceramics Novel Developments and Applications

S8 18th Intl Symp on APMT for Structural & Multifunctional Materials & Systems

S9 Porous Ceramics Novel Developments and Applications

S15 8th International Symposium on Additive Manufacturing and 3-D Printing Technologies

S6 Advanced Materials and Technologies for Rechargeable Energy Storage

S13 Development & Applications of Adv Ceramics & Composites for Nuclear Fission/Fusion Energy Sys

S7 18th Intl Symp on Functional Nanomaterials & Thin Films for Sustainable Energy Harvesting

S8 18th Intl Symp on APMT for Structural & Multifunctional Materials & Systems

S16 Geopolymers Inorganic Polymers and Sustainable Construction Materials

FS1 Bioinspiration/Green Processing & Related Technologies of Advanced Materials

S9 Porous Ceramics Novel Developments and Applications

S8 18th Intl Symp on APMT for Structural & Multifunctional Materials & Systems

FS1 Bioinspiration/Green Processing & Related Technologies of Advanced Materials

FS2 Advanced Materials for Thermoelectric and Thermionic Energy Conversion

FS3 Nanostructures and Low-Dimensional Materials for Chemical Sensors

FS5 High Voltage Materials for Advanced Electrical Applications

S10 Modeling and Design of Ceramics and Composites

S12 Design and Applications of Nanolaminated Ternary Transition Metal Carbides/Nitrides and Borides, Their solid solutions and 2D Counterparts

S14 Crystalline Materials for Electrical Optical and Medical Applications

S1 Mechanical Behavior and Performance of Ceramics & Composites

S3 21th Intl Symp on Solid Oxide Cells Materials Science & Technology

S6 Advanced Materials and Technologies for Rechargeable Energy Storage

S7 18th Intl Symp on Functional Nanomaterials & Thin Films for Sustainable Energy Harvesting

S9 Porous Ceramics Novel Developments and Applications

S2 Advanced Ceramic Coatings for Structural/Environmental & Functional Applications

FS1 Bioinspiration/Green Processing & Related Technologies of Advanced Materials

S6 Advanced Materials and Technologies for Rechargeable Energy Storage

S2 Advanced Ceramic Coatings for Structural/Environmental & Functional Applications

S9 Porous Ceramics Novel Developments and Applications

FS1 Bioinspiration/Green Processing & Related Technologies of Advanced Materials

FS2 Advanced Materials for Thermoelectric and Thermionic Energy Conversion

FS3 Nanostructures and Low-Dimensional Materials for Chemical Sensors

FS4 Ceramic/Carbon Reinforced Polymers

FS5 High Voltage Materials for Advanced Electrical Applications

S10 Modeling and Design of Ceramics and Composites

S12 Design and Applications of Nanolaminated Ternary Transition Metal Carbides/Nitrides and Borides, Their solid solutions and 2D Counterparts

S14 Crystalline Materials for Electrical Optical and Medical Applications

S1 Mechanical Behavior and Performance of Ceramics & Composites

S3 21th Intl Symp on Solid Oxide Cells Materials Science & Technology

S6 Advanced Materials and Technologies for Rechargeable Energy Storage

S7 18th Intl Symp on Functional Nanomaterials & Thin Films for Sustainable Energy Harvesting

S9 Porous Ceramics Novel Developments and Applications

S6 Advanced Materials and Technologies for Rechargeable Energy Storage

FS1 Bioinspiration/Green Processing & Related Technologies of Advanced Materials

FS4 Ceramic/Carbon Reinforced Polymers

S1 Mechanical Behavior and Performance of Ceramics & Composites

S7 18th Intl Symp on Functional Nanomaterials & Thin Films for Sustainable Energy Harvesting

FS2 Advanced Materials for Thermoelectric and Thermionic Energy Conversion

S10 Modeling and Design of Ceramics and Composites

S14 Crystalline Materials for Electrical Optical and Medical Applications

Monday, January 29, 2024

Plenary Session

Plenary Session
Room: Coquina D/E
Session Chairs: Young-Wook Kim, University of Seoul; Jie Zhang, Institute of Metal Research, Chinese Academy of Sciences

8:50 AM

(ICACC-PLEN-001-2024) James I. Mueller Memorial Award: Better Properties Do Not Always Yield Better Performance: Mechanism-Based ApproachFor Understanding the Impact Behavior of Ceramics

G. Subhash*[1]

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

The bed-rock principle of materials science 'better properties yield better performance' holds good for applications involving elastic response of materials and structures. However, such a doctrine falls short in materials designed to resist penetration during high velocity impact. In these applications, a material's inelastic and fracture behaviors play a dominant role in providing better performance although its mechanical properties may be inferior to its competing alternatives. Three examples are highlighted to illustrate the importance of mechanisms-based approach in understanding this behavior: (i) Boron carbide is lighter and stronger than silicon carbide, but the latter is preferred in light-weight protective systems; (ii) spinel outperforms sapphire in high-velocity impact studies despite having lower strength and stiffness; Finally, (iii) a historic example is presented where the oldest fort in the US, the Castillo de St Marcos, St. Augustine, FL, withstood cannon-ball attack by the British Army against the Spanish fort in 1700s even though the fort walls were built with a highly porous sedimentary rock called 'Coquina', which is simply an aggregate of loosely bonded seashell fragments.

9:30 AM

(ICACC-PLEN-002-2024) ECD Bridge Building Award: Novel single crystals for electro-optical applications

K. Shimamura*[1]

1. National Institute for Materials Science, Japan

Electro-optical technology progress in a wide range of applications, and still demands the further development. Our intention is to explore novel single crystal materials for diverse applications, and to implement them in the industrial use. β-Ga2O3 single crystal was proposed as a brandnew wide-gap (Eg = 4.8 eV) semiconductor in 2001. 2 inch size β-Ga2O3 single crystals were grown by the EFG technique. Shotky barrier diode of it was demonstrated in 2009. Since then, tremendous considerations for the industrial implementation have been on going. A new concept of high-brightness white LED/LDs based on Ce: Y3Al5O12 (YAG) single crystal phosphor plates (SCPPs), which can overcome the conventional temperature- and photo-degradation problems, has been proposed. SCPPs demonstrated excellent thermal stability, high values of luminous efficacy and increased quantum efficiency. The implementation has started in 2017. Tb3(Sc1-xLux)2Al3O12 (TSLAG) single crystals have been designed for high-power laser machinery. It showed a higher visible transparency and a larger Faraday rotation than the conventional Tb3Ga5O12 (TGG). In 2013, mass production has started. A drastic enhancement of the light yield of Ce:Li6Y(BO3)3 (LYBO) single crystals by ~600% is achieved. Ce:LYBO could be of interest as efficient, low-cost, and stable solid-state materials for portable thermal neutron detection.

10:40 AM

(ICACC-PLEN-003-2024) Plenary: Multifunctional ceramic, polymer and composite biomaterials for bone tissue regeneration and treatment

E. Pamula*[1]

1. AGH University of Science and Technology, Department of Biomaterials and Composites, Poland

Infected or critical size bone injuries that do not heal spontaneously need special therapies and/or tissue engineering approach. Biomaterials supporting the treatment of such bone lesions should be designed to kill pathogenic bacteria, followed by facilitating osteogenic cell signalling and bone tissue expression processes. In our research, we design multifunctional biomaterials that provide mechanical support and are integrated with bone tissues (titania and zirconia ceramics) or are made of biodegradable polymers, so their disappearance from the treated lesion is correlated with the ingrowth of native tissue. Both types of biomaterials can be modified to deliver drugs or biologically active substances to support bone tissue regeneration. We found that the deposition of calcium phosphate, collagen, or sulphated hyaluronan on polymer scaffolds promotes cell osteogenic differentiation and the healing of osteochondral defects [3,4]. We also developed biomaterials dedicated to infected bone in the form of polymeric micro-/nanoparticles loaded with antibiotics [1,2,5,6]. They can be used as a component of injectable matrices [5,6] or be immobilised on the pore walls of scaffolds, to obtain implantable medical devices, when mechanical support is particularly essential [1,2]. The developed biomaterials release the drugs in a controlled manner to be adapted to the clinical needs.

11:20 AM

(ICACC-PLEN-004-2024) Plenary: Materials and Designs to Mitigate Thermal Runaway Propagation in Lithium-ion Cell and Battery Shipments

J. Jeevarajan*[1]

1. UL Resarch Institutes, Electrochemical Safety Research Institute (ESRI), USA

Our research studies on various materials and designs to determine their efficacy in preventing the propagation of thermal runaway in lithium-ion cells and modules as prepared for shipment will be presented.

13th Global Young Investigator Forum

13th Global Young Investigator Forum: Microstructure, thermo-mechanical properties
Room: Coquina D
Session Chairs: Meelad Ranaiefar, NASA Glenn Research Center; Yan Chen, Oak Ridge National Lab

1:30 PM

(ICACC-GYIF-001-2024) Irradiation, nanomechanical and thermal performance of high entropy carbides for nuclear applications (Invited)

K. Bawane*[1]; L. Trinh[3]; Z. Hua[1]; L. Wadle[3]; L. Malakkal[1]; L. He[2]; B. Cui[3]

1. Idaho National Laboratory, USA
2. North Carolina State University, Nuclear Engineering, USA
3. University of Nebraska–Lincoln, Mechanical & Materials Engineering, USA

The concept of multi-principal element represents the state-of-the-art and provides a novel direction for the design of materials with exceptional properties for extreme environments particularly in nuclear reactors. Although still in early stages of development, high entropy carbides (HECs) have shown the potential to outperform traditional ceramic materials. This talk will present an overview of irradiation behaviors, nanomechanical and thermal performance of novel HECs. Spark plasma sintering was used to fabricate dense HECs with different compositions (ZrC, (ZrHf)C, (ZrHfNb)C, (ZrHfNbTa)C, (ZrTaNbTi)C, (ZrTaTiNbW)C, (ZrHfNbTaTi)C). Laser sintering approach was also used to fabricate (ZrTaTiNbW)C HEC. Irradiation was performed on select compositions using high energy Zr ions. Mechanical performance was studied using in-situ micropillar and in-situ nanopillar compression testing inside scanning electron microscope (SEM) and transmission electron microscope (TEM), respectively. Thermal properties were measurements using a spatial-domain thermal reflectance (SDTR) technique. Microstructure damage after irradiation and deformation was revealed using TEM characterization.

2:00 PM

(ICACC-GYIF-002-2024) Irradiation-induced dimensional and stress changes in carbon coatings of TRISO fuel particles (Invited)

Q. Zhang*[1]; H. Huang[1]; J. McGladdery[2]; N. Tzelepi[2]; D. Goddard[3]; S. Knol[4]; J. A. Vreeling[4]; M. Davies[5]; D. Liu[6]

1. University of Bristol, School of Physics, United Kingdom
2. National Nuclear Laboratory, Central Laboratory, United Kingdom
3. National Nuclear Laboratory, Preston Laboratory, United Kingdom
4. NRG, Netherlands
5. Ultra Safe Nuclear Corporation, USA
6. University of Bristol, United Kingdom

To achieve the net-zero commitment, UK government has set an ambition of 24 GW nuclear capacity by 2050 for reliable and low carbon power, which accelerates the development and deployment of tri-structural isotropic (TRISO) coated fuel particles for High Temperature Reactors. To understand the influence of high temperature fast neutron irradiation on the microstructure and mechanical properties of carbon coatings in TRISO particles, in this work, two types of surrogate TRISO particles, PyC-1 (Kernel/Buffer/PyC) and PyC-buffer (Kernel/Buffer) from PYCASSO (Pyrocarbon irradiation for Creep And Swelling/Shrinkage of Objects) experiments were studied. Thickness changes in buffer and PyC layers after low dose (1.63 ×10^25 n/m^2) and high dose (2.02 ×10^25 n/m^2) neutron irradiations at 1000°C were evaluated using X-ray computed micro-tomography aided with Dragonfly AI segmentation, showing differences of irradiation-induced shrinkage in buffer and PyC layers. Residual stresses in buffer and PyC layers of as-manufactured particles and high/low-dose neutron-irradiated particles were compared using Focused Ion Beam-Digital Image Correlation. This work found that residual stress in the PyC layer of PyC-1 particles changed from tensile tangential stress to compressive tangential stress after neutron irradiation, due to the irradiation-induced dimensional behavior of the buffer and PyC coatings.

2:30 PM

(ICACC-GYIF-003-2024) Thermochemical and microstructural contributions of high temperature particle erosion durability in CMAS exposed EBCs (Invited)

J. L. Stokes*[2]; M. J. Presby[2]; L. C. Hoffman[3]; J. A. Setlock[1]; B. J. Harder[2]

1. University of Toledo, Environmental Effects and Coatings Branch, USA
2. NASA Glenn Research Center, Environmental Effects and Coatings Branch, USA
3. HX5, LLC, Environmental Effects & Coatings, USA

Particulate/debris damage caused by ingestion of calcium magnesium aluminosilicates (CMAS) hinders the use of environmental barrier coatings (EBCs) to protect SiC-based ceramic matrix composite components in next generation gas turbine engines. Similarly, ingestion of any debris in the engine can lead to mechanical damage and recession of coatings due to particulate erosion. Investigating particulate interactions at relevant engine conditions is crucial in determining limiting mechanisms in the operating lifetime of EBCs. This study assesses the effects of extrinsic phase formation and microstructural changes due to CMAS interactions on the erosion durability of $Yb_2Si_2O_7$-based EBCs 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 on erosion durability at various impingement angles were evaluated.

Special Focused Session on Diversity Entrepreneurship and Commercialization

Special Focused Session on Diversity, Entrepreneurship, and Commercialization

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

1:30 PM

(ICACC-DIV-001-2024) Jubilee Global Diversity Award: Nanostructured bioceramics as a multifunctional delivery platform for the regeneration of functional (hard and soft) tissues

S. Fiorilli*[1]

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

To overcome the challenges associated with compromised tissue regeneration, extensive research efforts are currently devoted to the design of clinical solutions able to simultaneously target the multiple and interlocked causes underlying unsuccessful healing. Due to the extremely high surface area for storage and delivery of therapeutics (i.e. drugs, ions, biomolecules), as well as the easy post-functionalisation to impart on-demand release properties, nanostructured bioceramics are successfully exploited as smart multifunctional nanocarriers in the field of tissue regeneration. This contribution will provide an overview of the research activities, mostly in the framework of collaborative projects funded by the European Union, regarding the inclusion of bioactive nanocarriers into composite 3D scaffolds, injectable cements and flexible nanofibrous patches, as advanced approaches for preventing adverse remodelling in bone, skin and cardiac tissues. Comprehensive physical-chemical characterisation and in vitro/in vivo biological assessment conducted in collaboration with clinicians are also reported, highlighting the related open challenges and future perspectives.

2:10 PM

(ICACC-DIV-002-2024) Jubilee Global Diversity Award: Creation of a new resource circulation loop realized by diversity and new separation technology

C. Tokoro*[1]

1. Waseda University, Japan

The circular economy is attracting attention as a concept that combines environmental load reduction, such as carbon neutrality, with resource circulation. This concept aims to achieve a harmonious well-being of the economy, society, and the environment through various resource circulation loops, including sharing, maintenance, reuse, and recycling. To realize this goal, we need to have diverse values for products. In addition, diverse and varied separation technologies are necessary for products reclamation. Based on this concept, the authors have been studying high-precision and energy-saving solid separation technologies to create a new resource circulation loop. For example, we have developed a high-precision and energy-saving separation technology for metallic particles, foils, and wires from a spent product without using chemicals or heating. These separation technologies are effective for separating metals from products such as lithium-ion batteries and solar panels, for which demand is expected to grow rapidly toward carbon neutrality, and we have confirmed that these technologies are advantageous from the LCA perspective.

3:10 PM

(ICACC-DIV-003-2024) Jubilee Global Diversity Award: Design and Manufacturing of New Functional Ceramic Composites

Y. Li*[1]

1. Dartmouth University, Thayer School of Engineering, USA

The route of devising polymer-derived ceramics (PDCs), which relies on heat treatment to convert preceramic polymers to ceramics, presents a flexible and energy-efficient approach to fabricate a broad spectrum of ceramics and in-situ ceramic-polymer composites with binary or multinary phases. Understanding the relationship among processing parameters, phase composition and material response holds an important key for property tailoring of PDC composites in different engineering applications. In this presentation, I will talk about how to tailor the mechanical response through structure architecture design, phase transition control and energy dissipation pathway prediction. A few case studies will be provided to discuss the role of PDCs in healthcare, renewable energy and aerospace applications.

3:50 PM

(ICACC-DIV-004-2024) Commercialization of high temperature electrolysis for green hydrogen and syngas production (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 and syngas. We present new results for ceramic based steam electrolysis systems: SOE (solid oxide electrolysis). SOE offers three essential unique advantages: 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. 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. Complete reversibility. SOE systems can be operated in the reverse mode, i.e they can be used as fuel cells using hydrogen or/and syngas or other hydrocarbon fuels to generate power. 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 challenge in a future hydrogen economy. For commercialization and up-scaling of SOE-Stack production is essential. We give an up-date on this issue.

4:20 PM

(ICACC-DIV-005-2024) Team Development for Improving Project Outcomes (Invited)

M. Reigel*[1]

1. Saint-Gobain Ceramics & Plastics, USA

Working in teams is a necessary and expected part of almost any STEM career. However, as scientists and engineers, we are rarely taught the skills needed to build a successful team. Studies have shown that diversifying your team leads to better ideas, productivity, and outcomes. But what does diversity mean and how can you achieve it? Using examples from her own experiences and work on diversity efforts with ACerS and ASM International, Dr. Reigel will discuss details to consider when putting together a new team, the impact a team can have on how you develop a product, and how diversity can help foster a culture of inclusion and success.

4:50 PM

(ICACC-DIV-006-2024) On the design of novel perovskites for functional applications

A. Azure*[2]; S. Gupta[1]

1. University of North Dakota, Mechanical Engineering, USA
2. United Tribes Technical College and University of North Dakota, USA

The talk will be divided into two parts. During part-A, the lead presenter, Ms. Alexa Azure, will give her professional journey as a enrolled member of the Standing Rock Sioux Tribe. She began her college journey at United Tribes Technical College (UTTC) in the Tribal Environmental Science and Art/Art Marketing. Upon graduation, she transferred to the University of North Dakota for Chemical Engineering, earning her bachelor's degree in 2012. Shortly after she graduated, the department chair of Tribal Environmental Science at UTTC reached out to her as their engineering instructor left mid-semester. In 2018, Azure was awarded the American Indian College Fund Faculty of the Year or UTTC and Chapter Advisor of the Year through the American Indian Science and Engineering Society (AISES). Azure has gone from the sole engineering instructor to the general education chair to most recently, the engineering chair. In part-B, Azure will explain her research in University of North Dakota as a doctoral scholar on synthesis and characterization of novel pervoskites.

S2 Advanced Ceramic Coatings for Structural/Environmental & Functional Applications

SYMPOSIUM 2: Thermal Barrier Coatings I

Room: Flagler C
Session Chairs: Peter Mechnich, DLR - German Aerospace Center; Douglas Wolfe

1:30 PM

(ICACC-S2-001-2024) High Temperature Oxidation Behaviour of Ni-based Alumina-Forming Bond Coat Materials: A Brief Survey (Invited)

J. Jedlinski*[1]

1. AGH University of Kraków, Faculty of Materials Science and Ceramics, Physical Chemistry and Modeling of Processes, Poland

The β-NiAl intermetallic compound and γ/γ' Ni-Al-based alloys, which contain small amounts of reactive elements (Hf, Y, …) and higher contents of Pt (or Pd, Rh), are applied as Bond Coats in Thermal Barrier Coating systems on superalloys. Their superior high temperature oxidation resistance stems from the formation of α-Al2O3 protective scale. The additions provide significant improvement of the scale protectiveness in particular upon thermal cyclic conditions. Vast evidence of such influence does not, however, result in sufficient understanding of the observed effects. Therefore, it is difficult to formulate premises concerning the composition and microstructure of the Bond Coat layers. It is proper to add that the Bond Coat layer in Thermal Barrier Coating system is essentially responsible for the protection efficiency of the entire system against degradation caused by hot gases. This paper is focused on a comprehensive summary of the state of the art in this field, critical discussing the major issues and problems as well as on challenges related to the optimizing the design of the possibly best effective Bond Coats. The most important general issues are related to describing the kinetics and mechanisms of oxidation, the effect of exposure conditions, materials composition and microstructure as well as to phenomena affecting the interfacial Bond Coat - α-Al2O3 layer strength.

2:00 PM

(ICACC-S2-002-2024) On Lifetime Prediction of Thermal Barrier Coatings

K. Chen*[1]

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

Fracture mechanics and damage mechanics were often combined to conduct lifetime prediction of thermal barrier coating (TBC) systems. One of these approaches typically used subcritical crack growth and/or propagation rate formula to predict TBCs lifespan. This lifetime dominated by crack growth mechanism was supported by either furnace cycle test or burner rig test of TBCs in terms of the observed life data and microstructural evolution. However, there are also test data in terms of crack density and its crack length evolution versus thermal cycles, in which the results demonstrate that it is the crack nucleation density rather than its crack growth rate that dominates the lifespan of TBCs. In this presentation, a mechanics/physics-based model was proposed regarding the crack nucleation density and its associated crack growth length versus thermal cycles. The net crack density between the competition of crack nucleation density and reduction of crack density due to crack growth and/or merged cracks during thermal cycles represents the experimentally measured crack density. The energy release rate and the fracture toughness kinetic evolution versus thermal cycles were also formulated. The proposed model was then applied to evaluate the lifespan of TBCs. A comparison was made of the proposed model with both crack growth dominated lifetime model and the crack nucleation dominated method.

2:20 PM

(ICACC-S2-003-2024) TBC Thermal Cycling Tests: Laser Rig Test vs Simulated Flame Torch Rig Test

Y. Wang*[1]; L. Zhao[1]; P. Hsu[1]

1. Florida Institute of Technology, Mechanical Engineering, USA

Thermal barrier coatings (TBCs) are critical for the gas turbine hot components design and performance. There is a strong interest in understanding the performance and endurance of TBC systems. For that purpose, a high-temperature, high heat flux, temperature gradient test rig is a more cost-effective test method than an engine testing stand. Although there has been an ISO testing standard, ISO 13123 Metallic and other inorganic coatings - Test method of cyclic heating of TBCS under temperature gradient, most of the temperature gradient test rigs do not follow exactly the requirements given in the standard. Additionally, there are conflicting statements given in the standard. In this study, the laser rig test results were compared with the flame torch test results. SEM images of sample cross sections after the test by both methods are presented and discussed. Test samples were under the initial 150 hours of kiln heating to generate sufficient thickness of thermally-grown oxide layers and then placed in the laser rig under different heating conditions until 20% of topcoat delamination was detected. The flame torch test is simulated by a heating pattern provided by the laser rig. The reason for using laser heating to simulate flame torch heating is to ensure well-controlled temperature conditions in both test methods. A detailed discussion of the laser rig design and functions is also given.

2:40 PM

(ICACC-S2-004-2024) Development of Luminescence Lifetime-Based Surface Temperature Mapping for Environmental Barrier Coatings

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

Luminescence lifetime-based temperature mapping for thermal barrier coatings (TBCs) has been previously demonstrated up to temperatures approaching 1200 °C. This approach requires a higher temperature capability for useful application for SiC/SiC ceramic matrix composite components protected by environmental barrier coatings (EBCs) from below 1200 °C up into the 1300 to 1500 °C temperature range. The much higher background thermal radiation at these higher temperatures is a challenging issue. In addition, the luminescing sensing layer must exhibit thermochemical compatibility with the underlying EBC. To meet these requirements, Er-doped Y_2SiO_5 was selected as the luminescing sensing layer material both for its enhanced temperature sensitivity above 1300 °C and its compatibility with rare-earth disilicate EBC topcoats. Both localized spot temperature measurements as well as luminescence lifetime imaging-based temperature mapping were demonstrated up to 1560 °C for Er-doped Y_2SiO_5 disks and up to 1380 °C from a 15 μm thick Er-doped Y_2SiO_5 layer at the surface of a $Sc_2Si_2O_7$-based EBC topcoat. No degradation of the Er-doped Y_2SiO_5 temperature sensing surface layer was observed. The source of the novel enhanced temperature sensitivity will be discussed as well as a path towards the compatibility between the sensing layer and EBC topcoat reaching higher temperatures.

3:20 PM

(ICACC-S2-005-2024) Rare earth and transition metal doping of ZrO_2-$YTaO_4$ for radiative barrier coatings

V. Champagne*[1]; J. Deijkers[2]; C. Lothrop[2]; D. Clarke[1]; H. N. Wadley[2]

1. Harvard University, Materials Science, USA
2. University of Virginia, USA

To increase the thermal efficiency of gas turbines, higher turbine inlet temperatures are necessary. By the Stefan-Boltzmann law, as temperature increases, the total energy radiated increases by temperature to the fourth power. However, most low thermal conductivity oxides currently used as TBCs like 7YSZ and GZO are transparent in the ‘thermal radiation window’ which is the wavelength range in which most of the energy flux in a high temperature environment like a gas turbine occurs. In this work, we substituted rare earths like Yb which exhibit strong, selective absorption and transition metals like Fe which exhibit broader absorption, both in the thermal radiation wavelength range, in the ZrO_2-$YTaO_4$ system to produce radiative barrier coatings. While it is important for high temperature coatings to absorb and block IR radiation, other important properties such as phase stability, low thermal conductivity, and minimal CTE mismatch with the substrate material must be maintained. By designing coatings with these favorable properties, higher temperature-capable coatings can be realized.

3:40 PM

(ICACC-S2-006-2024) A Thermal Conductivity Model of Porous Air Plasma Sprayed Yttrium-Stabilized Zirconia Coatings

L. Zhao*[1]; P. Hsu[1]

1. Florida Institute of Technology, Mechanical Engineering, USA

A micro-structure based effective thermal conductivity of a porous yttrium-stabilized zirconia (YSZ) coatings prepared by air-plasma spray (APS) method is compared with the measurement conducted in a laser rig. An analytical model is developed to predict the conductivity of the porous material based on its porosity and morphology, incl. the pore size distribution, pore aspect ratio, and pore orientation relative to the heat flow direction. The model is verified through comparisons with numerical simulation results using a commercial Multiphysics software tool. The experimental measurement of the YSZ topcoat conductivity is obtained in a laser rig. The laser rig uses a 2 kW CO2 laser as the heating source, in combination of specially designed scan patterns and sample holder, to produce a uniform temperature distribution on the heated topcoat surface. In the rig, a one-dimensional temperature gradient through the thickness of coated button sample is established. The sample is a 0.3 mm YSZ topcoat deposited on the bond coated Inconel 718 substrate disc. The topcoat conductivity is inverted from the overall conductivity obtained from the test rig. The comparison of the model predicted conductivity is within 20% of the measured value. Possible discrepancies and improvements to the model and experimental method are discussed.

S4 Protective Ceramics-Fundamental Challenges and New Developments

SYMPOSIUM 4: Protective Ceramics - Fundamental Challenges and New Developments

Room: Ponce de Leon
Session Chair: Kristopher Behler, DEVCOM-Army Research Lab

1:30 PM

(ICACC-S4-001-2024) Reactive Infiltration of Silicon Melt into C/C Preforms: Mechanism, Modeling, and Disturbing Effects (Invited)

P. J. Hofbauer*[1]

1. ArianeGroup, High Temperature Materials & Technologies, Germany

The Liquid Silicon Infiltration (LSI) process, involving the infiltration of molten silicon into porous structures, has been employed for decades in the production of C/C-SiC and C/SiC, as well as for environmental barrier coatings. However, the phenomena at the infiltration front have not been sufficiently understood, leading to high rejection rates and variations in quality. One contributing factor is the presence of temperature gradients, generated by the intense exothermal reaction during the silicon infiltration into porous carbon preforms to form SiC. To mitigate the risk of cracks caused by thermal stress, an understanding of the infiltration and reaction kinetics is essential. Another significant factor is the presence of oxygen impurities in the furnace atmosphere during infiltration. These impurities can substantially disrupt the siliconization process, resulting in discoloration of component surfaces and incomplete infiltration of the pore channels. This presentation elucidates the mechanisms occurring at the infiltration front, provides a model for describing infiltration and reaction kinetics, and highlights the disruptive effects of oxygen. Furthermore, it offers measures to minimize these negative effects.

2:00 PM

(ICACC-S4-002-2024) Reactive melt infiltration of liquid silicon based alloys into porous carbonaceous materials: Modelling and 1D to 3D experimental validation (Invited)

M. Naikade[2]; T. Graule[2]; L. Weber[3]; A. Ortona*[1]

1. SUPSI, MEMTi, Switzerland
2. Empa, Laboratory for High Performance Ceramics, Switzerland
3. EPFL, Switzerland

An idealising mathematical model describing the reactive flow of Si-X alloy in a single micron sized capillary channel of carbon as well as in carbonaceous preforms is presented. The model is further expanded to evaluate the infiltration depth in porous carbonaceous preform for a given composition of Si-X alloy and infiltration temperature. The analysis is formulated in general terms and is hence applicable to a large variety of Si-C-refractory metal systems of potential interest. Graphite blocks with laser machined channels and porous carbonaceous preforms, made from graphite and silicon carbide (SiC) powders, with varying graphite powder mass fractions and particle sizes, were infiltrated at 1500 °C and 1700 °C by Si-8 at. pct Zr alloy to produce dense Si-Zr-SiC composites. The experiments were performed in a graphite chamber vacuum furnace at 10^{-2} mbar. The most desirable results were obtained for preforms composed of a mixture of graphite and SiC powders, with preforms containing 15-20 % mass fraction of graphite and infiltrated at 1500 °C. The banding of the Zr-rich phase observed in the cross-sections of SiC-C preforms infiltrated by the Si-Zr alloy may help in decoding the reactive infiltration process with binary alloys.

2:30 PM

(ICACC-S4-003-2024) Design of brittle solids for extreme dynamic environments: Challenges and developments

A. Zare*[1]; D. G. Giovanis[3]; K. Ramesh[2]

1. Washington State University, School of Mechanical and Materials Engineering, USA
2. Johns Hopkins University, Mechanical Engineering, USA
3. Johns Hopkins University, Department of Civil and Systems Engineering, USA

Designing structural materials with improved mechanical performance is inherently challenging since macroscopic mechanical properties are dictated by deformation and failure mechanisms that operate across the multiscale hierarchy. For brittle solids under extreme dynamic environments of high stresses and strain rates, the challenge is further exacerbated by the lack of fundamental understanding of mechanisms and relevant structure-property relationships. Over the last decades, the Materials in Extreme Dynamic Environments Collaborative Research Alliance (MEDE CRA) studied boron carbide (B_4C) as a model ceramic material and (using a materials-by-design approach) guided the microstructural design of new B_4C ceramics to achieve improved impact performance. Here, we present a detailed overview of the MEDE findings and investigate the underlying uncertainties that are important to such design efforts, including lack of curated experimental data for model calibration and simplifying assumptions about the microstructure-property relationships. A framework is also introduced to quantify the uncertainties arising from material properties in the impact performance and the role of recent developments in experimental characterization on minimizing such uncertainties is discussed. Findings of this work provide useful guides for developing realistic materials-by-design frameworks for extreme environments.

2:50 PM

(ICACC-S4-004-2024) Thermal and mechanical properties of silicon carbide bonded diamond composites with broad variations of microstructure

B. Matthey*[1]; S. Kunze[1]; M. Herrmann[1]

1. Fraunhofer IKTS, Germany

Diamond-SiC composites are materials that exhibit outstanding properties in a wide range of different applications. This is due to the following properties which in particular only diamond stands out: very high hardness and very high thermal conductivity. This offers the opportunity to develop advanced materials with adapted microstructures. In addition, the technical requirements for application-specific microstructures and materials are becoming increasingly demanding. Moreover, the requirement on tolerances needs appropriate ceramic-based technologies to produce reliable components. Diamond-SiC composites as wear resistant materials can be used for bearings and seals in pumps also in subs sea applications. To prove the suitability of these materials, application related wear tests like sand blasting test were evaluated. Through the modification of the material, with a wide range of microstructures with high diamond grain sizes as well as high diamond contents, different applications can be addressed depending on which specific properties are required. Specifically for this purpose, some structure-properties correlations will be presented. Additionally, for the understanding of the thermal properties synthetic microstructure were generated to simulate the thermal conductivity.

3:30 PM

(ICACC-S4-005-2024) Data-driven Design and Discovery of High Hardness Ceramics for Extreme Environments

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

A high-throughput material-agnostic strategy is proposed to design compositionally complex ceramics with desired attributes for extreme environments by establishing design rules that permit accelerated discovery. First, we focus on well-understood, design spaces (e.g., Si-C-N system), by identifying new stoichiometries and structures. Evolutionary structure searches coupled to density functional theory calculations are applied to predict the ground state and metastable structures. These searches aim to find structures with low energies and maximize the targeted property (e.g., hardness). Hence, the algorithm serves to both, generate and screen data 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 on how chemical additions affect hardness and stability.

3:50 PM

(ICACC-S4-006-2024) A Machine Learned Interatomic Potential for Silicon Carbide

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

Interatomic potentials are surrogate models that govern the physics of atomistic simulations and provide immense computational savings compared to ab initio molecular dynamics (AIMD). However, these reduced computational requirements may come with noticeable reductions in accuracy over ab-initio methods. Our goal is to develop a silicon carbide (SiC) machine-learned interatomic potential (MLIP), intended for simulating extreme environments. We train the MLIP by exploring various MLIP development packages and choosing the highest performing model. Training data was generated via a unified approach, employing a genetic algorithm, in an effort to fully define the potential energy surface and capture diverse atomic arrangements. Through leveraging machine learning methods we aim to model SiC with near ab initio accuracy with computational costs comparable to classical molecular dynamics. MLIP development efforts and results will be discussed.

4:10 PM

(ICACC-S4-007-2024) Influence of Bismuth Oxide as a Sintering Aid on the Densification of Cold Sintering of Zirconia

N. Bhootpur[1]; H. Brouwer[2]; Y. Tang*[1]

1. Delft University of Technology, Aerospace engineering, Netherlands
2. Delft University of Technology, Faculty of Mechanical, Maritime and Materials Engineering (3mE) Materials Science and Engineering (MSE), Netherlands

In the past decades, Zirconia (ZrO_2) has emerged as a promising technical ceramic, both as high temperature structural material and electrolyte for fuel cells, etc. The traditional synthesis of ZrO_2 with spark plasma sintering (SPS) usually requires a sintering temperature as high as 1200 °C. General interest in lowering the sintering temperature to reduce energy consumption and thermal stresses has led to research on two promising routes - cold sintering via temperature-dependent chemical reactivity and sintering aids, which facilitates mass transport and improves densification. Here we combine both by developing a single-step sintering process benefitting from both water vapor through the in-situ conversion of $Zr(OH)_4$ to ZrO_2 and liquid phase Bi_2O_3 as a sintering aid. The resultant ZrO_2 has a relative density above 80% with a sintering temperature as low as 900 °C, significantly higher than that of ZrO_2 without sintering aids, which had a relative density of 54%, both sintered at 50 MPa. The dependence of porosity of sintered samples as a function of sintering pressure (range: 50MPa - 300MPa) and temperature (range 400 °C – 1200 °C) is mapped out as guidance for further material property design. A linear relationship between hardness and relative density was found, with a maximal hardness of 6.6 GPa achieved in samples with 30% porosity.

4:30 PM

(ICACC-S4-008-2024) Refractory Metal-Reinforced SiC-B_4C Composites Fabricated by Hot-Pressing

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1. DEVCOM-Army Research Lab, SURVICE Engineering, USA
2. DEVCOM Army Research Laboratory, USA

Ceramic matrix composites reinforced with metal fibers are a topic of increasing interest due to potential fracture toughness improvements offered by the metal fiber. Previous work has focused on infiltration-based fabrication methods, but little has been done using sintering techniques. For this presentation, ceramic composites were fabricated by hot-pressing ceramic/metal powder mixtures to investigate high-temperature reactions. W or Mo powders (5 vol%) were mixed with SiC and/or B_4C and hot-pressed for 2hrs at 2050°C with an applied pressure of 21 MPa under flowing high-purity Ar. The metal powders were coarse (>50μm), while the ceramic powders were fine (<1μm). The resulting phases were determined using XRD, and microstructures were examined by SEM and EDS, focusing on the metallic particles to determine the extent of the reaction zone. For mixtures with B_4C, significant interdiffusion is expected due to the wide stoichiometric range of B_4C.

4:50 PM

(ICACC-S4-009-2024) Fundamentals of Liquid Silicon Infiltration for Synthesizing SiC Composites

J. LaSalvia*[1]; A. A. DiGiovanni[1]; S. D. Walck[1]; C. Garcia[2]; T. W. Scharf[2];
B. Matthey[3]; S. Kunze[3]; M. Herrmann[3]

1. DEVCOM Army Research Laboratory, USA
2. University of North Texas, Materials Science and Engineering, USA
3. Fraunhofer IKTS, Germany

Liquid silicon infiltration (LSI) is useful in synthesizing near-net, complex-shaped monolithic ceramics and ceramic composites for structural, thermal, and wear applications. LSI involves capillary-driven infiltration of a pyrolyzed ceramic preform with a Si-melt that reacts with carbon sources to form SiC. If successful, the result is a fully dense body consisting of the ceramic with a SiC/residual Si matrix. However, to be successful, the process requires technical consideration of several critical phenomena and practical issues which can result in incomplete infiltration or undesired microstructures and properties if not properly addressed. These include wetting, capillary flow, pyrolysis, reaction rate and enthalpy, as well as practical issues associated with particle packing, carbon sources, Si-melt wicking, and effects of atmosphere composition. In this presentation, several of these critical phenomena/practical issues, along with experimental examples, are reviewed and discussed.

5:10 PM

(ICACC-S4-010-2024) Diamond-SiC Microstructures Evolved from Infiltrated and Simulated Powder Packings

A. A. DiGiovanni*[1]; M. C. Guziewski[1]; J. Sietins[1]

1. DEVCOM Army Research Laboratory, USA

As an engineering material, liquid silicon infiltrated (LSI) composites, are attractive because they can be made into near-net complex-shaped composites suitable for use in structural, thermal, and wear applications. Integration of diamond greatly improves wear and thermal properties. A simple approach to understanding the complex spatiotemporal development of phases in LSI diamond composites was examined using coarse grain diamond powders between 25 and 180 microns. Packed powder assemblies were evaluated using x-ray computed microtomography (XCT) and then infiltrated with a phenolic resin. Post-pyrolyzed assemblies were similarly evaluated via XCT with experimental results digitized and imported in a custom Python-based code capturing fluid infiltration and phase reactions. 3D powder compacts were simulated with LAMMPS discrete element codes and the formation and growth of SiC approximated using a linear model with termination threshold based on experimental limits. Comparisons between experimental observations and modeling are presented and discussed in the context of particle distribution, phase and permeability evolution.

S13 Development & Applications of Adv Ceramics & Composites for Nuclear Fission/ Fusion Energy Sys

SYMPOSIUM 13: Novel ceramics materials for nuclear systems

Room: Ballroom 4
Session Chair: James Wade-Zhu, UKAEA

1:30 PM

(ICACC-S13-001-2024) Will "high entropy" carbide ceramics be enabling materials for nuclear energy applications? (Invited)

B. Cui*[1]; F. Wang[1]; L. Trinh[1]; X. Yan[1]; K. Bawane[2]; Z. Hua[2]; C. Dennett[2]; L. Malakkal[2]; L. He[3]; Y. Lu[1]

1. University of Nebraska–Lincoln, USA
2. Idaho National Laboratory, USA
3. North Carolina State University, USA

The concept of multi-principal component has created promising opportunities for the development of novel "high entropy" ceramics for extreme environments encountered in advanced nuclear energy systems, as it expands the compositional space of ceramic materials with tailored physical and chemical properties within a single-phase solid solution. The first "high entropy" carbide ceramics (HECC), $(Hf_{0.2}Zr_{0.2}Ta_{0.2}Nb_{0.2}Ti_{0.2})C$, with a single phase rock salt structure, was synthesized by spark plasma sintering in 2018. The unique physical properties of some HECC, such as higher hardness, high-temperature strength, lower thermal conductivity, and improved irradiation resistance than the monocarbides, have been observed. These promising properties may be attributed to compositional complexity, atomic-level disorder, lattice distortion, and other fundamental processes related to defect formation and phonon scattering. This presentation focuses on processing, microstructures, and evaluations of the performance of these novel ceramic materials in extreme high-temperature and irradiation environments.

2:00 PM

(ICACC-S13-002-2024) Radiation damage of ion-irradiated high entropy ceramics

K. Wang*[1]

1. Alfred University, USA

Ultrahigh temperature ceramics, such as carbide, boride, and MAX phase, exhibited exceptional high-temperature thermomechanical properties. Also, the emerging high entropy materials have indicated superior radiation tolerance. Therefore, high entropy ceramics (HECs) are supposed to be excellent candidate materials for advanced nuclear reactors. However, the radiation response of HECs is still not well investigated. Herein, we fabricate bulk HECs by mechanical alloying and the subsequent spark plasma sintering (SPS). The HECs are irradiated by heavy ions and helium ions under various irradiation conditions. The radiation hardening is investigated by nanoindentation. The microstructural evolution, including radiation-induced dislocation loops, cavities, segregation, phase stability etc., is interrogated using analytical scanning/transmission electron microscopy (S/TEM). The radiation response of HECs is compared with binary ceramics. The results of this work will provide insights into the radiation behavior of HECs under extreme irradiation environments.

2:20 PM

(ICACC-S13-003-2024) Cermet waste forms for immobilizing the waste from advanced reactors

R. Saini*[1]; S. K. Sundaram[2]; A. Goel[1]

1. Rutgers University, Materials Science and Engineering, USA
2. Alfred University, Inamori School of Engineering, USA

The high-level waste (HWL) expected to be produced at the back end of advanced reactor (AR) fuel cycles shall consist of significant quantities of stainless steel, undissolved solids (UDS), graphite, metal oxides, and halide-based salts. It is difficult, or prohibitively inefficient, to immobilize this diversity of HLW into borosilicate glass, especially at high waste loadings. Consequently, the state-of-the-art for reprocessing HLW from the advanced reactors includes multi-step, complex flowsheets, and multiple waste form concepts with less-than-ideal waste loading, resulting in a significant repository footprint. An alternative waste form concept accommodating multiple waste streams at increased waste loading could reduce the life cycle cost by orders of magnitude. In this context, we aim to develop a simple, scalable route for immobilizing multiple waste streams from any targeted advanced reactor fuel cycle into a durable cermet waste form. Accordingly, the cermets comprising metallic waste (stainless steel or Hastelloy), and reconditioned salt/graphite waste, for example, sodalite, SiC, have been prepared by spark plasma sintering or hot uniaxial pressing and characterized using several state-of-the-art techniques. The results obtained from these investigations shall be presented.

2:40 PM

(ICACC-S13-004-2024) Hydrogen Barrier Coatings to Improve Thermal Stability of Hydride Moderators

R. H. Bohanon*[1]; F. R. Caliari[2]; E. Garcia Granados[2]; S. Sampath[2]; E. P. Luther[1]; S. S. Raiman[3]

1. Los Alamos National Lab, USA
2. Stony Brook University, Center for Thermal Spray Research, USA
3. University of Michigan, USA

Hydrides of zirconium and yttrium show significant promise as neutron moderator materials. Their superior moderating power compared to traditional solid-state moderators such as beryllium or graphite may enable reduced core sizes and greater power densities in advanced thermal spectrum reactors. However, rapid thermal decomposition at elevated temperatures presents a challenge to their implementation. Coating the hydride surface with a ceramic hydrogen permeation barrier is one potential method of diminishing hydrogen loss and reducing thermal decomposition of the hydride. To rapidly identify optimal coating methods, two strategies for surface modification were pursued in parallel: direct application with thermal spray and thermally grown oxide. Specimens of ZrH_x and YH_x were sprayed with a series of common oxide coatings. Selection of spray coating materials was informed by a separate chemical compatibility study. Simultaneously, similar samples of ZrH_x and YH_x were oxidized in air and pure oxygen. The results of both coating methods were examined with optical and electron microscopy. This talk will present the initial results of the compatibility study and both coating 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.

S15 8th International Symposium on Additive Manufacturing and 3-D Printing Technologies

SYMPOSIUM 15: Vat Photopolymerization / Stereolithography I

Room: Coquina H
Session Chair: Fiona Spirrett, Osaka University

1:30 PM

(ICACC-S15-001-2024) Additive Manufacturing of YSZ Ceramics for Solid Oxide Electrolyzer (SOECs) Applications (Invited)

M. Khakzad[1]; M. Minary*[1]

1. University of Texas at Dallas, Mechanical Engineering, Afghanistan

Hydrogen is increasingly recognized as a versatile energy source with applications ranging from powering cars to fulfilling industrial requirements. The challenge is to produce hydrogen in an environmentally friendly and efficient manner. Solid Oxide Electrolysis Cells (SOECs) stand out as a significant solution due to their efficiency and compatibility with green energy. However, the widespread adoption of SOECs is hindered by factors such as high cost and limited durability. The intricate, multi-layered structure of SOECs contributes to their high manufacturing costs. These devices use a variety of materials, including both porous and dense ceramics like Yttria-Stabilized Zirconia (YSZ). YSZ is important for its ionic conductivity and thermal stability, especially since SOECs operate at high temperatures around 750°C. In our research, we focus on utilizing Additive Manufacturing (AM) to produce YSZ ceramics through photopolymerization techniques. This has the potential to reduce costs and improve durability. We will also discuss the thermal shock behavior of additively manufactured YSZ. The results of this work are significant for the development of next-generation SOECs that are both cost-effective and durable.

2:00 PM

(ICACC-S15-002-2024) CeO_2-stabilized zirconia composites: Addressing current challenges in Digital Light Processing

E. Fiume*[1]; B. Coppola[1]; B. Inserra[1]; T. Jean Marc[1]; L. Montanaro[1]; P. Palmero[1]

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

Ceramic stereolithography (CSL) is a fast-growing technology with a transversal deployment through relevant research fields as biomedicine, energy/environment and aerospace. The winning combination of CSL with advanced ceramic materials led to the production of highly-performant components with superior structural/chemical properties and surface quality. Among all, CeO_2-stabilized zirconia (CSZ) stands out for high flexural/compressive strength and fracture toughness in extreme environments, making it suitable as structural component in several fields. However, CSZ processing by CSL presents significant challenges related to CeO_2 absorbance in the UV range and the need for grain size refinement strategies. In the present study, a commercial CSZ powder was used as basic material to obtain composite powders by a novel surface modification route, then dispersed within a commercial liquid monomer to yield printable ceramic slurries. The ball milling process, used to obtain a finely-dispersed powder with a controlled particle size distribution, affected slurries rheology, curing depth and, consequently, green/fired properties. Resulting delamination issues were efficiently managed by targeted thermal treatments on ball-milled powders upstream of the printing. Finally, the effect of secondary phases on zirconia transformability, microstructural and mechanical properties was investigated.

2:20 PM

(ICACC-S15-003-2024) Vat photopolymerization of alumina-toughened zirconia

M. Schwentenwein*[1]; J. Sohl[1]; S. M. Allan[2]

1. Lithoz GmbH, Austria
2. Lithoz America, LLC, USA

Alumina-toughened zirconia (ATZ) is a very promising candidate for different technical applications as well as permanent implants. The advantages of ATZ are its very goodt biocompatibility, chemical inertness, strength, fracture toughness, hardness and abrasion resistance. In this study, an ATZ grade with 20wt% alumina was used for additive manufacturing (AM) through a vat photopolymerization (VPP) approach by employing the so-called lithography-based ceramic manufacturing (LCM) technique. The feasibility of fabricating dense and defect-free components from ATZ with a wall-thickness up to 20 mm was demonstrated, which was unprecetented for additively manufactured technical ceramics by VPP techniques to date. This capability paves the way to enable the fabrication of a multitude of different designs ranging from dental implants to significantly larger constructs such as artificial hip joints or other joints or permanent medical implants to different technical components. This contribution also investigated the influence of hot isostatic pressing of AM-fabricated ATZ parts. Using this advanced sintering approach a bending strength of more than 1100 MPa in a 4-point bending-setup could be achieved in first experiments.

2:40 PM

(ICACC-S15-004-2024) Tailoring the damage tolerance of 3D printed ceramics with lithography-based ceramic manufacturing

J. C. Sänger*[1]; A. Hofer[2]; J. Schlacher[1]; R. Bermejo[3]

1. Montanuniversität Leoben, Austria
2. Montanuniversitaet Leoben, Materials Science, Austria
3. Montanuniversitaet Leoben, Institut fuer Struktur- und Funktionskeramik, Austria

Vat-Polymerization techniques promise the highest accuracy of all additive manufacturing technologies. Ceramics can can benefit from designs beyond their monolithic counterparts in terms of mechanical response. In this work, the possibilities of three different concepts are explored, using Lithography-based Ceramic Manufacturing (LCM) technique, its multi-material capabilities and combining 3D-printing with rapid sintering. Multilayer design of alumina and alumina-zirconia materials are manufactured using the 2K-(LCM) technology and can reaching a biaxial strength of 1GPa. This is associated with the residual stresses upon cooling down from sintering, in comparison to 650 MPa on 3D-printed bulk alumina. The concept was successfully transferred to a three-dimensional design. Textured alumina microstructure combining alignment of high aspect ratio templates with Templated Grain Growth method, to reach biaxial strength of 670 MPa. Compared to 570 MPa measured on equiaxed alumina motivated the combination of additive manufactured textured alumina parts with superior mechanical strength. Rapid Sintering allows to design highly precise grain compositions, but was limited to simple geometries for pressure assisted rapid sintering. Pressure less spark plasma sintering (PL-SPS) enables the combination of the favorable properties achieved with additive manufacturing and rapid sintering.

S17 Advanced Ceramic Materials and Processing for Photonics and Energy

SYMPOSIUM 17: Multi functional materials

Room: Coquina G
Session Chairs: Federico Polo, Ca' Foscari University of Venice; Eva Hemmer, University of Ottawa

1:30 PM

(ICACC-S17-001-2024) Ion-gated transistor and atomic force microscopy studies of metal oxide electrode materials in Li-ion batteries to prolonge their life time (Invited)

C. Santato*[1]; J. Herrera Garza[1]; F. Soavi[2]; L. Neres Chagas Da Silva[1]; L. Pereira Camargo[1]

1. Ecole Polytechnque de Montreal, Canada
2. U Bologna, Italy

Electronic and ionic transport governs lithium-ion battery (LIB) operation. The in operando study of electronic transport in lithium-ion transition metal oxide (LMOx) cathodes at different states of charge enables the evaluation of the state of health of LIBs and the optimization of their performance. We report on electronic transport in LIB cathode materials at different states of charge controlled in operando in ion-gated transistor (IGT) configuration. In this contribution we will discuss lithium titanate- and titanium dioxide-based IGTs in presence of litium ions in the ionic gating medium, focusing on the effect of the potential window probed and potential scanning rate. The device studies will be complemented by nanoscale, in operando studies by force-distance profiling atomic force microscopy measurements, revealing the satructure of electrical double layers at the ionic gating medium/oxide film interfaces.

2:00 PM

(ICACC-S17-002-2024) Photocatalysis and photosensitization using atomically precise metal nanoclusters for solar energy harvesting and conversion (Invited)

N. Pinna*[1]; Y. Wang[1]; Y. Liu[1]

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

Metal nanoclusters (NCs) with atomic precision are a unique family of metal nanomaterials that are readily crystalized into single crystals, representing ideal models to unravel structure-property relationship at atomic level. In the first part of the talk, we demonstrate the differences of working principle between metal NCs and their particle counterparts in photocatalytic system. The metal NC modified TiO_2 catalyst is found to exhibit a fiver times higher performance than TiO_2 modified with metal nanoparticles in the photocatalytic H_2 production reaction· In the second part, we present the strategy to tune the charge transfer pathways of metal NCs sensitized semiconductors in photoelectrochemical system. While metal NCs serve as catalyst for oxidation reactions when loaded on n-type semiconductor, they serve as catalyst for reduction reactions when loaded on p-type semiconductor· In the last part, we will use Au_{25} NC as an example to demonstrate how the protecting ligand and the composition of the metal NC influence the overall performance of a NC/semiconductor system in photocatalytic H_2 production.

2:30 PM

(ICACC-S17-003-2024) Phase engineering vanadium dioxide-based thin film battery electrodes via doping (Invited)

D. Koch[1]; A. Mirzaei[1]; W. Xiang[1]; H. Dai[1]; J. Capdevila[1]; A. Payeur[1]; S. Sun[1]; M. Chaker*[1]

1. INRS, Energie materiaux télécommunications, Canada

Thin film Li-ion batteries (TFLBs) offer high energy density, mechanical flexibility, high safety, and a microscale cell thickness compatible with miniaturized electronic devices for the Internet of Things. Compared to conventional Li-ion batteries, few compounds have been investigated as TFLB electrode materials to date, of which some of the most promising ones are vanadium oxides. Despite the high voltages and appreciable capacities which can be reached with layered vanadium oxide thin film cathodes, materials like vanadium pentoxide commonly suffer from poor cycling stability and a limited cycle life. To mitigate chemo-mechanical deterioration and improve the cycling stability of such vanadium oxide-based thin film electrodes, we investigated non-layered, network-like vanadium dioxide polymorphs. Using elemental doping, we were able to stabilize different phases of VO_2 which differ in their physical and chemical properties and compare their electrochemical capabilities as TFLB electrodes. Besides phase engineering, we also studied how heteroatom doping affects the structural, electronic, and morphological properties of VO_2 phases and influences their reaction with Li. Our results provide valuable insights into the possibility of tailoring the properties of VO_2 via doping to enable the application of this promising material as high-performance TFLB electrode in the future.

3:20 PM

(ICACC-S17-004-2024) Luminescent Theranostic Nanoplatforms (Invited)

F. Vetrone*[1]

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

Luminescent nanomaterials that can be excited, as well as emit, in the near-infrared (NIR) have been investigated for use in a plethora of applications related to human health and biology. They have received considerable attention for theranostic (therapy and diagnostic) platforms where light is used as an external trigger. NIR light can penetrate tissues much better than high-energy light especially when these wavelengths lie within the three biological windows where tissues are optically transparent. At the forefront of NIR excited nanomaterials are rare earth doped nanoparticles (RENPs), which due to their 4f electronic energy states can undergo conventional (Stokes) luminescence and emit in the three NIR biological windows. However, unlike other nanoparticles, they can also undergo a multiphoton process (known as upconversion) where the NIR excitation light is converted to higher energies resulting in anti-Stokes luminescence spanning the UV-visible-NIR regions. Due to the versatility of their optical properties, it now becomes possible to generate high-energy light (UV or blue) in situ to trigger other light activated therapeutic modalities (i.e., drug release) while using the NIR emission for diagnostics (i.e., bioimaging, nanothermometry). Here, we present the synthesis of diverse NIR excited (and emitting) RENPs and demonstrate how their luminescence properties can be exploited for potential use in diverse biomedical applications.

3:50 PM

(ICACC-S17-005-2024) Catalysts of Tomorrow: Green Nanomaterials Paving the Green Energy Path (Invited)

R. Naccache*[1]

1. Concordia University, Chemistry and Biochemistry, Canada

Carbon dots have garnered significant interest with their compact size, versatile and tunable surface chemistry, simple and low-cost synthesis. While some of the reported works on these dots have focused on catalytic applications, their investigation as heterogeneous catalysts for the production of biofuels such as biodiesel remains unexplored. Biodiesel fuel production remains one of the most promising alternatives to non-renewable fossil fuels such as conventional oil and diesel. However, many challenges persist in its production particularly related to the tedious purification processes, high cost of production and unsustainable practices. Thus the need for the development of novel, sustainable and metal free heterogeneous catalysts is required. Here we show that these dots can trans/esterify the conversion of oils to biofuels with sustained catalytic efficiency for at least five reaction cycles. Our second and third-generation catalysts allow for a significant reduction of reaction temperature and operate at ambient pressure without a significant increase to the catalyst loading. Our work now tackles waste and inedible oils in order to reduce the overall cost of the process. This cost-efficient, sustainable and effective heterogeneous catalyst can have positive economic and environmental repercussions that can further drive academic and industrial research in this burgeoning area.

4:20 PM

(ICACC-S17-011-2024) Efficient Solar-Light-Driven Photodegradation of Metronidazole by Nickel Hexacyanoferrate Nanocubes (Invited)

F. Polo*[1]; E. Lushaj[1]; L. Liccardo[1]; M. Bordin[1]; E. Moretti[1]

1. Ca' Foscari University of Venice, Molecular Sciences and Nanosystems, Italy

Environmental pollution, particularly that one regarding the presence of drugs in wastewater, has dramatically increased and is also posing serious issues for the public health. In recent years, the interest of the scientific community has been devoted to address these issues by developing new engineered materials. Here we describe our recent findings concerning the photocatalytic activity of Ni-hexacyanoferrate (Ni-HCF) nanocubes, which has been investigated in the photodegradation of aqueous solutions of metronidazole (MDZ) under simulated solar light. Ni-HCF showed very promising results leading to ~95% yield for the MDZ degradation after 6 hours. The remarkable performance can be attributed to a higher ability in separating the charge carriers and to a lower charge transfer resistance, as demonstrated by electrochemical measurements. These achievements highlight the possibility to combine the performance of an earth-abundant catalysts with a renewable energy source for environmental remediation, meeting the requirements of a sustainable development.

4:50 PM

(ICACC-S17-007-2024) Customizing inorganic nanomaterials synthesis for environmental remediation (Invited)

E. Moretti*[1]; L. Liccardo[1]; M. Bordin[1]

1. Ca' Foscari University of Venice, Department of Molecular Sciences and Nanosystems, Italy

The synthesis of inorganic nanomaterials has been revolutionized by the impact of chemical approaches that allow us to precisely tune the size, composition, morphology, and microstructure of the solid-state materials produced. In particular, tuning the morphological features of a material has emerged as an important strategy to improve its catalytic/photocatalytic performance, and there has been extensive research to develop highly active ceramic systems rationally designed with a controlled shape and an ordered porosity at the nano/microscale for environmental applications. This talk will focus on the importance of tuning the morphological features of a catalyst as a strategy to improve its photoactivity, focusing on how rationally designing inorganic nanomaterials can lead to physico-chemical and optical properties able to enhance the performance in 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. Photodegradation of selected drugs will be presented as successful case history.

5:20 PM

(ICACC-S17-008-2024) 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 the 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 $SrCl_2$ structures scaffolded by clay, cellulose, and graphene networks. The porous $SrCl_2$ structure maintains the macro- and micro-structure, accommodating the volume swing after 20 ammonia absorption–desorption cycles and demonstrating stability for long-term seasonal energy storage.

S18 Ultra-High Temperature Ceramics

SYMPOSIUM 18: Compositionally Complex UHTCs

Room: Coquina A
Session Chairs: William Fahrenholtz, Missouri University of Science & Technology; Stefano Curtarolo, Duke University

1:30 PM

(ICACC-S18-001-2024) Hardness and Elevated Temperature Strength of Compositionally Complex Boride and Carbide Ceramics (Invited)

W. Fahrenholtz*[1]; L. Feng[1]; G. Hilmas[1]

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

Compositionally complex ultra-high temperature carbide and boride ceramics were synthesized by carbothermal and boro-carbothermal reduction processes. Dense ceramics were produced by direct current sintering. Separate series of compositionally complex carbide and boride ceramics were prepared with equal ratios of five transition metal atoms. At room temperature, hardness was found to be higher for both boride and carbide ceramics when Cr, Mo, or W was included in the composition. The compositionally complex carbide ceramics retained their room temperature strength up to at least 1800°C, which is a higher temperature compared to carbides containing only one transition metal. For compositionally complex borides, the strength increased from about 500 MPa at room temperature to a maximum of about 750 MPa at 2000°C. The presentation will discuss reasons for strength retention in these compositionally complext materials.

2:00 PM

(ICACC-S18-002-2024) Mechanical properties and wear characteristics of (Ti-Zr-Nb-Ta-Hf)C/(Ti-Zr-Nb-Ta-Hf)B_2 dual-phase ceramics

A. Naughton Duszova*[1]

1. The Institute of Materials Research, Slovak Academy of Sciences, Slovakia

Fine-grained (Ti-Zr-Nb-Ta-Hf)C/(Ti-Zr-Nb-Ta-Hf)B_2 dual-phase high-entropy boride/carbide ceramic was prepared from powders synthesized via a boro-carbothermal reduction and sintered by spark plasma sintering. The nanohardness of the HEC and HEB grains are very high with values of 37.4 ± 2.3 GPa and 43.3 ± 2.9 GPa, respectively. The Young's modulus of HEC grains with a mean value of 536.5 ± 34.2 GPa is significantly lower in comparison to HEB grains with a mean value of 766 ± 45.7 GPa. Vickers hardness HV1 of the developed HEC/HEB ceramics is very high with a value of 29.4 ± 2.0 GPa which is the highest between the up-to-now reported dual-phase high-entropy ceramics. The developed system shows good indentation fracture resistance with a value of 3.9 ± 0.62 $MPam^{1/2}$. The most significant toughening mechanism is crack branching in large HEC grains with sizes from 1.0 μm to 1.5 μm. The wear characteristics were studied using ball-on-flat technique/dry sliding in air with SiC ball as tribological partner at applied loads 5 N, 25 N and 50 N with total sliding distance and sliding velocity 500 m and 0.1m/s. The friction coefficient values during the test were very similar, 0.57 in case of 5 N and 0.6 for applied load 25 N and 50 N. The dominant wear mechanisms were tribochemical reaction and tribo-layer formation.

2:20 PM

(ICACC-S18-003-2024) Synthesis and Properties of (Hf,Mo,Ti,W,Zr)B_2-(Hf,Mo,Ti,W,Zr)C Dual Phase Ceramics

S. Filipovic*[1]; G. Hilmas[1]; W. Fahrenholtz[1]; N. Obradovic[2]; S. Curtarolo[3]

1. Missouri University of Science and Technology, Materials Science and Engineering, USA
2. Institute of technical sciences of SASA, Materials, Serbia
3. Duke University, Materials Science, Electrical Engineering and Physics, USA

Dual phase high entropy ceramics are attractive due to potential synergetic effects of the constituents on mechanical properties and thermal stability. In this research, dense, dual phase (Hf,Mo,Ti,W,Z) B_2-(Hf.Mo.Ti.W,Zr)C powder was synthesized by varying the contents of the transition metals in the final product. The co-synthesis method was used to obtain boride and carbide constituents by boro/carbothermal reduction of mixtures of oxides and appropriate amounts of carbon black and B_4C. Solid solution formation and densification of the reacted powders were done utilizing two step spark plasma sintering or hot pressing processes. Phase compositions of the produced ceramics were identified by x-ray diffraction while chemical compositions were measured using energy dispersive spectroscopy. The final microstructures showed submicron grains due to pining effect of the two phases. Investigation of the mechanical properties showed increasing in Vickers hardness values s up to 48.6 ± 2.2 GPa for applied load of 0.49N for compositions with optimized elemental contents. Room temperature strength was also measured.

2:40 PM

(ICACC-S18-004-2024) Densification kinetics of high entropy ceramics during spark plasma sintering

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

1. Missouri University of Science & Technology, Materials Science and Engineering, USA
2. Duke University, Materials Science, Electrical Engineering and Physics, USA

High entropy ceramics have been densified by hot pressing and spark plasma sintering, but little information is available for their densification kinetics. In this study, a (Hf,Nb,Ta,Ti,Zr)B_2 high entropy boride, a (Hf,Nb,Ta,Ti,Zr)C high entropy carbide, and a dual phase ceramic containing both high entropy phases were densified by spark plasma sintering. The ram travel was used to determine the densification rates as a function of temperature. The activation energy for intermediate stage densification was determined from Arrhenius plots of densification rate data. X-ray diffraction was used for phase identification and scanning electron microscopy was used to examine the microstructure. Grain size measurements were used to determine the densification mechanism for each ceramic. This presentation will focus on the densification behavior and identification of the densification mechanisms.

3:20 PM

(ICACC-S18-005-2024) Extreme applications of high-entropy carbides (Invited)

S. Curtarolo*[1]

1. Duke University, Materials Science, Electrical Engineering and Physics, USA

Disordered multicomponent systems - occupying the mostly uncharted centers of phase diagrams - have been studied for the last two decades for their potential revolutionary properties. Very resilient compositions can be stabilized by maximizing entropy (configurational and/or vibrational) of (near) equimolar mixtures. The search for new systems is mostly performed with trial-and-error techniques, as effective computational discovery is challenged by the immense number of configurations: the synthesizability of high-entropy ceramics is typically assessed using ideal entropy along with the formation enthalpies from density functional theory, with simplified descriptors or machine learning methods. With respect to vibrations — even if they may have significant impact on phase stability — their contributions are drastically approximated to reduce the high computational cost, or often avoided with the hope of them being negligible, due to the technical difficulties posed in calculating them for disordered systems. In this presentation I will address many of the problems in the discovery of disordered systems, offer some data-based effective solutions, and discuss the avenues opened by the latter, especially for plasmonic applications.

3:50 PM

(ICACC-S18-006-2024) Synthesis and Characterization of High Entropy Carbonitrides (Invited)

L. Backman*[2]; J. Tsai[2]; H. Ryou[1]; E. Patterson[1]; S. Mills[1]; J. Wollmershauser[1]; E. Gorzkowski[1]; J. Maxwell[2]

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

The high temperature (T>1700°C), highly chemically reactive environments encountered during hypersonic flight present unique design challenges for materials scientists. Requirements for these materials include melting temperatures greater than 3000°C, high thermal and dimensional stability, good thermal shock resistance, low reactivity and low coefficients of thermal expansion. Before 2015, less than 15 elements or compounds were considered to have the thermochemical stability to be viable material system candidates for this application and development of materials with coincident mechanical stability (e.g. ductility, toughness) has lagged. The recent advent of the high entropy design paradigm has expanded the composition space for UHTCs significantly and provided unprecedented tunability of mechanical and chemical properties. This is of particular interest for hypersonic vehicle designs requiring both high temperature oxidation resistance as well as maintaining structural and dimensional integrity to maximize aerodynamic performance. This presentation will review design strategies for performance in high temperature environments and discuss material system candidates, with a special focus on carbonitride high entropy ceramics. Experimental work on the synthesis and characterization of high entropy carbonitrides will also be presented.

4:20 PM

(ICACC-S18-007-2024) Thermal and Electrical Properties of Single Phase High Entropy Carbides with Varying Compositions

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

1. 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 and the proposed ability to tailor the thermal properties with compositional changes. 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 phase 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 thermal diffusivity, heat capacity, and thermal conductivity of each HEC were measured using the laser flash method from room temperature to 1800°C. The room temperature thermal conductivity varied compositionally from 4.8 W/mK for ($Hf_{0.2}$,$Mo_{0.2}$,$W_{0.2}$,$Ti_{0.2}$,$Zr_{0.2}$)C to 9.1 W/mK for ($Hf_{0.2}$,$Nb_{0.2}$,$Ta_{0.2}$,$Ti_{0.2}$,$Zr_{0.2}$)C. The electrical resistivity was measured for each composition and the Weidmann Franz law was used to determine the phonon and electron contributions to thermal conductivity.

4:40 PM

(ICACC-S18-008-2024) Ablation Threshold and Temperature Dependent Thermal Conductivity of High Entropy Carbide Thin Films

M. Milich*[1]; K. Quiambao-Tomko[2]; M. D. Hossain[3]; J. Tomko[2]; J. Maria[3]; P. E. Hopkins[2]

1. University of Virginia, Mechanical and Aerospace Engineering, USA
2. University of Virginia, USA
3. Pennsylvania State University, USA

High entropy carbides (HECs) are a promising new class of ultra-high temperature ceramics that could provide novel material solutions for leading edges of hypersonic vehicles, which experience extreme temperatures and thermal gradients. Although the mechanical and thermal properties of HECs have been studied extensively at room temperature, few works have examined HEC properties at high temperatures or considered these materials' responses to thermal shock. In this work, we use time-domain thermoreflectance to measure the thermal conductivity of a five-cation HEC up to 1200 °C. We also measure the temperature dependent thermal conductivity of a HEC deposited with varying CH4 flow rate to investigate the effects of nonstoichiometric carbon content on the thermal properties of HECs. To compare the thermal shock resistance of HECs with a refractory carbide, we conduct pulsed laser ablation measurements to determine the fluence threshold the HECs can withstand before damaging and relate this resulting fluence threshold to theoretical hardness.

S19 Molecular-level Processing and Chemical Engineering of Functional Materials

SYMPOSIUM 19: Functional Carbides & Nitrides

Room: Ballroom 3
Session Chair: Gurpreet Singh, Kansas State University

1:30 PM

(ICACC-S19-001-2024) Investigation of polymer-derived ceramics as thermal and environmental barrier coatings: Oxidation behavior (Invited)

N. Petry[2]; A. S. Ulrich[3]; M. Bik[4]; M. T. Sitarz[4]; R. Riedel[5]; E. Ionescu[5]; M. C. Galetz[2]; M. Lepple*[1]

1. Justus-Liebig-University Giessen, Department of Inorganic and Analytical Chemistry, Germany
2. DECHEMA-Forschungsinstitut, Materials and Corrosion, Germany
3. University of Bayreuth, Metals and Alloys II, Germany
4. AGH University of Science and Technology, Faculty of Materials Science and Ceramics, Poland
5. Technical University Darmstadt, Materials Science, Germany

Polymer-derived ceramics (PDCs) are a promising class of materials for use as thermal (TBCs) and/or environmental barrier coatings (EBCs). T/EBCs are used as a protective layer on structural metallic or ceramic components in harsh conditions. PDCs are suitable for this purpose as they can be chemically modified, e. g. with metalorganic compounds, to form finely dispersed ultra-high temperature ceramic (UHTC) precipitates, forming nanocomposites (PDC-NCs). Furthermore, Si-based PDCs form a silica scale that may be protective against further corrosion in oxidizing atmospheres. In addition, PDCs show a versatile processability as the polymeric precursors are liquid or can be dissolved in organic solvents, which allows the usage of common polymer deposition techniques. This also allows high-temperature stable and/or corrosion stable filler particles to be introduced into the PDC matrix to further optimize the behavior. In this work different protection concepts were investigated, the focus of the study was always the oxidation behavior, depending on the chemical modification and the microstructure. The oxidation behavior of differently modified PDCs such as PDC-NCs with UHTC ((Hf,Ta)C) nanoprecipitates or PDC as sinter additive for UHTC (ZrB_2) were investigated as bulk materials. In addition, the protective effect of a coating of Al-modified PDC on Cr was studied.

2:00 PM

(ICACC-S19-002-2024) Sol gel-based syntheses towards functional carbides (Invited)

N. Kubitza[2]; C. Birkel*[1]

1. Arizona State University, USA
2. Technical University Darmstadt, Eduard-Zintl-Institute, Germany

The synthesis of functional carbides and nitrides, especially when pushing for new chemical compositions, is far from trivial. Materials of interest include layered solids, such as MAX phases, and potential magnetocalorics, e.g. antiperovskite phases. Our group uses diverse preparation techniques to access new versions of these intriguing types of materials, recent examples include Cr_2GaC in the shape of carbonaceous microwires, hollow and full microspheres as well as hitherto unknown carbonitride phases, such as $Cr_2GaC_{1-x}N_x$ and $V_2GaC_{1-x}N_x$. We specialize in non-conventional methods, such as sol-gel chemistry and microwave heating to synthesize the target compounds. In this talk, I will highlight our molecular-level synthesis strategies, especially those based on sol-gel chemistry, that we have developed to access complex carbides and carbonitrides. Examples that I will discuss include MAX phases as well as the antiperovskite Mn_3GaC. We have elucidated the formation mechanism in detail through thermal analyses and ex situ X-ray and neutron diffraction measurements. All materials are characterized by diffraction, microscopy, and element-specific spectroscopy techniques. Another thrust in our group is microwave heating as a non-conventional heating technique and I will show our efforts on the in situ monitoring of non-conventional reactions using Raman spectroscopy.

2:30 PM

(ICACC-S19-003-2024) Defect and Dopant Energy Levels in Next Generation Nitride Phosphors (Invited)

A. Moewes*[1]

1. University of Saskatchewan, Physics & Engineering Physics, Canada

My talk will focus on Eu-doped nitride and oxide semiconductors used in lighting applications. The global energy savings achievable with modern pc-LEDs are very large. This research presents direct measurements of rare earth energy levels, critical to the color, efficiency and performance of LED phosphors. Modern phosphors use the $5d^1$ to $4f^{n+1}$ transition of Eu^{2+} (Ce^{3+}), which is an excited state since Eu^{2+} has no 5d electrons in the ground state. These 5d states are very sensitive to the surrounding crystal and therefore key Eu^{2+} luminescence parameters like wavelength and efficiency can be tailored by the choice of host lattice. We are able to experimentally directly determine the energetic separation of the Eu 5d state and the conduction band. We also directly observe conduction to valence band and 4f to valence band transitions in X-ray excited optical luminescence spectra of a series of cutting-edge phosphors. In a series of new nitride semiconductors, intraband states caused by defects are monitored by luminescence and the measurements are compared to our density functional theory calculations.

S1 Mechanical Behavior and Performance of Ceramics & Composites

SYMPOSIUM 1: Mechanical testing and characterization of ceramic matrix composites (CMCs)

Room: Coquina E
Session Chairs: Marina Ruggles-Wrenn, Air Force Institute of Technology; Dong Liu, University of Bristol

1:30 PM

(ICACC-S1-001-2024) Compressive Strength of CMC Tubular Components in High-Temperature Reactor (HTR) Nuclear Applications: ASTM Draft Standard for Axially-Loaded Tubes (Invited)

M. G. Jenkins*[1]; J. E. Gallego[1]

1. Bothell Engineering and Science Technologies, USA

US DOE is supporting the use of 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 addresses the following experimental issues -- test specimen geometries/preparation, test fixtures, test equipment, interferences, testing modes/procedures, data collection, calculations, reporting requirements, precision/bias.

2:00 PM

(ICACC-S1-002-2024) Inplane Shear Strength Characterization Methods of MI SiC/SiC CMC

Y. Zhou*[1]

1. GE Aviation, Engineering Material System, USA

In this study, inplane shear strength of SiC/SiC CMC have been evaluated by 4 different testing methods: +/-45 degree tensile, isopescu shear, off angle tensile and double notch compression (DNC) tests. Experimental results showed that, for low porosity, high density matrix CMC, only modified DNC can generate correct shear failure mode.

2:20 PM

(ICACC-S1-003-2024) Assessment of a SiC_f/SiC Ceramic Matrix Composite through acoustic emission and vibration analysis

S. Jeffs*[1]; J. Stephen[1]; Z. Quiney[1]; G. Garcia Luna[2]

1. Swansea University, Institute of Structural Materials, United Kingdom
2. Rolls-Royce plc., United Kingdom

SiC_f/SiC Ceramic Matrix Composites (CMCs) offer a considerable weight and operating temperature advantage over existing nickel-based superalloy systems used in the gas turbine engine. Due to the complex structure of CMCs there are a number of damage modes that can occur, such as matrix cracking, interfacial cracking and fibre failure. As such there is a requirement to understand how and when these damage modes take place and what they indicate in respect to the material condition, using techniques that are in-situ and ex-situ. This work investigates damage and condition assessment of a SiC_f/SiC CMC subject to axial mechanical loading through use of Acoustic Emission (AE) and vibration testing. AE is a technique that involves capturing elastic waves emitted during loading, with each event providing a characteristic frequency response. Vibration testing was performed by means of a cantilever beam arrangement, testing between loading steps. For the vibration test analysis, natural frequency values were extracted using the Fast Fourier Transform (FFT) from the response of an accelerometer coupled to the beam. A comparison of these frequencies at the different levels of mechanical loading was then conducted to interpret the damage and condition of the CMC material relative to the non-damaged condition, whilst aiming to relate the observed changes with the AE analysis.

2:40 PM

(ICACC-S1-004-2024) Influence of grinding on the damageable behaviour of SiC/SiC filament wound tubes under cycling tension-compression loadings (Invited)

E. Baranger*[1]; C. Morel[2]; J. L. Lamon[3]; J. Braun[5]; C. Lorrette[4]

1. LMPS (Université Paris-Saclay, CentraleSupélec, ENS Paris-Saclay), France
2. Université de Toulouse, Institut Clément Ader, France
3. CNRS, France
4. Université Paris-Saclay, CEA, Service de Recherche en Matériaux et procédés Avancés, France
5. CEA/DAM, France

SiC/SiC composites are attractive candidates for fuel cladding applications in pressurised light-water nuclear reactors. In this study, fuel cladding tubes were manufactured by filament winding, and the matrix was deposited by CVI (Chemical Vapor Infiltration). After these first steps, a grinding procedure was applied to obtain the suitable dimensions and roughness of tubes. This study aims at evaluating the grinding steps' influence on tubes' mechanical behaviour. Different grades were manufactured, from raw to highly ground external surfaces. Cycled tension-compression tests have been carried out. The different phases of the loading cycles have shown that crack opening/closure during cycles allows the splitting of the contributions of the different crack networks that are either perpendicular or parallel to the loading direction. It has been observed that the internal slip and damage in the tensile direction do not depend on the seal coat thickness but on the core layers, whatever the material grade. Damage in the shear plane strongly depends on the thickness of the inner seal coat layer. It is shown that this layer plays a significant role in carrying the load.

3:30 PM

(ICACC-S1-005-2024) Damage characterization and multiscale modeling of joint failure in oxide/oxide ceramic matrix composites (Invited)

K. Sanghvi[1]; B. Mazurowski[2]; C. A. Duarte[2]; J. Lambros*[1]

1. University of Illinois Urbana-Champaign, Aerospace Engineering, USA
2. University of Illinois at Urbana-Champaign, Civil and Environmental Engineering, USA

Bearing failure in bolted joints is caused by a series of damage events near the bolt region. Here we investigated the various failure modes leading to bearing failure in oxide/oxide ceramic matrix composites and developed a multiscale model for predicting damage evolution. Short beam shear experiments were conducted to evaluate interlaminar shear strength and study delamination behavior. To characterize the numerous simultaneous damage mechanisms leading to bearing failure, a half-hole pin-bearing compressive set-up was developed. In situ and ex situ inspection methods were employed: Stereo-Digital Image Correlation (3D-DIC) for measurement of full-field displacement (in-plane and out-of-plane) and in-plane strain fields during loading, and postmortem optical, SEM microscopy, and X-ray tomography for damage quantification. A three-dimensional finite element model was developed to simulate the experiments using three separate damage models from the literature. The failure model parameters, and the elastic properties, were extracted from separate calibration experiments. The displacement and strain fields obtained from the 3D model show a good correlation with the DIC-generated fields in the elastic regime. The comparative performance of the three models in predicting the observed damage was also evaluated.

4:00 PM

(ICACC-S1-006-2024) Contribution of X-ray tomographic imaging to studies of ceramic-matrix composites damage and ablation

G. L. Vignoles*[1]; G. Couegnat[1]; O. Caty[1]; A. Ebel[1]; F. Rebillat[1]; L. Maille[1]; Y. Lepetitcorps[1]; J. Braun[1]; S. Couthures[1]; T. Malard[1]; M. Fradin[2]; T. Bourdeau[1]

1. University Bordeaux, LCTS - Lab for ThermStructural Composites, France
2. ArianeGroup SAS / LCTS, France

X-ray Computerized Tomography has become an important technique for the characterization of ceramic-matrix composites (CMC). Not only giving access to the 3D internal structural details of these complex materials (woven architecture, pores distributions, etc.) it also allows, thanks to digital volume correlation, to get an insight into their structural evolution under solicitations. In this talk we will describe three different application cases in which X-ray CT in conjunction with DVC analysis and/or specific experimental setups provide information on the damage and/or ablation of CMCs. First, the progressive cracking of self-healing SiC/Si-B-C composites is studied thanks to an in-situ tensile testing apparatus under synchrotron X-ray CT using a home-made DVC code. The crack healing phenomenon and its competition with oxide plug volatilization are also evidenced in another CT-based experiment. Second, oxidation of C/C composites is assessed by CT and DVC in terms of recession rate and affected depth, providing sufficient support for a detailed model of their ablation. Third, the same type of data is obtained during the ablation of a C/C/UHTC composite, observed in a novel in-situ oxyacetylenic torch testing facility placed in a synchrotron X-ray beam.

4:20 PM

(ICACC-S1-007-2024) Electrical Resistance of Non-Oxide CMCs: Health Monitoring and Design Considerations

A. Gupta*[1]; G. N. Morscher[1]

1. University of Akron, Mechanical Engineering, USA

For CMCs where the matrix is electrically conductive DCPD techniques may be utilized to detect composite states such as conductive constituent content (e.g., Si in melt-infiltrated composites) or local defects such as delamination or porosity. The following four ER measurements were performed a. Bulk Resistivity b.Through-thickness c. Axial & d. Surface, along the gauge section of the dogbone specimens & microstructural analysis was done. The bulk resistivity of the samples taken for the study linearly co-relates with the infiltrated Si-content of the matrix even with different percentages & types of porosities present. For through thickness measurements absolute values of the measured potential represent Si-content but are unresponsive for processing defects. The local flow of current for axial type measurements is able to respond to & locate the local & distinct type porosity and is sensitive to "dry-slurry" type defects as well as to rounded porosity but stays inert for homogenously present defects. Surface measurements were able to establish the comparison for local current flow, short-range current flow & long-range current flow. The results institute the potential of electrical resistance techniques as a valuable tool for assessing the properties and integrity of CMCs with conductive matrices, offering insights into both material composition and the presence of specific types of defects.

4:40 PM

(ICACC-S1-008-2024) Matrix Cracking Onset Analysis and Pattern Recognition of Acoustic Emission Signals to Characterise Damage Modes in SiC/SiC Ceramic Matrix Composites

Z. Quiney*[1]; L. Gale[2]; S. Pattison[2]; G. Garcia Luna[2]; C. Newton[1]; M. R. Bache[1]; S. Jeffs[1]

1. Swansea University, Institute of Structural Materials, United Kingdom
2. Rolls-Royce Plc., Materials Engineering, United Kingdom

In the context of high-performance gas turbine engines, ceramic matrix composites (CMCs) offer advantageous material and mechanical properties. Acoustic emission has been successfully used in laboratory experiments on CMCs to better understand the damage mechanisms that accumulate under mechanical and thermal stresses. This study into SiC$_f$/BN/SiC CMCs first set out to investigate the "matrix cracking AE onset stress" under monotonic tension across a range of temperatures up to 1100°C. Results of three measurement approaches were in good agreement with existing literature and showed little variation with temperature. Unsupervised pattern recognition was subsequently applied in efforts to link different AE signal characteristics to damage mechanisms within the CMC. Clusters of waveforms appearing to correspond to matrix cracking and interfacial debonding/sliding were identified at room temperature and continued to be recognised after the introduction of waveguides at high temperature. Further tests were conducted using specimens representing the CMC constituent elements: single-phase RBSC bars, fibre bundles and mini-composites, in attempts to validate signals associated with specific damage modes. Further work aims to investigate whether changes in cluster activity are linked to changes in material chemistry under thermal exposure.

5:00 PM

(ICACC-S1-009-2024) Characterization of stress-induced solid particle erosion behavior of oxide/oxide CMCs in a combustion environment

F. Mirza*[1]; G. N. Morscher[1]

1. University of Akron, Mechanical Engineering, USA

This study investigates solid particle erosion behavior of an oxide/oxide ceramic matrix composite in two distinct phases. The first part of the experiment examines the effect of elevated temperature erosion on CMCs' and how erosion affects its remaining strength, experiment was conducted at 800°C and 1200°C with varying velocities 200m/s and 350m/s respectively. Mass loss, erosion rate and microscopy of erosion crater per impact was conducted to evaluate the extent of damage incurred. Additionally, post-erosion room temperature tension tests were performed to determine retained strength of CMCs. The results obtained from the first section of the study served as a baseline for the second part of the experiment where the relationship between high-temperature solid particle erosion and the fatigue life of composite were further investigated. The focus of the second part was to assess the impact of erosion craters on fatigue behavior of Oxide/Oxide CMCs in an identical erosive environment. The results show that mass loss and erosion rate increase with increase in erosive condition with velocity being more deleterious. Residual strength tests post erosion shows that stress-induced erosion has minimal impact on the remaining strength of composites. Nevertheless, erosion process itself can significantly weaken the materials.

5:20 PM

(ICACC-S1-010-2024) Damage Tolerance Study in Quasi-Statically Indented Alumina-Based Oxide/Oxide Ceramic Matrix Composites

V. Sodisetty*[1]; A. K. Singh[1]

1. Baylor University, Mechanical Engineering, USA

In this study, alumina-based oxide/oxide ceramic matrix composite laminates with varying degree of quasi-static indentation damage are subjected to edgewise compression till catastrophic failure. The goal of these experiments is to establish the relationship between the nature and extent of pre-existing damage with the residual compressive strength. Pre-existing damage in the laminates exist in the form of delaminations, fiber fracture, matrix cracking and major through-thickness cracks. In addition, they are quantified with the damage metrics of dent depth, dent area and planar delamination area. Digital image correlation is used to monitor strain as well as progression of damage during edgewise compression tests. The residual strength value is correlated to the dominant pre-existing damage mode in the laminates. A higher percentage of strength is retained in laminates without through-thickness transverse cracks in comparison to those with the cracks.

S3 21th Intl Symp on Solid Oxide Cells Materials Science & Technology

SYMPOSIUM 3: System design and demonstration

Room: Ballroom 1-2
Session Chair: Federico Smeacetto, Politecnico di Torino

1:30 PM

(ICACC-S3-001-2024) Overview of U.S. DOE's Reversible Solid Oxide Fuel Cell (R-SOFC) Program (Invited)

P. Burke*[1]

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

The U.S. DOE's Reversible Solid Oxide Fuel Cell (R-SOFC) Program, managed by DOE's National Energy Technology Laboratory (NETL), is focused on addressing technical issues related to the commercialization of SOFC and R-SOFC technologies. This effort encompasses pilot-scale testing projects aimed at validating solutions to these issues. Additionally, the Program is actively developing synergistic solid oxide electrolysis cell (SOEC) technology, which offers a potentially attractive option for hydrogen production due to its high efficiency and system flexibility. Aside from the development of standalone SOEC systems, developers are exploring the potential use both SOFCs and SOECs in a single hybrid device in order to produce electricity during periods of high demand and produce hydrogen during off-peak demand times. To successfully advance these technologies from their current state to the point of commercial readiness, the Program's near-term objectives include developing and validating proposed materials for improving the cost, performance, and reliability of R-SOFC systems; conducting RD&D efforts to address degradation at SOEC start-up and enabling technologies for the dynamic operation of SOEC/SOFC systems; and making progress towards achieving low-cost, high-efficiency power generation and hydrogen production.

2:00 PM

(ICACC-S3-002-2024) Status and challenges of Solid Oxide Cells in fuel cell, electrolysis and reversible operation (Invited)

M. Kusnezoff*[1]; S. Megel[1]; N. Trofimenko[1]; S. Mosch[1]; S. Rothe[1]; V. Sauchuk[1]

1. Fraunhofer IKTS, Germany

Solid Oxide Cells have unique ability to be operated in fuel cell, electrolysis, and reversible mode. The presentation will give an overview of literature data on different types of stacks and cells summarizing their performance and degradation characteristics in different operation modes. Especially, the performance and degradation issues for operation with different gas compositions in fuel/steam compartment at different temperatures will be addressed and conclusions about existing limitations for different stack and cell concepts made. The overview of factors limiting the performance and corresponding degradation mechanisms for performance deterioration for different types of materials used in stacks will be provided. The outline for possible improvement of performance characteristics for different cell and stack concepts will be given.

2:30 PM

(ICACC-S3-003-2024) Solid Oxide Fuel Cells for the commercial sector: Results of the Comsos EU project (Invited)

M. Santarelli*[1]; M. Gandiglio[1]; P. Marocco[1]

1. Politecnico di Torino, Energy, Italy

In the COMSOS EU project, SOFCs are investigated as a possible solution for decentralised energy generation in commercial buildings, with best-in-class efficiency without emitting pollutants into the atmosphere, and fuel flexibility (e.g., hydrogen, biofuels). More than 10 SOFC units (ranging from 9 to 60 kW) have been installed worldwide and the results of their operation (currently more than 34,000 hours) are presented: a very high electrical efficiency at nominal power (> 60%), a wide modulation range (from 40-50 to 100%) in which the efficiency is kept stable, and almost no NOx, CO and particulate matter (PM) emissions to the atmosphere (measured on site). Then, a MILP-based optimization model (making use of the real data from COMSOS demonstration units) is presented to shed light on the role of SOFC systems in the commercial sector. SOFC systems become economically viable when the spark spread (discrepancy between electricity and gas costs) is in the range of 0.05-0.1 €/kWh and the SOFC investment cost is less than about 6000 €/kW. Environmental sustainability of SOFC systems is highly dependent on the electricity carbon intensity and the degree of fuel decarbonization. Results show that even fossil natural gas-fed SOFC has lower CO_2 emissions than the reference scenario (without SOFC) in case of electricity grid intensities higher than 300 gCO_2/kWh.

S5 Next-Generation Bioceramics and Biocomposites

SYMPOSIUM 5: Next Generation Bioceramics and Biocomposites

Room: Coquina B
Session Chair: Katalin Balazsi, Centre for Energy Research HAS

1:30 PM

(ICACC-S5-001-2024) Supportless Ceramic Additive Manufacturing of Calcium Phosphate in a Hydrogel Bath (Invited)

H. Yun*[1]

1. Korea Institute of Materials Science, Republic of Korea

With the development of additive manufacturing (AM) technology, various bioceramic scaffolds that can induce volumetric bone tissue regeneration are being actively developed. Meanwhile, the shapes of bone defects are complex with overhanging structures that always require additional manufacturing of sacrificial support structures. The AM of support structures increases the overall process time and material consumption. Breaks of the additive manufactured ceramic structures may also occur when sacrificial supports are removed from the original structure. To overcome this limitation, our group suggests a novel ceramic AM process in a hydrogel bath by combining the material extrusion technique and hydrolysis reaction of calcium phosphates. A hydrogel bath with proper viscosity can mechanically support the fabricated complex overhanging 3D shapes without the support structure when the calcium phosphate ink is extruded into the bath and can promote the hydrolysis reaction to solidify the additive manufactured 3D structures instead of sintering. This novel process enables the fabrication of complex bone constructs with overhanging structures with reduced overall processing time and material consumption. The fabricated scaffold in the hydrogel showed enhanced osteogenic behaviors. Because the gel bath provides cell-friendly environment, we may also co-print calcium phosphates with cells.

2:00 PM

(ICACC-S5-002-2024) Synthesis and Characterization of 3D Printed PEEK-based Composites for Biomedical Applications (Invited)

A. Thorne[1]; S. Gupta*[1]

1. University of North Dakota, Mechanical Engineering, USA

Poly Ether Ether Ketone, PEEK, is a promising semicrystalline thermoplastic for orthopedic implants. Problem with PEEK however is its biologically inert plus traditional manufacturing processes limit the complexity of the geometry of these implants. To overcome these problems, the implants need to have additives or inorganic fillers to increase its cellular response and interaction. The implants can also be manufactured using an FDM 3D printer to have the ability to increase the geometric complexities of the implants. The course we decided to go with is using the fillers of carbon fiber and glass fibers. We chose this because surface coating treatments depend on physical attachments on surface which can result in abrasion, delamination, and shedding of debris into surrounding tissues, and no research had been done with 3D printing PEEK implants with carbon fibers and glass fibers. In the end this study provides a novel strategy for developing high performance PEEK-based composite orthopedic implants, and have shown promise in improving the manufacturing time, complexities, and easy of manufacturing orthopedic implants.

2:30 PM

(ICACC-S5-003-2024) Development and characterization of TPMS hydroxyapatite scaffolds

R. Gabrieli*[1]; A. Schiavi[2]; M. Schwentenwein[3]; E. Vernè[1]; F. Baino[1]; L. D'Andrea[4]; P. Vena[4]

1. Politecnico di Torino, Applied Science and Technology (DISAT), Italy
2. National Institute of Metrological Research (INRiM), Italy
3. Lithoz GmbH, Austria
4. Politecnico di Milano, Italy

The anatomical and functional loss of bone tissue is one of the most debilitating problems in the world. The application of additive manufacturing technologies and computational methods can lead to the creation of scaffolds with microstructure and mechanical behavior comparable to those of bone, thus being potentially suitable to promote tissue regeneration. The present study aims to tackle this challenge by developing hydroxyapatite scaffolds with triply periodical minimal surfaces (TPMS). Specifically, three types of scaffolds with Diamond, Gyroid and IWP geometry were designed and fabricated using vat photopolymerization. These structures were adopted due to their interconnected geometries with a smooth surface, zero-mean curvature and large surface areas. Microstructural and morphological investigations were carried out, with focus on pore characteristics. Intrinsic permeability obtained by acoustic measurements was assessed to be in the range of 5.8×10^{-10} to 1.4×10^{-9} m^2, which is comparable to human cancellous bone. Young's modulus and compressive strength, experimentally determined from crushing tests, were within 3.1-7.6 GPa and 8-36 MPa, respectively. Overall, the produced scaffolds show promise for bone repair and deserve further investigation (e.g. influence of the different structures on bone cell behaviour).

2:50 PM

(ICACC-S5-004-2024) Synthesis and Robocasting of Hydroxyapettite, tri calcium phosphate and wollastonite based composites

R. Gowtham*[1]; G. Akshay[2]

1. Alagappa College of Technology, Anna Uiversity., Ceramic Technology, India
2. Anna University, Ceramic Technology, India

Title: Synthesis and Robocasting of Hydroxyapettite, tri calcium phosphate and wollastonite based composites. Abstract: Tissue engineering (TE) is crucial for addressing organ regeneration, with a specific focus on bone reconstruction, susceptible to trauma, neoplasms, and aging. Our research centers on innovative biomaterials in bone tissue engineering (BTE), including tricalcium phosphate (TCP), hydroxyapatite (HAp), and wollastonite (WS). HAp synthesis repurposes waste biomaterials: orange peels and Moringa oleifera (drumstick) leaves, enhancing ceramic powders for tissue regeneration. Direct ink writing (DIW) enables 3D structure creation at room temperature. Meticulous heat treatment ensures sintering structural integrity, minimizing cracks. Subsequent evaluations encompass physiochemical, surface, and mechanical properties, guiding in vitro and in vivo testing phases. Our research project, "GREEN PRINT HEALTH," bridges materials science and healthcare, emphasizing sustainability and medical innovation. Collaboration spans academia, medical institutions, and industry. Beyond BTE, our work explores hydroxyapatite nanoparticles (HAp NPs) in cancer therapy and diagnosis. Our vision shapes a greener, healthier future, uniting science and humanity.

S6 Advanced Materials and Technologies for Rechargeable Energy Storage

SYMPOSIUM 6: Electrode/electrolyte interface characterization for lithium batteries

Room: Ballroom 5
Session Chairs: Palani Balaya, National University of Singapore; Valerie Pralong, CNRS ENSICAEN

1:30 PM

(ICACC-S6-001-2024) Probing Buried Interfaces in situ Through Neutron Scattering (Invited)

G. Veith*[1]

1. Oak Ridge National Laboratory, USA

This presentation will detail the application of neutron scattering approaches to study the chemistry and dynamics of buried interfaces that mediate and direct electrode formation processes and transport properties. Through their nature neutrons are exquisitely sensitive to low z materials like H, Li, C, and O. This sensitivity, along with careful experimental design, allows for interfacial studies with depth resolution on the order of 0.5 nm. This presentation will detail the use of neutrons to study the solid electrolyte interphase that mediates silicon electrodes and dynamics of polymer binders during electrode fabrication. The talk will be geared to audience members without prior experience with neutron scattering.

2:00 PM

(ICACC-S6-002-2024) Interfacial chemistry and electrolyte approaches for enabling metal anode batteries (Invited)

J. Popovic-Neuber*[1]

1. University of Stavanger, Norway

Continuous solid electrolyte interphase (SEI) and dendrite growth are some of the crucial issues preventing the commercialization of batteries depending on the implementation of alkali and alkaline earth metal anodes. Understanding of the SEI growth on alkali and alkaline earth metals in contact with liquid electrolytes under open circuit potential conditions important for shelf aging is still in its infancy. The ionic transport and stability of the complex composite SEI material under current is highly dependent on the salt chemistry. Most recent ion transport and morphological studies show that the initial SEI formed in contact with liquid electrolytes on Li/Na/Mg/K is nanoporous. Based on the electrochemical impedance spectroscopy measurements and related equivalent circuit models, I will discuss SEI densification.Finally, I will present a possibility of using gas/solid and atomic layer deposition synthesis pathways for preparation of artificial SEIs for both liquid-based and solid-state batteries. I will discuss the method to circumvent dendrite formation by using electrolytes with high cationic transference number. In soft matter electrolytes this is possible when (i) interfacial effect enables preferential anionic adsorption, (ii) the size and steric effects control the association, (iii) ion transport proceeds through percolating amorphous clusters in polymer-in-salt electrolytes.

2:30 PM

(ICACC-S6-003-2024) Towards High Energy Density Batteries: Ultrathick Electrodes with Architectures by Spark Plasma Sintering and Hard Templating Approach (Invited)

V. Seznec*[1]; P. Rozier[2]

1. Laboratoire LRCS, France
2. Laboratoire CIRIMAT, France

High energy density electrodes could be obtained by altering the electrode architectures. In pursuit of increasing the energy density, some methods suggest fabrication of (ultra)thick electrodes while others concentrate on the production of electrodes with a minimum amount of inactive materials (binder-free, conductive additive-free, integrated current collectors, etc). Owing to a rapid and highly efficient sintering process, Spark Plasma Sintering offers the luxury of obtaining simultaneously binder-free and (ultra)thick electrodes which suffer no structural degradation. In this work, we demonstrated that SPS could be used to synthesize a common Na-ion battery cathode material – $Na_3V_2(PO_4)_2F_3$ – in only 40 min which resembles a phase-pure compound with small particle size showing slightly enhanced electrochemical performance when compared to the solid-state synthesized NVPF. SPS-synthesized NVPF requires no further treatment and could be used directly as an active material component in the production of our thick binder-free electrodes. In this presentation, the detailed study on the correlation between the (ultra)thick electrode architecture (porosity, pore size/shape distribution, tortuosity, etc), synthesis/sintering parameters, nature of precursors, and the electrochemical performance will be discussed (both for Li- and Na-ion batteries).

S8 18th Intl Symp on APMT for Structural & Multifunctional Materials & Systems

SYMPOSIUM 8: Microwave processing, SPS, flash sintering, high pressure assisted sintering I

Room: Coquina F
Session Chair: Hisayuki Suematsu, Nagaoka University of Technology

1:30 PM

(ICACC-S8-001-2024) Oxidation resistant dense silicon carbide ceramics (Invited)

P. Sajgalik*[1]; O. Hanzel[1]; M. Hicak[1]; A. Kovalcikova[3]; Y. Kim[2]

1. Institute of Inorganic Chemistry, Slovak Academy of Sciences, Ceramic Department, Slovakia
2. University of Seoul, Dept. of Materials Science & Engineering, Republic of Korea
3. Institute of Materials Research, Slovak Academy of Scienes, Slovakia

Freeze-granulated silicon carbide powder was densified to the full density without any sintering aids by rapid hot-pressing at the temperature range 1850-1900 °C. This densification temperature is at least 150-200 °C lower compared to the up to now known solid state sintered silicon carbide powders. Static and dynamic oxidation resistance of this way prepared ceramics is excellent. The static oxidation at 1450 °C for 204 h is almost negligible in comparison to the LPS sintered SiC materials. In the dynamic regime the ceramics sustained 1900 °C for 60 s without any visible damage. The dynamic oxidation at 2250 °C for 60 seconds still not damaged the sample completely. It seems that this material is really suitable for ultra-high-temperature applications.

2:00 PM

(ICACC-S8-002-2024) Advanced processing techniques for B_4C-TiB_2-Based Materials: Sintering with and without pressure

S. Failla*[1]; S. Taraborelli[1]; D. Sciti[1]

1. Institute of Science, Technology and Sustainability for Ceramics (CNR-ISSMC), Department of Chemical Sciences and Materials Technologies, Italy

B_4C-TiB_2-based materials represent a class of exceptionally hard ceramics widely recognized for their ballistic applications. The production process plays a critical role in tailoring their properties. This study investigates the influence of sintering techniques, specifically pressure-assisted and pressureless sintering, on the microstructural evolution and mechanical characteristics of material composites with a volume ratio ranging from high in B_4C to high in TiB_2. The high energy milling process was used to reduction the starting ceramic powders and introduce WC impurities as sintering aids. The materials were sintered at temperatures ranging from

1900 °C to 2200 °C. These materials exhibit 96-98% of relative density and remarkable hardness, making them essential in ballistic contexts. Through XRD and SEM characterization, we elucidate the influence of sintering conditions on densification and grain growth. Pressure-assisted sintering accelerates the densification process resulting in refinement of the microstructure and enhanced mechanical properties. In contrast, pressureless sintering, despite the absence of applied pressure and longer dwell time at maximum temperatures, produces competitive outcomes, demonstrating its viability for cost-effective production. This comparative analysis offers valuable insights into optimizing the powder processing for B4C-TiB2-based materials.

2:20 PM

(ICACC-S8-003-2024) Electrical, thermal, and mechanical properties of spatially tailored fiber orientations in 3D printed carbon-carbon composites for EFAS/SPS

J. Rufner*[1]; A. Preston[1]; A. Gorman[1]; A. Matthews[1]

1. Idaho National Lab, Materials Science and Manufacturing, USA

Graphite tooling is the material of choice for electric field assisted sintering (EFAS), a.k.a. spark plasma sintering (SPS). Development of alternative tooling materials such as carbides, metal alloys, and composites are required as EFAS technology is scaled up to industrially relevant sizes where parts larger than 12″ are desired and graphite tooling materials are expensive, difficult to source, and have sub-optimal properties. Alternative tooling materials in the form of continuous fiber 3-dimensionally (3D) printed carbon fiber reinforced carbon. 3D printed carbon-carbon with multiple custom fiber orientations, e.g. uniaxial, quasi-isotropic, and spiral were investigated for their impact toward mechanical, thermal, and electrical properties to the final part. The anisotropic properties were leveraged to design die sets for parts ranging from 20mm – 150mm for use in EFAS furnaces which enabled both greater mechanical strength of tooling, as well as 40-50% energy savings compared to graphite without altering the resultant sintered product. The reduced energy usage also translates into reduced ram temperatures in the EFAS instrument, which ultimately enhances the capabilities of any given EFAS machine, as ram over-temperature is one of the primary limiting factors in operation.

S15 8th International Symposium on Additive Manufacturing and 3-D Printing Technologies

SYMPOSIUM 15: Vat Photopolymerization / Stereolithography II

Room: Coquina H
Session Chair: Majid Minary, University of Texas at Dallas

3:20 PM

(ICACC-S15-005-2024) Fabrication of complex components for functional and structural applications by ceramic stereolithography (Invited)

F. Spirrett*[1]; A. Oi[1]; S. Kirihara[1]

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

Ceramic stereolithography can be utilised for fabrication of complex geometries for various applications. Design and computational analysis of self-similar fractal geometries was carried out for applications such as fluid transportation, heat transfer, and environmental control. Hilbert curve structures were successfully fabricated out of composite glass-ceramic pastes by the ceramic stereolithography method, producing dense sintered ceramic parts with self-filling geometries. Further exploration of geometrical freedom was carried out to allow terahertz wave control through the introduction of micro-defects in the form of point- or plane- shaped cavities in lattice structures as photonic crystals. Titania microphotonic crystals were fabricated with high relative density through optimisation of stereolithography laser processing parameters and post-process heat treatment. The measured and calculated photonic band gaps were in good agreement, with a perfect photonic band gap of 0.26 to 0.44 THz opened in the sintered titania lattice.

3:50 PM

(ICACC-S15-006-2024) Tracking the effects of thermal post-processing on 3D-printed 94wt% debased alumina via stress mapping and chemical characterization

S. G. Gomez*[1]; D. Cillessen[2]; J. Duay[2]; K. Sadzewicz[2]; E. MacDonald[1]

1. The University of Texas at El Paso, Aerospace & Mechanical Engineering, USA
2. Sandia National Laboratories, USA

Vat photopolymerization (VPP) of ceramics has been highly recognized in the field of novel fabrication practices for numerous applications. Lithography ceramic manufacturing (LCM) is a type of VPP technology that enables high resolution through the utilization of pixels to project micron-sized squares of light to photosensitive ceramic suspensions. High dimensional accuracy green bodies can be achieved through the optimization of printing parameters. In this study, we demonstrate the impact to dimensional performance on both debased 94% alumina and the high purity (99.8%) alumina when exposed to thermal environments. The chemical composition and stresses were tracked for the 3D-printed components using Fourier-transform infrared spectroscopy (FTIR), X-ray fluorescence (XRF), and Raman spectroscopy at various thermal stages. SNL is managed and operated by NTESS under DOE NNSA contract DE-NA0003525.

4:10 PM

(ICACC-S15-007-2024) Dimensional performance of a debased 94% alumina manufactured with Lithography-based Ceramic Manufacturing (LCM)

D. Cillessen*[1]; S. G. Gomez[2]; E. MacDonald[2]

1. Sandia National Laboratories, USA
2. The University of Texas at El Paso, Aerospace & Mechanical Engineering, USA

Lithography-based ceramic manufacturing (LCM) as an enabling technology reduces development time and provides components with material properties suitable for many developmental applications at Sandia National Laboratories. The LCM technology utilizes digital light processing (DLP) in which projected 450 nm light cures a photosensitive resin through a layer-by-layer process. The lithography process with projected light is known for its ability to create a high resolution, dimensionally accurate green body ceramic. Through the manipulation of printing parameters, we study the impact to dimensional performance at each stage of the manufacturing process. The material selected for this study is a SNL developed debased 94% alumina incorporated into a photocurable resin. The dimensional performance of this material was compared to previously developed SNL debased 94% alumina and high purity (99.8%) alumina. It was determined that a contour offset of -20ìm and an exposure energy of 184mJ/cm2 achieved the highest accuracy for the designed component. SNL is managed and operated by NTESS under DOE NNSA contract DE-NA0003525.

4:30 PM

(ICACC-S15-008-2024) Enhancing Piezoresistivity of Polymer Derived SiOC Ceramics with β-Silicon Carbide Nanopowder Reinforcement

M. Rahman*[1]; S. Kim[1]

1. University of Calgary, Mechanical and Manufacturing Engineering, Canada

3D printing technologies have opened up new possibilities for fabricating ceramic materials. Ceramics fabricated from liquid photosensitive resin using 3D printing offer geometric flexibility with high precision while reducing processing complexity. Utilizing a low-cost 3D printer based on masked stereolithography (MSLA) technology, we successfully fabricated a highly piezoresistive SiOC-SiC polymer derived Ceramic Matrix Composite (PDCMC). The polymer derived SiOC ceramic demonstrated piezoresistive properties when pyrolyzed above 1400 °C, attributed to the formation of free carbon within the amorphous matrix. However, despite displaying piezoresistive properties, the high pyrolysis temperature adversely affected the shrinkage, density, and mechanical properties, leading to increased surface cracking and porosity in the 3D-printed SiOC. Introducing β-SiC into the amorphous SiOC matrix resulted in a significant enhancement of piezoresistive properties at lower pyrolysis temperatures (1100 °C). This newly fabricated PDCMC at a lower pyrolysis temperature exhibited Young's modulus and hardness of 60 GPa and 11.3 GPa, respectively. This 3D-printed composite ceramic material displayed a gauge factor of approximately 8987 at ambient temperature, surpassing previously reported values.

4:50 PM

(ICACC-S15-009-2024) Tailoring powder properties for the light based volumetric additive manufacture of Ceramics

J. Guenster*[1]; J. C. Sänger[2]; B. Pauw[1]

1. BAM Federal Institute for Materials Research and Testing, Germany
2. Montan University et Leoben, Austria

Manipulating ceramic powder compacts and ceramic suspensions (slurries) within their volume with light requires a minimum transparency of the materials. Compared to polymers and metals, ceramic materials are unique as they offer a wide electronic band gap and thus a wide optical window of transparency. The optical window typically ranges from below 0.3 μm up to 5 μm wavelength. Hence, to penetrate with light into the volume of a ceramic powder compound, its light scattering properties need to be investigated and tailored. In the present study we introduce the physical background and material development strategies to apply two-photon-polymerization (2PP), and other volumetric methods for the additive manufacture of filigree structures within the volume of ceramic slurries.

5:10 PM

(ICACC-S15-010-2024) Stereolithographic Additive Manufacturing of Solid Electrolytes for Effective Energy Storage

S. Kirihara*[1]; F. Spirrett[1]

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

In stereolithographic additive manufacturing (STL-AM), 2-D cross sections were created through photo polymerization by UV laser drawing on spread resin paste including nanoparticles, and 3-D models were sterically printed by layer lamination. The lithography system has been developed to obtain bulky ceramic components with functional geometries. As the row material of the 3-D printing, nanometer sized metal and ceramic particles were dispersed into acrylic liquid resins at about 60 % in volume fraction. These materials were mixed and deformed to obtain thixotropic slurry. The resin paste was spread on a glass substrate with 50 μm in layer thickness by a mechanically moved knife edge. An ultraviolet laser beam of 355 nm in wavelength was adjusted to 50 μm in variable diameter and scanned on the spread resin surface. Irradiation power was automatically changed for an adequate solidification depth for layer bonding. The composite precursors including nanoparticles were dewaxed and sintered in the air atmosphere. Through computer-aided smart manufacturing, design, and evaluation (Smart MADE), the ceramic components of lithium compounds for energy storage were fabricated as active contributions to Sustainable Development Goals (SDGs).

5:30 PM

(ICACC-S15-011-2024) 2PP-printed technical ceramics from transparent powder based feedstocks

J. C. Sänger*[2]; B. Riechers[1]; B. Pauw[1]; J. Guenster[1]

1. BAM Federal Institute for Materials Research and Testing, Germany
2. Montanuniversität Leoben, Austria

Two-Photon-Polymerization (2PP) promises highest accuracy and utilizing its resolution for technical ceramics opens a new class of materials. But, ceramic slurries for powder processing routes require ceramic particles, which introduce opacity in the feedstock. This is highly contradictory to light-based vat polymerization techniques, especially 2PP. Choosing nano-particles from ceramic-suspensions with a mean size of 5nm reduces the scattering at the particles sites sufficiently to create a mixture suitable for 2PP with a ceramic particle fraction of up to 80wt%. This enables the fabrication of yttria stabilized zirconia (YSZ) parts with a resolution of 500nm, the smallest for ceramic-AM so far. The nanoparticles, due to their higher sintering activity, allow to vary sintering regimes and create stable ceramic architectures already at sintering temperatures of 800°C, compared to 1450°C, the standard temperature for zirconia ceramics. The variation of sintering temperatures results in untypical particle sizes and grain boundaries. Together with the high resolution of 2PP architectures are created with mechanical properties different from monolithic zirconia. 2PP-structures show high mechanical strength even at lower densities and microplasticity at room temperature, depending on the sintering temperature, chosen geometry and building resolution. A ceramic meta-material is created.

S3 21th Intl Symp on Solid Oxide Cells Materials Science & Technology

SYMPOSIUM 3: Electrolysis and applications

Room: Ballroom 1-2
Session Chair: Massimo Santarelli, Politecnico di Torino

3:20 PM

(ICACC-S3-004-2024) Overview of DOE/FECM R-SOFC Technology Development (Invited)

J. Kim*[1]

1. University of Utah, Chemical & Fuels Engineering, USA

Reversible Solid Oxide Fuel Cells (R-SOFCs), as operating in a fuel mode, produce electricity, water and CO_2 using natural gas and up to 100% hydrogen. R-SOFCs can be configured to operate in reverse as an electrolyzer using power and water as inputs to produce hydrogen, with oxygen as a byproduct. This electrolyzer mode turns the R-SOFCs into a Solid Oxide Electrolyzer Cell (SOEC). SOECs essentially function as a SOFC in reverse and optimize the use of these system to reduce overall costs. The carbon dioxide produced from the process with natural gas as a fuel in a fuel cell mode can then be sequestered for storage or can be converted into CO in an electrolyzer mode. R-SOFCs can both store and produce energy with a single system and can contribute to clean energy generation/storage when paired with a renewable energy such as hydrogen (in fuel cell mode) or renewable electricity (in electrolysis mode). Hydrogen created from R-SOFCs is a promising fuel source and can be stored for future use when renewable energy sources are not available. When the grid demands power, the R-SOFC consumes the stored hydrogen to produce electricity. This presentation will provide a comprehensive overview of DOE/FECM R-SOFC program with emphasis on recent work highlighting specific recent accomplishments in the area of clean hydrogen production from R-SOFCs and power generation from small scale SOFCs.

3:50 PM

(ICACC-S3-005-2024) Status of the development of solid oxide fuel and electrolysis technologies in Poland (Invited)

P. Jasinski*[1]

1. Gdansk University of Technology, Department of Functional Materials Engineering, Poland

In the last few years Poland has become a very active player in the European hydrogen technology landscape. One of the technologies that are being intensively developed at several levels is solid oxide cell technology, including extensive basic and applied research as well as successful demonstrations of small/medium scale systems. This presentation will give a current overview of the projects, funding and results in the last years and show the near-term perspective.

4:20 PM

(ICACC-S3-006-2024) Role of Solid Oxide Cell Technology in Energy Landscape (Invited)

S. Elangovan*[1]; J. Hartvigsen[1]; J. Elwell[1]; J. Pike[1]; M. Hollist[1]; T. Hafen[1]; E. Alvarado[1]; D. Larsen[1]

1. OxEon Energy, USA

A solid oxide electrolysis (SOE) stack developed by the OxEon Energy team for the MOXIE (Mars Oxygen In-situ Resource Utilization Experiment) project has successfully operated 16 times aboard the Perseverance Rover on Mars, demonstrating production of rocket propellant grade (>99.6% purity) oxygen by electrolyzing Mars atmosphere carbon dioxide. Since the delivery of the stack for MOXIE, OxEon Energy has concentrated its effort on scaling up the SOE technology for both space missions and terrestrial applications. The development for manned mission scale stacks for future Mars and Lunar missions has provided a strong basis to address the immediate needs of energy and environmental challenges on earth. As solid oxide technology has a broad applicability to address many of the challenges on earth, the research focus ranged from materials development to provide reliability and robustness in operation, long term performance stability; cell design to improve performance; and stack operation to produce hydrogen and oxygen at moderate pressures. Operational conditions evaluated ranged from electrolysis of steam and carbon dioxide separately and together with a downstream conversion of the produced synthesis gas to hydrocarbon fuels. The technology advances have provided a basis for manufacturing scale up leading towards field deployment as the next stage of commercial pathway.

4:50 PM

(ICACC-S3-007-2024) Update on Sunfire activities: Advancing SOEC technology and durability insights (Invited)

H. Moussaoui*[1]; C. Tsai[1]; C. Geipel[1]

1. Sunfire, Stack development, Germany

Sunfire is a global leader in the field of industrial electrolyzers, offering innovative solutions to address a critical challenge in today's energy landscape. Sunfire's electrolyzer portfolio encompasses both alkaline and solid oxide (SOEC) technologies, enabling the transformation of energy-intensive industries, including chemical, fuel, and steel production. This presentation focuses on Sunfire's SOEC technology, which has achieved a groundbreaking milestone through the successful installation of the world's first SOEC system on a multi-megawatt scale (2.6 MW). Operating at a temperature higher than 800°C, Sunfire's electrolyzer harnesses industrial surplus heat to efficiently convert steam and/or carbon dioxide into hydrogen and/or syngas with exceptional efficiency. Compared to alternative electrolysis methods, Sunfire's SOEC technology is one of the market leaders due to its significantly reduced electricity consumption. However, the elevated operating temperature also accelerates stack degradation, a key area of research at Sunfire. This contribution presents novel findings from a comprehensive, long-term durability test involving two 30-cell stacks tested for up to 18,000 hours. The study investigates the evolution of Area Specific Resistance over time, aiming to gain deeper insights into the underlying degradation mechanisms and propose effective mitigation strategies.

5:20 PM

(ICACC-S3-008-2024) Upscaling and commercialization of SOEC for Power-to-X applications

P. Blennow*[1]; T. Heiredal-Clausen[1]; J. Rass-Hansen[1]; M. Hultqvist[1]; P. Moses[1]

1. Topsoe A/S, Denmark

Topsoe's vision is to be recognized as the global leader in carbon emission reduction technologies by 2024. Our solid oxide cell (SOEC) technology is crucial in future Power-to-X plants, with the most energy-efficient electrolysis solution that seamlessly integrates with downstream processes. Topsoe is one of the few companies globally with expertise and technologies ranging from electrolyzers to numerous downstream processes. This unique position allows us to transform renewable electricity, steam, biomass, and waste into zero-emission fuels and chemicals such as green hydrogen, green ammonia, eMethanol, and eFuels for a sustainable future. To increase electrolysis capacity, Topsoe is building an SOEC manufacturing facility in Denmark. The facility, operational in 2025, will produce electrolysis stacks and modules with an annual production capacity of 500 MW, with an option for further expansion. The TSP-2 stack platform is a dedicated SOEC stack designed for steam electrolysis for H_2 production at >3 bar, with the ability to operate at ambient pressure for CO_2 electrolysis to produce CO. The stack has a production capacity of >10 Nm^3 H_2/h and combines in a 12-stack CORE module with a production capacity of >120 Nm^3 H_2/h, contributing to a compact footprint. The presentation will provide an overview of Topsoe's various SOEC activities.

S6 Advanced Materials and Technologies for Rechargeable Energy Storage

SYMPOSIUM 6: Diagnostics and materials characterization for lithium batteries

Room: Ballroom 5
Session Chairs: Chunmei Ban, University of Colorado, Boulder; Olivier Guillon, Forschungszentrum Juelich

3:20 PM

(ICACC-S6-004-2024) Electrochemical sensor printed on the separator for detection of metal ions (Invited)

T. Paljk[1]; V. Bracamonte[2]; T. Syrovy[3]; S. Drvarič Talian[1]; S. Hočevar[1]; R. Dominko*[1]

1. National Institute of Chemistry, Slovenia
2. Universidad Nacional de Córdoba, Argentina
3. University of Pardubice, Czechia

Li-ion cells are prone to different degradation mechanisms. Among them, transition metal dissolution affects the degradation of the cathode and increases polarization in the battery. That can be currently monitored solely with external parameters like voltage, resistance, and temperature. A step forward in controlling as well as understanding the cell degradation would be enabled by built-in sensing systems that allow the collection of different information during battery functioning. The sensing technologies should be designed in a way of monitors the most detrimental process for the battery cell. In this contribution, we show how the advances achieved in the printed electronics fields can be applied to Li-ion batteries. This is achieved by using an electrochemical sensor that reliably detects manganese ions in the early stages of manganese dissolution. The sensor printed on the battery separator is located in a key position inside the cell. The used approach can be extended in numerous ways by changing the electrodes and/or the sensing composite compounds and the geometry. Most importantly, we proved that printed electronics can be used for sensing inside the battery cell without affecting the battery operation itself.

3:50 PM

(ICACC-S6-005-2024) Surface/Grain Boundary Engineering for Garnet Solid-State Electrolyte (Invited)

C. Ban*[1]

1. University of Colorado, Boulder, Mechanical Engineering, USA

All-solid-state batteries have emerged as a promising solution to mitigate the safety concerns associated with liquid electrolytes, thereby reducing the incidence of battery fires and the need for expensive on-board fire mitigation strategies. However, the practical implementation of solid-state electrolytes (SEs) faces challenges, mainly due to the susceptibility of Li metal anodes to form dendrites that breach SEs, resulting in short circuits and cell failure. Unlike existing methodologies, our approach focuses on inducing residual compressive stress at the surface of SEs through innovative surface modification. This presentation delves into the fundamental principles of stress engineering and its implications for the electrochemical behavior of all-solid-state batteries. Furthermore, we explore the interplay between surface and grain boundary science and their profound influence on electrochemistry. Our investigations unveil the remarkable potential of ion-implanted Garnet ceramic SEs, which enable Li symmetric cells to endure stripping and plating processes at room temperature, reaching current densities of up to 2.5 mA/cm^2. By integrating insights from spectroscopic and structural characterizations, this talk emphasizes the pivotal role of surface stress and chemical modifications in governing reversible electrochemical reactions.

4:20 PM

(ICACC-S6-006-2024) Development of Temperature Sensitive Paint and Battery Management System (BMS) for Energy Storage System (ESS) Safety (Invited)

B. L. Armstrong*[1]; C. Kumara[1]; H. Wang[1]

1. Oak Ridge National Lab, Material Science & Technology, USA

Safety and reliability have become the primary issues for the deployment of energy storage systems (ESS) and improving them is one of the long-term goals for the DOE. One of main issues for ESS safety is the difficulty of monitoring the temperature of every cell in a bank of hundreds and thousands of batteries. Even if a temperature sensor is placed at each cell, it may not be placed at the right location. Because battery failure usually starts from a hot spot at a single cell, it presented an impossible task to use temperature sensors effectively. As a result, it is hard to pin-point the source of any accident, especially after a battery fire or explosion. In ESS, Li-ion cells could take minutes or hours to reach the critical thermal runaway temperature of 130-140°C. The development of a temperature sensitive paint that can be applied to a battery package and/or ESS has been developed that can detect early thermal excursions such that measures can be taken before thermal runaway occurs. An overview of this technology will be presented. This research was sponsored by the U.S. Department of Energy (DOE), Office of Electricity (Imre Gyuk, Program Manager). This abstract has been authored by UT-Battelle, LLC, under contract DE-AC05-00OR22725 with the US Department of Energy (DOE).

4:50 PM

(ICACC-S6-007-2024) Using in-situ biasingTEM to directly measure the electrical properties of solid electrolyte interphase in rechargeable batteries

C. Wang*[1]

1. Pacific Northwest National Lab, USA

For better battery performances, solid electrolyte interphase (SEI) layer is expected to possess three ideal characteristics: electrically insulative, ionically conductive, and constant thicknes. These three characteristics are interactively correlated, typically, the thickness of SEI layer is controlled by the electrical properties of SEI layer. However, SEI layers do not seem to behave the ideal characteristics, rather the thickness of the SEI continuously increases during charge-discharge cycling and shelf storage, indicating that the SEI does not behave as an electrical insulator. We use in-situ bias transmission electron microscopy to directly measure the electrical properties of SEI. We discover that the current-voltage characteristics of SEIs resemble certain electrical conductance, rather than electrical insulation as conventionally assumed. We further demonstrate by tailoring the solvation sheath of the electrolyte, the electrical properties of the SEIs can be readily tuned, which inherently correlate to electrochemical properties. The work highlights the significance of electrical properties of the SEI layer and their tuning towards the enhanced performance of an electrochemical cell.

5:10 PM

(ICACC-S6-008-2024) Multi-Scale, Multi-Modal Non-Destructive Defect Characterization in Solid State Batteries

N. Johnson*[1]; Y. Trenikhina[1]; S. Kelly[1]; H. Bale[1]

1. Carl Zeiss Research Microscopy Solutions, USA

The identification, characterization, and prevention of failure modes in modern solid state batteries is a key focus area of materials engineering. The diagnosis or prevention of battery failure has large implications for device lifetime, performance, and safety. The defects that cause device failure are many and varied in their length scale and appearance. Defects can appear at the scale of millimeters, such as cracks or delamination between layers, or at the scale of microns, such as particle inclusions, voids, and secondary interphases. Identification of defects across all length scales is difficult to achieve with a single characterization method or instrument setup. Researchers typically trade off between characterizing batteries at the macroscale or microscale. We demonstrate a deep learning reconstruction model to build X-ray microscopy (XRM) datasets that have a high field of view (millimeters) and a high resolution (~1 um). A model trained on high resolution interior XRM data is used to upscale a large field of view, low resolution measurement. This upscaling reveals defects in the full-field-of-view that were unobservable before due to resolution limits. The presence of the defect is confirmed using scanning electron microscopy. This method shows strong promise in improving defect identification capabilities.

13th Global Young Investigator Forum

13th Global Young Investigator Forum: Microstructure, thermo-mechanical properties

Room: Coquina D
Session Chairs: Dong Liu, University of Bristol; Palani Balaya, National University of Singapore

3:20 PM

(ICACC-GYIF-004-2024) Achieving stable crack growth in ceramics at the nanoscale: Transformation toughening in zirconia (Invited)

O. Gavalda Diaz*[1]; M. Emmanuel[1]; K. Marquardt[2]; E. Saiz[1]; F. Giuliani[3]

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

The low fracture toughness of ceramic materials poses significant challenges for them to be used in structural applications. In many cases this is due to the low energy dissipated ahead of the crack tip in the absence of a large plastic zone. Several ways to enhance the fracture toughness have been proposed over the last years, including modifying the chemistry or microstructure of monolithic ceramics, to promote for example crack deflection or phase transformation ahead of the crack tip. However, these events happen at a micro/nanometre scale and are difficult to observe in real time. We present a new TEM in-situ nanomechanical setup which enables the propagation of stable cracks in brittle materials while observing their microstructures at the micron and nanoscale. This has been done by modifying the traditional Double Cantilever Beam (DCB) geometry used in conventional fracture experiments. We have used the setup to test a variety of structural ceramic materials, including SiC and partially and fully stabilised zirconia (PSZ and FSZ). Our measurements in zirconia show how tetragonal to monoclinic transformation happens in PSZ but not in FSZ. In PSZ, we are able to observe the nucleation and growth of the monoclinic phase ahead of the crack tip and its behaviour upon unloading. We also use the crack opening displacement (COD) to measure fracture toughness values at the nanoscale.

3:50 PM

(ICACC-GYIF-005-2024) Development of Functionally Gradient Ceramic-Metal Interpenetrating Composites for Ballistic Applications (Invited)

J. Alexander*[1]; J. Binner[1]; C. Footer[2]

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

Development of lightweight armour is crucial for improving personnel and vehicle protection, mobility, speed, and efficiency. Owing to their high abrasion resistance, ceramics are ideal candidates for ballistic applications. However, they demonstrate a poor multi-hit potential. This is due to the ceramic strike faces and ductile backing materials used in traditional armour having mechanical impedance mismatches. Because of this, the incoming compressive stress waves will be reflected as tensile stress waves at the interface of the ceramic strike face and can cause catastrophic failure. 3-3 interpenetrating composites (IPCs) have 3-dimensional interpenetrating matrices. Since both phases are interconnecting in all 3 dimensions, they can yield superior properties for stress wave attenuation, and therefore provide resistance to high-energy projectile impacts. Using these as an intermediary layer between the strike face and backing material to reduce the impedance mismatch is an attractive solution. In the current work, alumina foams infilled with molten aluminium-magnesium (Al-10Mg) alloy were assessed, and ballistic targets were produced by bonding the IPC to a monolithic alumina strike face during infiltration in a single process step.

4:20 PM

(ICACC-GYIF-006-2024) In situ Studies of Temperature- and Stress-induced Structure Evolution in Functional Oxides by Time-of-flight Neutron Diffraction (Invited)

Y. Chen*[1]; K. An[1]

1. Oak Ridge National Lab, USA

Non-equilibrium process is employed to realize the synthesis and functionality of materials by controlling dynamic and sometimes anisotropic changes in the structure at grain- and atom-scales. Capturing the key transients of the continuous and dynamic process is needed to probe the synthesis mechanism and to tailor materials properties, but it is hard via many commonly used post examinations. The time-of-flight neutron diffraction with both high flux and high resolution is demonstrated as a powerful tool to enable in situ studies of the oxides and ceramics. It characterizes phase- and lattice-specific structure anisotropic response in real time upon the external stimulus. Neutron diffraction has unique contrast to detect lightweight atoms and neighboring elements and their arrangement in the lattice of the compound. Here, the demonstrations are given with some model oxides and ceramics. On the high-temperature synthesis of energy storage materials, in situ neutron diffraction resolves the reaction thermodynamics, structure ordering and phase transition to figure out the mechanisms of synthesis and structure stabilization. In other applications, it reveals the stress-induced structure evolution and the deformation mechanisms in ceramics, such as anisotropic lattice straining, ferro-domain switching, and displacive phase transformation.

4:50 PM

(ICACC-GYIF-007-2024) Measuring the Wear and Abrasive Resistance of Air Plasma Sprayed Aluminum Oxide for Lunar Exploration

P. C. Latorre-Suarez*[1]; Q. Fouliard[2]; C. Wohl[3]; V. L. Wiesner[3]; S. Raghavan[1]

1. Emby-Riddle Aeronautical University, Aerospace Engineering, USA
2. University of Central Florida, Mechanical and Aerospace Engineering, USA
3. NASA Langley Research Center, USA

Wear-resistant ceramic materials can improve durability for exploration components during long-duration missions to the Moon's surface. Ceramic materials are lightweight, have multifunctional properties, and have high strength and wear resistance. Materials such as aluminum oxide, with high mechanical strength and high hardness, can help to improve the durability of structures used in space exploration. Lunar regolith, especially finer particles traveling at high velocities, can cause wear and abrasive damage to structural components that are requisite to enabling a sustained lunar presence. In the absence of an atmosphere or any form of erosion or fluid motion, regolith particles maintain their high velocity and abrasive properties for significant distances away from generation activities, e.g., lunar landers, etc. Air plasma sprayed (APS) aluminum oxide has demonstrated the potential to protect surfaces, and this study investigates the wear and abrasion resistance of APS aluminum oxide coatings via Taber abrasion experiments. In this work, an abrasive wheel manufactured with lunar regolith simulants with a mean particle size of 90 μm, was utilized to evaluate wear properties of specimens after 400 cycles and up to 5000 cycles.

5:10 PM

(ICACC-GYIF-008-2024) Innovative Small-Scale Investigation of MAX Phase's Single-Crystal Deformation and Fracture Response

M. Dujovic*[1]; M. Radovic[1]; A. Srivastava[2]; T. Ouisse[3]

1. Texas A&M University, Materials Science and Engineering (MSEN), USA
2. Texas A&M University, USA
3. Grenoble INP, France

MAX phases, atomically layered ternary carbides, and nitrides possess a unique set of mechanical properties due to their atomically layered hexagonal crystal structure with strong intralayer and weak interlayer bonding within it. Several attempts have been recently made to characterize these materials' highly anisotropic mechanical response on the single-crystal level using micromechanical testing of small-scale specimens milled from individual grains. Building on these recent works, we carried out a series of unconventional small-scale tests intending to scrutinize their deformation and fracture response. Two distinct approaches were used to elucidate (1) the effects of imposed deformation constraints on the mechanical behavior of the Cr_2AlC single crystal MAX Phase and (2) the effects of crystallographic orientation on the fracture response of the same MAX Phase. The former set of experiments showed that as the rigidness of the imposed constraint increases, MAX Phases tend to deform in a more pseudo-ductile manner. However, the latter set of experiments showed that initiation and propagation of delamination cracks in this material would, counterintuitively, depend on the crystallographic orientation relative to the applied load.

5:30 PM

(ICACC-GYIF-009-2024) Effect of microstructural differences in Alumina on the thermoelectric properties of polymer Composites for Thermal interface materials (TIMs)

P. Ramanujam*[1]; S. Khan[2]; H. Revankar[2]; S. P[2]; A. Acharya[2]; N. Kapuri[2]; S. K. Bal[1]; V. Tathavadkar[3]

1. Hindalco Speciality Alumina, Sales & Marketing, India
2. Hindalco Innovation Center- Alumina, Research & Development, India
3. Hindalco Industries Limited, MCOE-Technical, India

Alumina-reinforced polymer composites are attracting considerable attention due to their superior dielectric performance in conjunction with high heat-dissipating capabilities, suitable for electronic packages with enhanced thermal management systems. In this study, different particle morphologies of high crystalline alumina (HCA) were produced and characterized using XRD, XRF, and BET surface area. SEM was used to study the morphologies of the alumina powders. HCA1 and HCA2 exhibited equiaxed alumina particles whilst HCA3 and HCA4 exhibited platelet shapes. Also, a bimodal alumina was made and subsequently the effect of its high solids loading in the polymer composites. FT-IR and the rheology of the polymer-filler mix were studied to understand the flow dynamics, and the filler orientation using SEM. The resultant composite slabs were used to examine thermal conductivity, diffusivity, and heat capacities. A fluke thermal imaging camera was also used to understand the heat dissipation of the composite slabs and their electrical properties. This paper explains, in detail, the effect of the microstructural properties of alumina and the properties of the reinforced composite.

S13 Development & Applications of Adv Ceramics & Composites for Nuclear Fission/ Fusion Energy Sys

SYMPOSIUM 13: Ceramics and ceramic-based composites in nuclear fusion

Room: Ballroom 4
Session Chair: Bai Cui, University of Nebraska–Lincoln

3:20 PM

(ICACC-S13-005-2024) Progress of development in SiCf/SiC manufacturing technology for SCYLLA blanket (Invited)

T. Sugiyama[1]; C. Baus[1]; S. Ogawa[1]; P. Barron*[1]; A. D'Angio[1]; R. Pearson[1]; K. Mukai[1]; K. Kawasaki[1]; J. Lee[4]; S. Konishi[3]; T. Hinoki[2]

1. Kyoto Fusioneering Ltd. Japan
2. Kyoto University, Japan
3. Kyoto University, Institute of Advanced Energy, Japan
4. Kyoto University, Graduate School of Energy Science, Japan

In fusion energy systems, the choice of structural materials is limited for the temperature range above 500°C in combination with LiPb coolant. However, SiCf/SiC is a promising material for such harsh conditions. Kyoto Fusioneering (KF) and Kyoto University, with a user perspective, have been improving the TRL of SiCf/SiC composites to produce real fusion reactor components that can become commercially available. KF's SCYLLA© (Self-Cooled Yuryo Lithium Lead Advanced) blanket design is based on the development of SiCf/SiC composites. As a first step toward commercialization, a small breeding blanket SiCf/SiC composite module for use with LiPb coolant was designed and successfully fabricated to verify the manufacturing technology. The module consists of a SiCf/SiC box, a baffle to control the LiPb flow, and two tubes with flanges. The first wall side of the box was bonded with tungsten plates to withstand high heat fluxes. The closed box has joints where the SiC fibers cross at right angles to each other. Updates on fabrication, testing at KF's UNITY facility, and future R&D will be reported. In parallel, the structural design of a larger module for UNITY facility is being analyzed using 3D FEM, and the physical properties used as input were obtained from industrially manufactured plates used in the aforementioned module. The validity of the analysis and the challenges faced are reported here.

3:50 PM

(ICACC-S13-006-2024) Advanced ceramics and their contribution towards sustained fusion energy production (Invited)

J. Wade-Zhu*[1]; A. J. Leide[1]; M. T. Rigby-Bell[1]; H. M. Gardner[1]

1. UKAEA, Materials Division, United Kingdom

Commercial fusion reactors will be one of the most extreme environments for materials, operating at high temperatures under high stress while exposed to high energy neutrons and corrosive chemical interactions. The material challenges for sustained fusion energy generation are vast and require the development of novel "fusion-grade" material compositions with carefully engineered microstructures to achieve the required in-service performance and end-of-life waste characteristics. Ceramics will play an essential role in future fusion systems. Current applications include borides, carbides and hydrides as neutron shielding to protect the superconducting magnets that drive the plasma reaction, oxide coatings as permeation barriers to prevent the release of radioactive tritium, and silicon carbide composites as a high-temperature, corrosion-resistant structural material within the breeder blanket. Ceramics also form a core part of "rad-hard" materials, including diamond detectors, silica-based windows/mirrors/optical fibres, and silicon carbide electronic devices. In this presentation, an overview is provided on the current state of ceramics R&D at UKAEA. Key engineering challenges and core knowledge gaps are presented alongside detailed case studies of active projects being conducted at UKAEA seeking solutions and an improved understanding of material responses to simulated fusion environments.

4:20 PM

(ICACC-S13-007-2024) Solid Electrolyte Development for Direct LiT Electrolysis in Molten Li Mixtures (Invited)

B. L. Garcia-Diaz*[1]; C. S. Dandeneau[1]; D. Hitchcock[1]; P. Ganesan[1]; K. Brinkman[2]; R. Rajeev[2]; S. Jadhav[2]

1. Savannah River National Lab, USA
2. Clemson University, Materials Science and Engineering, USA

Liquid metals such as Li and PbLi are potential candidates for tritium breeding blanket configurations in fusion applications. To ensure safe and reliable operation of fusion pilot plant, the steady-state inventory of tritium in the breeding blanket must be kept sufficiently low. In the case of molten Li, the high solubility of LiT in the metal makes tritium extraction challenging. Current state-of-the-art extraction techniques (e.g., Maroni process) require halide salt addition and the use of expensive equipment. Savannah River National Laboratory (SRNL) has developed a method for tritium extraction via direct LiT electrolysis in molten Li mixtures. The technology utilizes solid-state Li-ion conducting $Li_7La_3Zr_2O_{12}$ (LLZO) electrolytes that are stable in molten Li-bearing mixtures. In this work, cubic Ga-doped LLZO was produced via sol-gel, microwave solution combustion synthesis (SCS), and solid-state reaction methods. During the SCS process, crystallization of the LLZO was induced prior to calcination due to the exothermic nature of the redox reaction. A processing technique involving 3D printing followed by reactive sintering was also devised to manufacture shaped solid electrolytes. The durability of the electrolytes upon immersion in molten Li and PbLi was characterized. The electrolytes were successfully employed in direct LiT electrolysis experiments conducted with molten Li mixtures.

4:50 PM

(ICACC-S13-008-2024) Structural composite R&D roadmap for fusion energy (Invited)

Y. Katoh*[1]; T. Koyanagi[1]; L. Snead[2]; S. E. Ferry[3]; H. Gietl[4]

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

Structural composites in certain forms are potential enabling materials for the future fusion power plants. Historically, the use of silicon carbide continuous fiber-reinforced silicon carbide-matrix composites has been assumed for liquid metal and/or helium cooled breeding blanket concepts. More recently, the need for damage-tolerant tungsten composite has been recognized to replace the bulk tungsten (to be) used for plasma-facing components (PFCs) such as the divertor and the first wall armor. Moreover, more refractory composites such as those with ultra-high temperature ceramics may prove desirable for certain PFCs. All these composite materials require significant R&D before the components can be designed with technical confidence. Recently, community of fusion materials science and technology experts started developing R&D roadmaps for fusion materials including the structural composites. This presentation introduces the activity and presents background and progress in the early phase of the structural composite R&D roadmapping for fusion energy. This study was partly supported by the US Department Energy (DOE), Office of Fusion Energy Sciences under contact DEAC05-00OR22725 with ORNL, managed by UT Battelle, LLC.

S19 Molecular-level Processing and Chemical Engineering of Functional Materials

SYMPOSIUM 19: Functional Carbides & Nitrides II

Room: Ballroom 3
Session Chair: Thomas Fischer, University of Cologne

3:20 PM

(ICACC-S19-004-2024) Solution processing of metal carbide composites (Invited)

G. Westin*[1]

1. Uppsala University, Sweden

Metal carbides and composites with various binder phase metals are due to their tunable hardness and toughness of great importance for a wide range of applications such as metal machining, rock excavation, and low friction durable coatings for automotive industry. The properties of the carbides and connected composites with metal binder phases have been constantly improved by design of microstructures and compositions which are reliant of the synthesis techniques. In most cases, powder mixing based techniques are used for compacts and vacuum based techniques for coatings. However, solution based processing of carbides offer greater flexibility in the material compositions and deposition on complex surfaces. Here all solution based routes to Zr/Cr/V carbides and solution based routes to coated carbide powders for improved sinterability are described, as well as their effect on the microstructures and properties. The improved sinterability of the metal coated powders allowed for very homogenous WC and NbC composites with Co and Ni/Ni-Nb alloys to be formed with excellent mechanical properties and possibility to achieve dense compacts with very low contents of binder phase.

3:50 PM

(ICACC-S19-005-2024) Kumada-rearrangement and Yajima-process investigated by AIMD Simulation

P. Kroll*[1]

1. University of Texas, Arlington, USA

The Kumada rearrangement describes the conversion of methylsilanes into carbosilanes. It is fundamental for the Yajima process, which produces silicon carbide-based materials through thermal conversion of polycarbosilanes. Here we present details of quantum-chemical (ab-initio) Molecular Dynamic (aiMD) simulations of hexamethyldisilane in the gas phase at elevated temperatures. The dominant mechanism of producing carbosilanes – the Kumada rearrangement – proceeds via Si–Si bond breaking and silylene production. Subsequently, the silylene reacts again with a silane, forming the Si–C–Si bond. A direct insertion of the methyl group into the Si–Si bond – usually stated in this context – is rare, as it involves highly unfavorable transition states. Our simulations of polymethylsilanes at elevated temperatures address the Yajima-process. In the much denser polymer system, we frequently observe hydrogen abstraction from methyl groups followed by a radical mechanism that yields Si–C–Si bonds. Production of terminal silylene units is common as well. Overall, we gather new insight into reaction paths and products from aiMD Simulations of these fundamental processes.

4:10 PM

(ICACC-S19-006-2024) Improved Oxidation Resistance of HfC-SiCN Ceramic Mini Composites by Using Single Source Liquid Phase Precursors

S. Mujib*[1]; A. Roy[1]; M. Rasheed[1]; B. Walke[1]; S. R. Arunachalam[2]; G. Singh[1]

1. Kansas State University, Mechanical and Nuclear Engineering, USA
2. Spirit AeroSystems Inc., USA

Hafnium carbide (HfC) is an ultra-high temperature ceramic with high melting point, chemical stability, hardness, and wear resistance. However, its low fracture toughness limits its applications in

extreme environments. The introduction of SiC into HfC ceramics can improve their toughness and oxidation resistance. The oxidation of HfC in the presence of SiC leads to the formation of a hafnium-containing silicate ($Hf_xSi_yO_z$) along with hafnia (HfO_2). This compound of silicate and hafnia limits oxygen diffusion better than SiO2 and HfO2 individually. HfO_2 also acts as a supporting structure to hold the $Hf_xSi_yO_z$ in place. In this study, single-source liquid-phase precursors for both HfC and SiCN were used to infiltrate carbon fiber (CF) cloths via a drop-coating process. The infiltrated CFs were pyrolyzed at 800 °C to achieve CF/HfC and CF/HfC-SiCN ceramic mini-composites. The cross-linked precursor to ceramic yield was observed to be as high as 90% when the procedure was carried out in an inert environment. Room-temperature tensile tests showed that the CF/HfC-SiCN sample could reach a tensile strength of ~240 MPa and an elastic modulus of 27 GPa among the composites. The oxidation study of the ceramic mini-composites showed that CF/HfC-SiCN samples were stable up to 1500 °C in ambient air.

4:30 PM

(ICACC-S19-007-2024) Sol-gel derived SiOC(H) particles as inorganic components of hybrid bigels based on polyelectrolyte complexes for HIV prevention (Invited)

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

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

Hybrid gels and bigels containing inorganic drug carriers embedded into a polymeric matrix emerge as suitable option with promising features for the prevention of the sexual transmission of HIV. We report on the preparation of freeze-dried polyelectrolyte bigels containing sol-gel derived mesoporous SiOC(H) particles for vaginal administration of drugs. The antiretroviral drug Tenofovir (TFV) and the poorly soluble Dapivirine (DPV) have been introduced into the hybrid The aim of this work is the development of freeze-dried polyelectrolyte bigels for vaginal administration of drugs. The selection of the temperature of the treatment for the consecution of the SiOC(H) particles allowed loading two drugs with different physicochemical characteristics which are subsequently released to the releasing media (simulated vaginal fluid) because of the presence of the polyelectrolytic complexes in the polymeric matrix. Impedance spectroscopy characterization allows identifying the different wettability of the vaginal films in the presence of the mesoporous SiOC(H) particles as well as the penetration of the fluid to allow the drugs to be released.

5:00 PM

(ICACC-S19-008-2024) Chemically Engineered Functional Nanostructures for Energy and Health Applications (Invited)

S. Mathur*[1]

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

Chemical nanotechnologies have played, in the past few decades a major role in the convergence of life, physical and engineering sciences leading not only to simple collaboration among the disciplines but to a paradigm shift based on true disciplinary integration. The successful synthesis, modification and assembly of nano building units such as nanocrystals and wires of different materials have demonstrated the importance of chemical influence in materials synthesis, and have generated great expectations for the future. Implications of chemistry as an innovation motor are now visible for knowledge leap forward in various sectors such as materials engineering for energy, health and security. Inorganic nanostructures inherit promises for substantial improvements in materials engineering mainly due to improved physical and mechanical properties resulting from the reduction of microstructural features by two to three orders of magnitude. This talk will present how chemically grown nanoparticles, nanowires and nanocomposites of different metal oxides open up new vistas of material properties, which can be transformed into advanced material technologies.

S8 18th Intl Symp on APMT for Structural & Multifunctional Materials & Systems

SYMPOSIUM 8: Microwave processing, SPS, flash sintering, high pressure assisted sintering II

Room: Coquina F
Session Chair: Pavol Sajgalik, Institute of Inorganic Chemistry, Slovak Academy of Sciences

3:20 PM

(ICACC-S8-005-2024) From Ultrafast Sintering with versus without Electric Fields to Controlling Microstructural Evolution with Applied Electric Fields (Invited)

J. Luo*[1]

1. University of California, San Diego, USA

In this talk, I will first review a series of our mechanistic studies of flash sintering [as summarized in a Viewpoint article: Scripta Materialia 146:260 (2018)]. In 2015, we reported that flash sintering generally starts as a thermal runaway [Acta Materialia 94:87 (2015)]. In 2017, we further reported that ultrahigh heating rates, on the order of ~100 K/s, can enable ultrafast sintering with and without applied electric fields [Acta Materialia 125:465 (2017)]. Subsequently, a general ultrafast high-temperature sintering (UHS) method was developed [Science 368:521 (2020)], which was applied, e.g., to sinter a $[(Ti_{0.2}Ta_{0.2}Mo_{0.2}W_{0.2}Zr_{0.2})B_2]$ based composite [Science Advances 8:eabn8241 (2022)]. Although we have demonstrated that ultrafast sintering can be achieved without an electric field, we have observed a spectrum of electric field induced microstructural evolution phenomena, e.g., an electrochemically induced grain boundary disorder-to-order transition in Bi_2O_3-doped ZnO to trigger abnormal grain growth [Nature Communications 12:2374 (2021); Advanced Engineering Materials 2201767 (2023)]. A most recent study created continuously graded microstructures with applied electric fields via locally altering grain boundary complexions [Materials Today, in revision]. Other on-going studies and unpublished results will also be presented and discussed.

3:50 PM

(ICACC-S8-006-2024) Interparticle-necking assisted grain size control and tetragonal phase stabilization of flash-sintered 1.5 mol% yttria-stabilized zirconia

F. Ong*[1]; K. Kawamura[2]; K. Hosoi[2]; B. Feng[1]; K. Matsui[1]; Y. Ikuhara[1]; H. Yoshida[1]

1. The University of Tokyo, Next Generation Zirconia Social Cooperation Program, Japan
2. Inorganic Materials Research Laboratory, Tosoh Corporation, Japan

One way to enhance the toughness of yttria-stabilized tetragonal zirconia is by reducing the concentration of yttria stabilizer. 1.5 mol% yttria-stabilized zirconia (1.5YSZ) has been shown to exhibit much higher toughness than conventional 3YSZ, and it is generally produced by the conventional pressureless sintering at 1300°C or higher for several hours. Flash sintering, a method which densifies a green body under a strong electric field at a relatively low furnace temperature within seconds, was attempted on 1.5YSZ at different furnace temperatures. When current-ramp-rate controlled flash sintering was performed on a green body at 1100°C, the sample achieved relative density above 99% with indentation toughness of about 17 MPa√m. On the other hand, when flash sintered at 900°C, despite the sample shrank and densified, it underwent spontaneous tetragonal-to-monoclinic phase transformation and disintegrated when cooled to room temperature. This was, however, avoided by calcining the green body at a higher temperature for a short time prior to flash sintering at 900°C. It was shown that the low electrical conductivity of green body was mainly caused by the poor interparticle necking rather than the low furnace temperature, where the highly localized Joule heating promoted grain growth and destabilized the tetragonal phase.

4:10 PM

(ICACC-S8-007-2024) Carbon Contamination Prevention During Spark Plasma Sintering of Transparent MgO and YAG

G. Grader*[1]; M. Sakajio[1]; M. Mann-Lahav[1]; G. E. Shter[1]; Z. Shay[2]

1. Technion - Israel Institute of Technology, Chemical Engineering, Israel
2. RAFAEL, Israel

Optically transparent ceramics are essential materials for a variety of optical applications. Spark plasma sintering (SPS) enables the application of very high heating and cooling rates, short dwell durations, and sintering at relatively low temperatures, which limits grain growth during densification which can improve transparency. This study introduces exceptionally highly transparent MgO ceramics via SPS by optimal incorporation of LiF. The effect of LiF content on the microstructural and optical properties is presented with emphasis on its function as a densification aid and an agent for minimizing residual carbon contamination. Fully dense MgO discs, 20 mm in diameter and 2 mm thick, with ~80% in-line transmission at 800 nm and >85% transmission in the infrared range (2–6 μm), are attained. These results demonstrate outstanding transparency only 7% below the theoretical value. In addition, this work strengthens the understanding of LiF action mechanism during sintering as a lubricant and as a gas-phase buffer minimizing inward diffusion of carbonaceous gasses. A second approach to minimize carbon contamination using metallic foils of tantalum and/or molybdenum adjacent to the samples will be discussed as well, and demonstrated on transparent YAG. These findings pave the way for fabrication of large, fully dense transparent MgO and YAG samples with nearly theoretical transparency.

4:30 PM

(ICACC-S8-008-2024) Reactive spark plasma sintering of ceramics with garnet structure for plasma etching applications

C. Stern*[1]; C. Schwab[1]; M. Kindelmann[2]; M. Stamminger[3]; I. Park[1]; M. Bram[4]; O. Guillon[4]

1. Forschungszentrum Juelich, Institute of Energy and Climate Research, Germany
2. Ernst Ruska-Centre for Microscopy and Spectroscopy with Electrons, Germany
3. Heraeus Noblelight GmbH, Germany
4. Forschungszentrum Juelich, Institute IEK-1, Germany

Yttrium-Aluminum-Garnet (YAG) is an advanced structural ceramic that has desirable properties for halogen plasma etching chambers used in semiconductor processing. The trend towards more aggressive etching environments requires new material systems with enhanced plasma resistance. To tackle this issue, there is an increasing interest in understanding the specific erosion mechanisms of etch-resistant ceramics. In this study, we present a novel approach to manufacture highly dense ceramics with garnet structure by means of reactive spark plasma sintering of the respective oxides. Targeting on improved plasma resistance compared to YAG, Y was completely replaced by lanthanides (Lu, Yb, Er) enabling us to clarify the role of these lanthanides in the YAG-type structure on the plasma-material interaction. For characterization, all samples were exposed to fluorine-based etching plasmas ($CF_4/O_2/Ar$) using an inductively coupled plasma (ICP) etch chamber. The resulting topography was characterized by atomic force microscopy (AFM). Under the given conditions, no physical erosion could be detected and the formation of a reaction layer was dominating. The induced chemical gradient has been analyzed by means of SIMS and STEM. Our results reveal the reaction layer is significantly altered if Y is replaced by lanthanides, which provides a route to study mechanisms of the plasma corrosion process.

4:50 PM

(ICACC-S8-009-2024) State of the art capabilities in Hot Isostatic Pressing for Advanced Ceramics

A. Magnusson*[1]; C. Beamer[2]; J. Shipley[1]

1. Quintus Technologies, Business development, Sweden
2. Quintus Technologies, Application Development, USA

Hot Isostatic Pressing, HIP, capabilities has constantly evolved over the years since the technology was introduced in the 60th, to now include features such as high pressure gas cooling and quenching, steered cooling rates and process atmosphere control. These capabilities will enable tailoring of the HIP process for enhancing different densification strategies and process control, opening up for new and innovative material properties design and processing. The intention of this presentation is to offer an insight into modern HIP capabilities and exemplify how these can be used in the manufacturing of high-performance ceramics materials and components.

5:10 PM

(ICACC-S8-010-2024) Effect of Au doping on cuprate superconductor superconductivity under high pressure

Z. Feng*[1]; Y. Noa[1]; T. Do[2]; T. Nakayama[1]; H. Suematsu[1]

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

The doping of impurity atoms at the Cu-sites in the CuO_2 planar sites and charge reservoir layer were very efficient tool for the understanding of intrinsic mechanism of superconductivity in oxide HTSC. Au^{3+} ions have similar ionic radii to Cu^{2+} ions (0.68 Å and 0.57 Å, respectively), so some scientists have tried to introduce gold ions into charge reservoir layer and CuO_2 planes. However, superconductors have been synthesized only in the Au-Ba-Ca-Ln-Cu system (Ln = rare earth) under high pressure and temperature conditions. In the present study, we have successfully synthesized the $(Au_x, Cu_{1-x})Sr_2CaCu_2Oy$ ((Au,Cu)-$1^{(Sr)}$212) single-phase in the (Au,Cu)-Sr-Ca-Cu-O system under high pressure of 5.5 GPa and heating at 1150°C for 3 h. And under high temperature and pressure, Au reacts with the SrO_2 oxidizer to form Au^{3+}. (Au,Cu)-1(Sr)212 exists with two substitutable Cu sites, and in order to analyze the effects produced by the different sites, $Sr_2Ca_2(Cu_{3-x},Au_x)O_y$ ($0^{(Sr)}$223) with no charge reservoir layer was successfully synthesized as a comparison. According to the experiment of increasing doping amount, doping at different sites with increasing content all inhibit superconductivity.

S5 Next-Generation Bioceramics and Biocomposites

SYMPOSIUM 5: Next Generation Bioceramics and Biocomposites

Room: Coquina B
Session Chair: Cristina Balagna, Politecnico di Torino

3:30 PM

(ICACC-S5-005-2024) Biomimetic polymeric scaffolds for 3D in vitro bone tissue models (Invited)

S. Fare'*[1]

1. Politecnico di Milano, Dept Chemistry, Materials and Chemical Engineering, Italy

3D in vitro models have been developed to reproduce mechanical, biochemical, and structural bone microenvironment. In this work, gas-foamed polyether urethane (PU) foams (PUF) and 3D printed thermoplastic PU (3DP-PU) designed with different patterns are proposed as scaffolds for in vitro models of bone tissue. PUFs as metastases model. PUF porosity was suitable to mimic trabecular bone structure. Human adipose-derived stem cells were cultured and differentiated into osteoblasts on the PU foam, as confirmed by Alizarin Red and RT-PCR, offering a bone biomimetic microenvironment to co-culture with bone-derived tumor-initiating cells. Tumor aggregates were observed by e-cadherin staining and SEM; modification in CaP distribution was identified by SEM-EDX and associated with the presence of tumor cells. 3DP-PU as tumor bone model. 3D printed scaffolds have pores with a precise and regular geometry (0-90°, 0-45-90-135°, 0-60-120°). Porosity evidenced values in the porosity range of the trabecular bone. Compressive modulus varied between 2 and 4 MPa, depending on the printed pattern. Biomimetic coating on 3DP-PU promoted cell colonization, compared to the uncoated scaffold. In conclusion, based on the obtained results, scaffolds with different geometries have been successfully produced and are suitable for mimicking the bone tissue, in order to produce a 3D in vitro model useful for bone pathologies research.

4:00 PM

(ICACC-S5-006-2024) Diffusion-Controlled Formation of Biomineral-Hydrogel Composites toward Biomedical Applications

S. Yang*[1]

1. Korea National University of Education, Chemistry Education, Republic of Korea

A biological system employs intricate strategies to produce inorganic-organic composites, such as bones, shells, and teeth, which possess superior mechanical properties to protect and support the body. Taking inspiration from nature's approach, numerous methods have been developed to replicate biogenic composites, primarily utilizing soluble macromolecules and insoluble matrices. However, the regulated diffusion of ions has not received as much attention in the past, despite the fact that mass transfer in living organisms is meticulously controlled, involving both passive diffusion and active transport across cellular walls, membranes, or the extracellular matrix. In our series of studies, we have placed significant emphasis on the slow and controlled diffusion of ions to emulate the synthetic process of biogenic composites. Within this diffusion-controlled system, the rate of ion supply is regulated by the diffusion of ions into hydrogels. This approach not only benefits the regulation of mineralization but also mimics the intimate interaction between organic and inorganic materials. By drawing insights from biomineralization, biomimetic methods can be effectively employed to synthesize biomaterials that closely resemble bioactivity and possess superior mechanical properties.

4:20 PM

(ICACC-S5-007-2024) Effects of different amounts of calcium citrate in bone repairing materials on apatite formation in simulated body fluid test

Y. Wang*[1]; T. Yokoi[2]; M. Shimabukuro[2]; M. Kawashita[2]

1. Graduate School of Medical and Dental Sciences, Tokyo Medical and Dental University, Japan
2. Institute of Biomaterials and Bioengineering, Tokyo Medical and Dental University, Japan

Recent studies suggest that calcium citrate (CC) is a good candidate for bone repairing material. However, citrate ions inhibit both nucleation and crystal growth of hydroxyapatite (HAp). Octacalcium phosphate/gelatin (OCP/Gel) has biodegradability and can be replaced by new bone in previous studies. Since it is expected to explore the synergistic effects of CC combined with OCP/Gel to enhance bone regeneration, in this study, we investigated the HAp formation of different amounts of CC and it combined with OCP/Gel in simulated body fluid (SBF) test. A 0.047 g and 0.0094 g of CC powder and CC/Gel, OCP/Gel, OCP/CC/Gel, and Gel with different sizes, namely 5 mm in thickness, 5 mm in diameter and 5 mm in thickness, 14 mm in diameter were soaked in SBF for 7 days. The samples were characterized by X-ray diffraction and scanning electron microscope. From the results, CC in the high dose prevented the formation of HAp, but HAp formation was observed in samples with low dose of CC. Among all samples, the small-sized OCP/CC/Gel (5 mm in thickness and 5 mm in diameter) showed the highest density of HAp formation after the SBF test. Hence, a low dose of CC not only avoided inhibiting HAp formation in the SBF test but also exhibited enhancing HAp formation by combining with OCP/Gel.

4:40 PM

(ICACC-S5-008-2024) Environmentalfriendly preparation and structural characterization of calcium silicates derived from eggshell and silica gel

M. Houria Kaou[1]; M. Furko[1]; H. B. Rachid[1]; K. Balazsi*[1]; C. Balazsi[1]

1. Centre for Energy Research, Hungary

In present work, the preparation of calcium–silicate-based composites consisting of natural waste from calcium source as eggshell and silica gel from a desiccator as a silicon source both presenting alternative materials for cheap preparation of eco-friendly calcium–silicate bioceramics has been investigated. The effect of the CaO/SiO2 ratio on microstructural properties has also been studied. The pseudowollastonite formation has been observed in the case of 40 wt.% CaO and 60 wt.% SiO2 with lowest porosity and highest density 2.6 g/cm3. In the case of 50 wt.% CaO and 50 wt.% SiO2, the phase transformation from pseudowollastonite to wollastonite was observed. Increasing the calcium content caused higher apparent porosity with 19%. It was shown that the development of novel ceramics from reused waste, eggshell, or silica can be an optimal solution for the low-cost preparation of calcium silicates with potential applications in medicine or cement, food industry.

Tuesday, January 30, 2024

13th Global Young Investigator Forum

13th Global Young Investigator Forum: Ceramics for batteries

Room: Coquina D
Session Chairs: Bai Cui, University of Nebraska–Lincoln; Yuki Nakashima, National Institute of Advanced Industrial Science and Technology (AIST)

8:30 AM

(ICACC-GYIF-010-2024) Fast-chargeable, Safe and Inexpensive Li-ion Batteries (Invited)

M. Law[1]; S. S. Ramakrishnan[1]; K. Morekonda Ganesh Babu[1]; P. Balaya*[1]

1. National University of Singapore, Singapore

Fast charging of Li-ion battery (LIB) in electric vehicles (EVs) would reduce refueling time and accelerate the transition to electric transportation. However, charging at high current can degrade LIB performance, affecting the EV range; life and safety characteristics of the battery pack. For example, LIB using graphite as the anode cannot be fast charged, which otherwise, will result in thermal runaway. Currently, a fast-chargeable battery technology is based on a $Li_4Ti_5O_{12}$ (LTO) anode coupled with a $LiNiMnCoO_2$ (NMC) cathode, where the safety of the battery is assured. However, LTO has a limited capacity (theoretical 170 mAh/g) and is expensive to produce. Anatase TiO_2, as an alternative, is not only inexpensive, but it also has a high theoretical capacity of 335 mAh/g (assuming an uptake of 1 mole of Li ion). In this talk, we will present our efforts on the development of a fast-chargeable, safe and inexpensive LIB technology using mesoporous TiO_2 coupled with nanostructured $LiMnFePO_4$. We will demonstrate that these electrodes consist of micron-sized mesoporous secondary particles, comprising interconnected primary nanoparticles. Such a unique physical morphology of these nanostructured electrode materials contributes to impressive storage properties, rate performances and cycle life as demonstrated using commercial-type cylindrical cells.

9:00 AM

(ICACC-GYIF-011-2024) The Role of Grain Boundaries in the Ion and Electron Transport Properties of Solid Lithium-Ion Electrolytes (Invited)

C. Ban*[1]

1. University of Colorado, Boulder, Paul M Rady Department of Mechanical Engineering, USA

Solid electrolytes have emerged as a promising solution to enable lithium-metal anodes and mitigate the safety concerns associated with liquid electrolytes. The promising solid electrolytes are typically comprised of polycrystalline inorganic materials created through processes such as powder compaction or sintering. During this manufacturing process, numerous grain boundaries form between individual crystalline particles, exerting a substantial influence on the properties of solid electrolytes. It is widely acknowledged that lithium nucleation occurs at the grain boundaries, with subsequent propagation along these boundaries due to the presence of minor interfacial compressive stresses and relatively high electronic conductivity. Here, we explore the electrical properties of solid electrolytes and investigate the impact of grain boundaries on their electrical properties by using three model materials including garnet-type $Li_{6.5}La_3Zr_{1.5}Ta_{0.5}O_{12}$, argyrodite-type Li_6PS_5Cl, and NASICON-type $Li_{1+x+y}Al_x(Ti,Ge)_{2-x}Si_yP_{3-y}O_{12}$. Furthermore, this presentation will discuss our new surface modification, which can effectively prevent dendrite nucleation and propagation within the solid electrolytes. Our results show that the modified garnet electrolytes exhibits exceptional cycling stability over 10,000 hours.

9:30 AM

(ICACC-GYIF-012-2024) Design and Development of Novel Cathode and Solid Electrolyte Materials for All-solid-state Li-ion Batteries Assisted by In Situ Characterizations (Invited)

H. Chen*[1]

1. Georgia Institute of Technology, Mechanical Engineering, USA

All-solid-state Li-ion batteries with solid electrolytes are promising rechargeable batteries expected to replace current Li-ion batteries (LIBs) with liquid electrolytes, as they offer better safety and higher energy density. Their commercialization is majorly limited by the solid electrolyte as none of the existing materials satisfy all the required properties, including high ionic conductivity, low electronic conductivity, wide electrochemical stability window, good chemical stability, etc. Halide-based Li-ion conductors, such as Li_3M_6 (M=In, Y, Sc, etc.; X=halogens) family compounds, offer better overall properties than existing oxide and sulfide electrolytes. Here we report our recent progress in designing novel halide solid electrolytes with ultra-high ionic conductivity (up to 25 mS/cm at room temperature) and in understanding the origin of the high conductivity. Cutting-edge ex situ and in situ synchrotron X-ray and neutron techniques such as diffraction and pair distribution function analysis are used to reveal unprecedented insights of the structure-property relationship in these new materials. Based on the study of electrolytes, novel cathode materials offering high energy density and very low cost are also developed. The novel all-solid-state battery systems are expected to cut the $/Wh cost to ~30% of current LIBs.

S13 Development & Applications of Adv Ceramics & Composites for Nuclear Fission/ Fusion Energy Sys

SYMPOSIUM 13: Graphite and carbon materials for nuclear applications

Room: Ballroom 4
Session Chair: William Chuirazzi, Idaho National Lab

8:30 AM

(ICACC-S13-009-2024) Microstructure and mechanical property correlation in the pyrocarbon buffer layer of TRISO fuel particles (Invited)

Y. Zhang*[1]

1. University of Wisconsin Madison, Department of Nuclear Engineering and Engineering Physics, USA

The tristructural isotropic (TRISO) fuel particle has the potential of substantially improving the safety of current and future nuclear reactors. It contains a uranium-bearing fuel kernel, enclosed sequentially by a pyrocarbon buffer, an inner pyrocarbon (IPyC), a SiC, and an outer pyrocarbon (OPyC) shells, respectively. Understanding the mechanical behavior of the buffer layer is critical for further improving the performance of TRISO particles. This talk focuses on microstructure and property correlation in the buffer, which is made or porous pyrocarbon. FIB-SEM tomography shows that both the average porosity and its fluctuation increase radially, with elongated pores orientated along the circumferential direction preferentially. Informed by the experimental characterization, finite-element-method (FEM) modeling using the BISON code shows that the heterogeneous distribution of porosity dictates the stochastic nature of buffer fracture. Further, combined atomistic simulations and experiments show that the low-density carbon matrix is made of randomly oriented nanosized crystallites, displaying isotropic elastic properties. Under tension and compression, the matrix exhibits super-elasticity, which is mediated by bending and buckling of the crystallites.

9:00 AM

(ICACC-S13-010-2024) In-situ synchrotron x-ray tomography observations of thermal strains in nuclear graphite via digital volume correlation and image analyses

E. Berry*[1]; J. D. Arregui-Mena[2]; C. I. Contescu[2]; P. D. Edmondson[2]; W. Bodel[5]; X. Zhang[5]; V. Kachkanov[6]; P. Mummery[4]; N. C. Gallego[3]; J. B. Spicer[1]

1. Johns Hopkins University, Materials Science and Engineering, USA
2. Oak Ridge National Lab, Materials Science and Technology Division, USA
3. Oak Ridge National Lab, Chemical Sciences Division, USA
4. University of Manchester, Nuclear Materials, United Kingdom
5. University of Manchester, School of Mechanical, Aerospace, and Civil Engineering, United Kingdom
6. Diamond Light Source, United Kingdom

Graphite is being considered as a moderator of fast neutrons and to serve as a structural material within the next-generation graphite-moderated reactors in the United States. During the operation of a graphite-moderated power plant, graphite components are subjected to thermal gradients and neutron irradiation effects that modify the size and shape of pores. To understand the evolution of the microstructure of graphite under thermal gradients, this research combined in situ heating experiments at 500 °C and imaging using synchrotron x-ray computed tomography (XCT). Three graphite grades were selected to understand and compare the effects of different phases in graphite IG-110, PCEA, and NBG-18. The synchrotron x-ray computed tomography data was systematically analyzed using image analysis and digital volume correlation to understand the porosity changes. This research marks a pioneering effort to provide in-situ, systematic characterization of the microstructural evolution of nuclear graphite associated with the effects of thermal expansion at high temperatures. The findings underscore the influence of microstructure and pore connectivity on graphite behavior under high-temperature gradients.

9:20 AM

(ICACC-S13-011-2024) Characterization of the neutron irradiation effects in glassy carbon

J. D. Arregui-Mena*[1]; T. Koyanagi[2]; D. Cullen[2]; M. Zachman[2]; P. D. Edmondson[2]; Y. Katoh[2]

1. Oak Ridge National Lab, Nuclear Materials Science & Technology Group, USA
2. Oak Ridge National Laboratory, USA

Glassy carbon is an allotrope of carbon that has garnered attention for its potential applications in fission and fusion processes due to its remarkable chemical inertness and mechanical properties. Materials like glassy carbon fall into the category of non-graphitizable carbon materials because traditional thermal treatments cannot graphitize them. Despite these characteristics, glassy carbon does display some degree of short-range order that resembles graphitic structures. While the effects of neutron irradiation on graphite have been extensively studied, the impact of neutron-induced defects in non-graphitizable materials has received less attention. This research aims to gain insights into the effects of irradiation on glassy carbon through a combination of multiscale characterization techniques and thermal property measurements. In this research, samples irradiated at 220C, 630C, and 820C were characterized using advanced electron microscopy techniques. This research not only advances our understanding of the effects of irradiation on glassy carbon but also sheds light on similar materials with comparable atomic structures, including non-graphitizable components in nuclear graphite, carbon-carbon composites, pyrolytic carbons, and the carbon interfaces in SiC-SiC composites.

9:40 AM

(ICACC-S13-012-2024) Nitrogen and Chlorine Levels in Modern and Historic Graphite and Their Impact on i-Graphite Waste

L. Snead*[1]; K. Shirvan[2]; D. Sprouster[1]

1. Stony Brook University, USA
2. Massachusetts Institute of Technology, USA

Waste burden of irradiated graphite (i-graphite), is currently >250,000 tons worldwide, having been the moderator-of-choice for a host of early research reactors and power reactors. Currently, many advanced reactors assume graphite use. In the absence of significant fuel cross-contamination from fuel particles ^{14}C,^{36}Cl,^{3}H, and ^{60}Co are isotopes driving waste management approach for i-graphite. As ^{3}H and ^{60}Co will have substantially decayed within decades of reactor shutdown, they are of near-term concern for operation/maintenance, shipping and storage. However, the isotopes of ^{14}C and ^{36}Cl drive the long-term disposal management schemes. Development of economically attractive and environmentally sound i-graphite management strategies are paramount to limit environmental impact of waste i-graphite waste and ultimate acceptability of graphite reactors. In this presentation we discuss sources, levels, and efforts under study to mitigate the issues of ^{14}C and ^{36}Cl in i-graphite as part of an ongoing DOE-NE I-NERI project. As example, as ^{14}C primarily results from neutron capture of native ^{13}C and neutron capture of ^{13}N which can be present in as-processed graphite, or surface adsorbed, we will present a survey of nitrogen levels of current and historic graphite materials, parsing the sources of the ^{13}N and discussing the implications of fabricating lower N graphite materials.

S15 8th International Symposium on Additive Manufacturing and 3-D Printing Technologies

SYMPOSIUM 15: Additive Manufacturing Processing, Characterization, and Applications I

Room: Coquina H
Session Chair: Dariusz Kata, AGH University of Science and Technology

8:30 AM

(ICACC-S15-012-2024) Fractographic observations from a pilot round robin on flexural strength of a photolithography manufactured high-purity alumina (Invited)

G. D. Quinn*[1]; R. Maier[2]

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

A pilot round robin exercise on flexural strength of additively-manufactured high-purity aluminum oxide bend bars was conducted. Six international laboratories prepared bend bars using Lithaloz 350 ceramic slurry and several models of Lithaloz Cerafab printing systems. Bend bars were made in both horizontal and vertical build orientations. Over 150 as-fired bars from the participating laboratories were broken at NIST in accordance with ASTM standard C 1161, 4-point loading, configuration B. Fractographic analysis found the fracture origin in almost every specimen. A surprising variety of new and interesting flaw types were identified and correlated to Weibull strength distribution diagrams. The work was coordinated by NIST in conjunction with ASTM Committee F42, subcommittee F42.05.05 on Ceramics.

9:00 AM

(ICACC-S15-013-2024) Novel Sintering Techniques for Low Temperature Densification of Ceramics

R. Maier*[1]

1. National Institute of Standards and Technology, USA

Within the materials measurement laboratory (MML) at NIST (a metrology lab dedicated to improving materials measurement sciences) is a focus area project in additive manufacturing (AM) with goals devoted to the developement of reference measurements, tools, and procedures for ceramic AM. A hurdle to mass commercialization of ceramic 3D printing is the expertise required to sinter a printed part. Recent advancements in cold-sintering (sintering ceramics to full density at fractions of traditional processing temperatures) are being explored as methods to integrate ceramic AM with more user-friendly and carbon-reducing post-processing steps. In situ test fixtures have been built at NIST and measurements have recently been conducted on cold-sintered materials at synchrotron beamlines. Recent results of in situ X-ray scattering experiments on cold sintering of ZnO and ex situ X-ray tomography experiments will be presented. These experiments offer critical insights into developing in situ measurements of transient phase changes that occur during processing of oxide/binder systems and will be discussed in terms of plans for ongoing NIST ceramic AM research.

9:20 AM

(ICACC-S15-014-2024) Promoting 3D Additive Manufacturing Technology in Japan's Advanced Ceramics Industry

H. Takemura*[1]; K. Kuroki[1]

1. Japan Fine Ceramics Association, Japan

In recent years, 3D additive manufacturing has become a familiar technology for high-mix, low-volume production of value-added products, and 3D printers for resins have penetrated the world at the level of individual household use. On the other hand, 3D additive manufacturing technology for ceramics still faces many challenges and is still in the developmental stage compared to the preceding resin and metal technologies. The Japan Fine Ceramics Association (JFCA) has launched a study group with the aim of promoting the use of 3D printers for manufacturing advanced ceramics. Through these activities, JFCA hopes to promote 3D additive manufacturing technology throughout the ceramics industry by clarifying the similarities and differences with resin and metal, while developing raw material powders and modeling methods, and discovering new applications in the aerospace, medical, energy, and other fields that take advantage of the stable physical properties of ceramics. In 2021, ISO/TC261 (Additive manufacturing) and ASTM F42 (Additive manufacturing technology) established a Joint WG (JG82) with respect to the international standardization of 3D additive manufacturing in the field of advanced ceramics. JFCA will also promote international standardization in the field of 3D additive manufacturing of advanced ceramics.

9:40 AM

(ICACC-S15-015-2024) Sol-gel processes for additive manufacturing of porous and dense components

G. Franchin*[1]; A. Zanini[1]; P. Colombo[1]

1. University of Padova, Industrial Engineering, Italy

Additive Manufacturing with liquid feedstock demonstrates the potential for a facile production of ceramic components: in fact, they possess suitable rheology for material extrusion, while several physical and chemical approaches exist to add photocurable capability for vat photopolymerization and even volumetric AM approaches. The use of liquid precursors aids both fabrication and sintering. Through a sol-gel synthesis, a high versatility in terms of composition and microstructural development can be reached, allowing for the production of structures with controlled (non stochastic) and hierarchical architectures. Examples of dense, multicomponent glass structures as well as nanoarchitected carbide targets for nuclear physics application will be presented as a proof of concept.

S16 Geopolymers Inorganic Polymers and Sustainable Construction Materials

SYMPOSIUM 16: Synthesis, processing, microstructure

Room: Coquina C
Session Chair: Enrico Bernardo, University of Padova

8:30 AM

(ICACC-S16-001-2024) Geopolymer Nanocomposites Reinforced with Carbon-Based Nanomaterials: State-of-the-art and Underlying Mechanisms (Invited)

A. Akono*[1]

1. North Carolina State University, USA

Geopolymers are inorganic polymers that are intrinsically nanostructured. Prior studies have shown that geopolymers are nanogranular with both grain and pore size in the orders of tens of nanometers. The nanostructured character of geopolymers opens a wide range of opportunities for creating multifunctional materials by combining them with other nanomaterials such as carbon-based nanomaterials. For the last seven years, my research group has been investigating the influence of carbon-based nanofillers such as multiwalled carbon nanotubes, carbon nanofibers, or graphene nanoplatelets on the structure and properties of metakaolin-based geopolymers. To this end, we have employed advanced methods such as statistical nanoindentation, microscopic scratch testing, analytical characterization techniques, or high-resolution scanning electron microscopy. I will present our main findings. I will discuss advanced synthesis routes for novel geopolymer nanocomposites. I will discuss basic microstructural enhancement mechanisms promoted by the high specific surface area of carbon-based nanomaterials. Overall, we observed molecular modifications leading to an enhancement in mechanical properties such as elasticity, strength, creep, and fracture toughness. Our results pave the way towards novel nanostructured and multifunctional enhanced-performance geopolymer composites.

9:00 AM

(ICACC-S16-002-2024) Synthesis and characterization of geopolymers using organic bases (Invited)

D. Samuel*[1]; W. M. Kriven[1]

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

It is well-known that geopolymers can be used as forming methods for crystalline alkali aluminosilicate ceramics (nepheline, leucite, and pollucite). It is also possible to replace the alkali cations with another metal or organic cation by ion exchange in order to alter the composition of the end ceramic. However, ion exchange is limited by diffusion in geopolymers, hence it is useful only for small/thin bodies and porous geopolymers. To avoid the need for ion exchange, we worked to synthesize geopolymers using guanidine, tetramethylguanidine, and tetramethylammonium hydroxide rather than alkali hydroxides. We characterized both the successful and unsuccessful products of the organic base compositions and compared them to conventional sodium geopolymers using nuclear magnetic resonance spectroscopy, powder x-ray diffraction, and scanning electron microscopy. Guanidine gives product materials which are very similar to sodium geopolymers while tetramethylguanidine and tetramethylammonium hydroxide do not.

9:30 AM

(ICACC-S16-003-2024) Investigation of Geopolymer Efflorescence Durability Problems; Causes and Possible Solutions (Invited)

P. Mokhtari*[3]; A. Ozer[3]; R. A. Sa Ribeiro[1]; D. Samuel[3]; W. M. Kriven[2]

1. INPA-National Institute for Amazonian Research, Green Building and Engineering Laboratory, Brazil
2. University of Illinois at Urbana-Champaign, USA
3. University of Illinois at Urbana-Champaign, Materials Science and Engineering, USA

Geopolymers are eco-friendly alternatives to traditional cement-based materials, boasting a reduced carbon footprint and impressive mechanical properties. However, the durability of geopolymer composites is threatened by efflorescence. This study aims to understand the causes of geopolymer efflorescence and find potential solutions. Experiments assessed the impact of relative humidity (RH%) and alkali activator solution molarity on efflorescence in geopolymer composites. Samples with sodium (Na) and potassium (K) bases were prepared with varying water glass molarity (11 and 13 mol). The samples were divided into three groups exposed to different average relative humidity ranges: (i) 0 to 70%, (ii) 70 to 90%, and (iii) 90 to 100%. Efflorescence occurrence and stability were observed, and mechanical tests evaluated compressive and flexural strength. Analytical techniques like x-ray diffraction, scanning electron microscopy, and energy dispersive spectroscopy were used to examine efflorescence deposits. The study provides valuable insights to combat efflorescence in geopolymer systems, guiding future research and development in this area.

10:20 AM

(ICACC-S16-004-2024) Thermal transitions in metakaolin-based geopolymer composites reinforced with fine sand particles and basalt or bamboo fibers (Invited)

R. A. Sa Ribeiro*[1]; M. G. Sá Ribeiro[2]; A. Ozer[3]; P. Numkiatsakul[3]; D. Samuel[3]; W. M. Kriven[3]

1. INPA-National Institute for Amazonian Research, Green Building and Engineering Laboratory, Brazil
2. National Institute for Amazonian Research (INPA), Green Building and Engineering Laboratory (LECVerde), Brazil
3. University of Illinois at Urbana-Champaign, Material Science and Engineering, USA

Simultaneous differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) were used for thermal analysis of metakaolin-based geopolymer matrices (MKGP) and composites reinforced with particles and fibers. This study focused on commercial waterglasses to activate MKGP for higher flexural strength and good thermal stability in sustainable construction applications. The following geopolymer matrices and composite designs were devised. Parameters varied included one type of commercial metakaolin; five commercial waterglass formulations; ball-milled sand and fine sand as particulate reinforcements; and basalt and bamboo fiber reinforcements. It can be concluded from TGA-DSC curves that as the water content increases from 11 M to 13 M, surface and bonded water removal occur more drastically while there was a crystallization peak of nepheline at 766°C in the DSC signal for Na-based geopolymers. Basalt does not affect the phase change or weight loss of composites, while bamboo fibers produce degradation peaks and hills due to decarbonization and burn out of lignin, hemicellulose, and cellulose at last by increasing temperatures.

S17 Advanced Ceramic Materials and Processing for Photonics and Energy

SYMPOSIUM 17: Multi-functional materials

Room: Coquina G
Session Chairs: Silvia Gross, University of Padova; Nicola Pinna, Humboldt-Universität zu Berlin

8:30 AM

(ICACC-S17-009-2024) Tuning the Optical Features of Lanthanides via Structural Control at a Nanoparticle Level (Invited)

N. Liu[1]; C. Homann[1]; E. Hemmer*[1]

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

Lanthanide-based nanomaterials are known to emit visible and near-infrared (NIR) light under NIR excitation, so called upconversion. Yet, challenges remain, including low emission intensity and efficiency of small nanoparticles (NPs), and reliable, fast synthesis routes. As material chemists, we tackle these challenges with new designs of Ln-NPs by chemically controlled synthesis and the establishment of structure-property-relationships. Sodium lanthanide fluorides ($NaLnF_4$) are our favorite materials, and we developed a fast and reliable microwave-assisted synthesis approach allowing crystalline phase and size control in the sub 20nm realm. Such control is crucial for the understanding of fundamental structure-property relationships and to optimize their optical properties when aiming for the design of multifunctional probes. For instance, $NaDyF_4$ is gaining interest as alternative MRI contrast agent, while the combination with Ln^{3+} ions might add photoluminescence capabilities. Yet, Dy is known to hamper the bright upconversion emission. Having a fast and reliable synthesis route towards core/multi-shell $NaLnF_4$ NPs on hand, we explored various nanoparticle architectures with the goal to optimize their optical properties, ultimately resulting in the design of multimodal probes.

9:00 AM

(ICACC-S17-010-2024) Scaling-up of Ceramic-based Components for Advanced Applications (Invited)

C. Busa*[1]

1. Technology Innovation institute, Advanced Materials, United Arab Emirates

On the way of the technology translation from laboratory scale to manufacturing, the pilot and scale-up processing hinder the way, and a vast amount of innovation stops there. At TII, we reckon the importance of easing this step, in particular, enabling synthetic avenues for high throughput and competitive production cost. We focus on the development of several materials, with particular attention to ceramics for a variety of applications, including energy storage components, as well as anti-soiling and superhydrophobic coatings. For example, in line with the UAE's targets for the next few years for a drastic reduction in fossil fuel consumption, ceramics hold a cumbersome role in a variety of technologies to reduce the actual energy requirements when embedded into coatings for thermal management. The ceramics development effort is also coupled with studies on crystallinity, purity, as well as hierarchical architectures, coating techniques, and properties assessment in regard to mechanical, thermal, and electrical properties, and a combination thereof. In particular, the light-matter interaction and the related properties are of main interest, in regards to the photonics and plasmonic ones. Furthermore, the processability for additive manufacturing purposes is being tackled, with particular attention to liquid feed formulations.

10:20 AM

(ICACC-S17-013-2024) Solvothermal synthesis of unusual transition metal oxides structures and their water remediation properties (Invited)

M. Epifani*[1]

1. CNR-IMM, Italy

Molybdenum dioxide (MoO_2) is a metallic conductor, recently introduced as efficient water remediation agent, rather a surprising property with respect to the metallic behavior. For confirming and clarifying such behavior, in this work MoO_2 nanocrystals were prepared by solvothermal treatment of a Mo chloromethoxide at 250 °C in oleic acid. In this way, the as-prepared materials were organics-free and could be tested without any high temperature treatment. The as-prepared monoclinic MoO_2 nanocrystals, with a mean crystallite size of 29 nm, were used in photodegradation tests of organic pollutants (methylene blue, rhodamine B, paracetamol). It was found that MoO_2 was an efficient adsorbent of methylene blue ($43\ mg\ g^{-1}$ for 1.5×10^{-4} M concentration) in the dark and an efficient photodegradation catalyst under visible light (all methylene blue removed from the solution after 240 min). From the analysis of the combined photodegradation tests of rhodamine B and paracetamol, it was clarified that direct sensitization was responsible for photodegradation. This finding was related to the work function value of metallic MoO_2, placed at more negative values if compared with other metallic materials. The results paved the way to further consider metallic materials for photodegradation applications.

10:50 AM

(ICACC-S17-014-2024) 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 mention briefly some other materials that we have tested.

11:20 AM

(ICACC-S17-015-2024) Achieving high two-dimensional photoconductivity in Gd-modified bismuth ferrite via ferroelectric polarization

H. I. Mana-ay*[1]; C. Chen[2]; R. Chien[1]; C. Tu[3]; P. Chen[1]

1. Ming Chi University of Technology, International Ph.D. Program in Innovative Technology of Biomedical Engineering and Medical Devices, Taiwan
2. Hwa Hsia University of Technology, Mechanical Engineering, Taiwan
3. Fu Jen Catholic University, Department of Physics, Taiwan

This work demonstrates the critical roles of ferroelectric polarization and illumination intensity in modulating local photoconduction properties of 7 mol% Gd-modified bismuth ferrite ceramics by a microscale atomic force microscopy-based characterization under a 405 nm-wavelength illumination. A sequential increase in photocurrent with increasing illumination can be attributed to an increased number of photogenerated charge carriers. Furthermore, an enhancement in the photocurrent upon an external electric-field poling is correlated to the adjustment of the internal electric field induced by ferroelectric polarization, which boosts the charge mobility. The enhanced photoconduction is linked to the domain reorientation and increased domain-wall density caused by an electric-field poling. The evolution plays a critical role in the electric field-enhanced photocurrent since domain walls serve as conductive transport networks for charge flow. These findings uncover the vital effects of ferroelectric polarization on the photoelectric properties of BFO-based materials, thus providing essential insight for optoelectronic applications.

S18 Ultra-High Temperature Ceramics

SYMPOSIUM 18: Super-hard UHTCs

Room: Coquina A
Session Chairs: Douglas Wolfe, Pennsylvania State University; Christopher Weinberger, Colorado State University

8:30 AM

(ICACC-S18-010-2024) Synthesis & Characterization of Bulk Materials Towards the Development of Spinodally-Hardened, Superhard High Entropy Ceramics (Invited)

D. E. Wolfe*[1]; C. DeSalle[2]; C. Ryan[2]; R. Crealese[2]; S. Divilov[2]; S. Curtarolo[3]; W. Fahrenholtz[4]; J. Maria[1]; H. Eckert[5]; D. Brenner[6]; E. Zurek[7]

1. Pennsylvania State University, USA
2. Penn State ARL, USA
3. Duke University, Materials Science, Electrical Engineering and Physics, USA
4. Missouri University of Science & Technology, Dept. of Materials Science and Engineering, USA
5. Duke University, Mechanical Engineering and Materials Science, USA
6. North Carolina State University, Materials Science and Engineering, USA
7. University of Buffalo, Chemistry Department, USA

The increasing demand for materials that exhibit superior thermomechanical and thermochemical properties during extreme environment operation is driven by applications such as aerospace/hypersonic vehicles, friction stir welding, and machining. High entropy ceramic alloys have garnered significant interest due to a combination of solid-solution strengthening, phase stability control, and precipitation hardening effects via coherent spinodal decomposition. With a focus on the synthesis, analytical characterization, and performance evaluation of bulk materials, experimental advances have been achieved in relation to the development of spinodally-hardened, superhard high entropy ceramics. Attrition milling studies of various constituent ceramic nanoparticles for subsequent consolidation via field-assisted sintering technology (FAST) with in-situ heat treatments have enabled the synthesis of high entropy carbonitrides. This study highlights and deconvolutes the phase dependence of elasto-plasticity while introducing in-situ sintering heat treatments to realize the effects of spinodal nucleation and decomposition on phase evolution and mechanical properties.

9:00 AM

(ICACC-S18-011-2024) Mechanisms of Anomalous Hardness in Sub-Stoichiometric Transition Metal Carbides (Invited)

B. Watkins[1]; G. Thompson[2]; C. R. Weinberger*[1]

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

Transition metal carbides are well known UHTCs that exhibit both extreme melting temperatures and high hardness. The mechanical properties of these materials are dependent on the carbon/ content as well as the transition metal. The hardness of the group IVB carbides decrease monotonically with loss of carbon while 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 examine the physical origins that are responsible for the anomolous hardness by examining how dislocation slip changes between the group IVB and VB carbides. First, we compute the slip resistance using density functional theory to compute generalized stacking fault curves as a function of chemistry. Then, we use a Peierls Nabarro model to directly compute the slip resitance. The differences in dislocation mobility between the two groups is able to rationalize the different trends in hardness.

9:30 AM

(ICACC-S18-012-2024) Optimizing Hardness of High-Entropy Boride Thin Films by Modulating the Bipolar HiPIMS Kick-Pulse

N. S. McIlwaine*[1]; J. Maria[1]; N. O. Marquez Rios[1]

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

Group 4-6B transition metal borides (HEBs) possess favorable refractory properties such as high hardness and high melting temperatures, which are useful for applications in advanced armors, cutting and metal-joining tools, and spacecraft thermal protection systems. HEBs contain within a single crystal structure a chemically disordered cation sub-lattice of ~4 or more different elements, which contribute to the advanced thermal and mechanical properties compared to binary transition metal borides. Physical vapor deposition of thin film high entropy borides through bipolar high-power impulse magnetron sputtering (HiPIMS) is an efficient synthesis process to explore this vast composition space. This research investigates the ability to improve the surface morphology and smoothness of high entropy HEB thin films through manipulation of the positive kick pulse during the HiPIMS operation cycle, which additionally has a positive effect of increasing the microhardness.

S19 Molecular-level Processing and Chemical Engineering of Functional Materials

SYMPOSIUM 19: Energy-Related Matters I

Room: Ballroom 3
Session Chair: Anke Weidenkaff, Fraunhofer IWKS

8:30 AM

(ICACC-S19-009-2024) Smart use of functional materials in polymers and their implecations in solid-state lithium metal batteries (Invited)

J. R. Nair*[1]

1. Fraunhofer Gesellschaft, IWKS, Germany

The increasing demand for energy storage devices from society and the appeal for making them sustainable fuel efforts to develop lightweight, durable, safe, and flexible power sources, especially in the field of lithium-based batteries. Solid-state batteries (SSBs) are expected to provide these properties on a large scale and at a competitive price. In this context, Li-metal batteries that use organic polymers as solid-state electrolytes and separators are consistently emerging as promising prospects. To increase the safety, processability, and long-term stability of solid-state lithium metal batteries, polymer composites have been explored as a viable alternative electrolyte. In this context, organic and inorganic materials, engineered nanomaterials, etc. have been used as fillers in polymer composites. Therefore, the use of various functional materials as fillers in polymer composites and their application as an electrolyte and separator in lithium metal batteries are interesting. The electrolyte fabrication processes, materials and their properties, and overall impact on battery performance are important criteria while exploring functional materials in electrolytes. In addition, the prospects of solvent-free polymerization and its potential application in the integration of SSB devices are vital for the large-scale adoption of sustainable batteries.

9:00 AM

(ICACC-S19-010-2024) Opportunities for polymer-derived ceramics composites in sodium and potassium-ion batteries (Invited)

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

1. Kansas State University, Mechanical and Nuclear Engineering Dept., USA
2. Kansas State University, Mechanical 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 (FTIR), Scanning Electron Microscopy (SEM), and X-ray Photoelectron Spectroscopy (XPS) 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.

9:30 AM

(ICACC-S19-011-2024) Topochemical reactions and the corresponding strategies to develop novel photocatalysts with tailored optical properties (Invited)

S. Perween*[1]; O. Clemens[1]

1. University of Stuttgart, Institute for Materials Science, Germany

The recent advances of Ruddlesden–Popper–type (RP) materials have attracted particular interest because of their unique structural, electronic, and optical properties that show a high potential as a photocatalyst. A few RP-type materials such as Sr_2TiO_4 and $Li_2CaTa_2O_7$ have been reported to be efficient hydrogen evolution catalysts. This lecture will present recent results on topochemical fluorination of a new class of photocatalyst, indate-based RP-type oxide (e.g. $LaBaInO_4$ to $LaBaInO_3F_2$) that shows a strong impact on the structural and optical properties of the materials developed as well as photocatalytic activities for sacrificial hydrogen evolution. The introduction of fluorine via topochemical fluorination reaction into the intercalation-based RP-type oxide adds a new dimension to control and tune the electronic and atomic structure of materials therefore affecting the physical properties. The synthesis of oxyfluorides has been obtained successfully via low-temperature topochemical reactions, and characterized by a variety of experimental analytical techniques. The structural-optical impact upon topochemical fluorination suggests a valuable tool to alter the optical properties of the materials developed while maintaining precise control of the fluorine content added to the RP-type oxide materials. Acknowledgment: DFG Fund within Project Number 518952364

S1 Mechanical Behavior and Performance of Ceramics & Composites

SYMPOSIUM 1: Environmental effects and thermomechanical performance of ceramic matrix composites (CMCs)

Room: Coquina E
Session Chairs: Gerard Vignoles, University Bordeaux; Craig Smith, NASA Glenn Research Center

8:30 AM

(ICACC-S1-011-2024) Fatigue of a SiC/SiC Ceramic Composite with an Ytterbium-Disilicate Environmental Barrier Coating at Elevated Temperature (Invited)

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

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

Tension-tension fatigue performance of a SiC/SiC composite with an ytterbium-disilicate environmental barrier coating (EBC) was investigated at 1200°C in air and steam. The composite is reinforced with Hi-Nicalon™ SiC fibers and has a melt-infiltrated matrix processed by chemical vapor infiltration of SiC with subsequent infiltration with SiC particulate slurry and molten silicon. The EBC consists of a Si bond coat and an $Yb_2Si_2O_7$ top coat applied via air plasma spraying. Tensile properties were evaluated at 1200°C. Tension-tension fatigue was examined for maximum stresses of 110-140 MPa. To assess the efficacy of EBC, experimental results obtained for the coated composite are compared to those for a control uncoated composite. Surface grit-blasting inherent in the EBC application process degrades tensile strength of the composite. However, the EBC effectively protects the composite from oxidation embrittlement during cyclic loading in air or steam. Fatigue runout set to 200000 cycles (55.6 h at a frequency of 1.0 Hz) was achieved at 110 MPa in air and steam. Retained properties of pre-fatigued specimens were characterized. Composite microstructure, along with damage and failure mechanisms were investigated. Damage and failure of the composite are attributed to growth of cracks originating from numerous processing defects in the composite interior.

9:00 AM

(ICACC-S1-012-2024) Modeling oxide scale growth behavior of CMC

S. Fukuhara*[1]; S. Kanazawa[2]; M. Begley[3]

1. IHI Corporation, Japan
2. IHI Americas Inc., USA
3. University of California, Santa Barbara, USA

When using CMCs in high-temperature environments, material degradation due to oxidation can significantly limit performance. In creep tests conducted at 1100°C and 700°C to characterize the effects of oxidation, it was found that the cross-sectional area of oxide scale generation within the CMC was smaller at the high-temperature condition of 1100°C, even though the oxide scale growth rate should be greater than at the mid-temperature condition of 700°C. In this study, we constructed a mathematical model to explain the physical mechanism of this phenomenon. The model assumes that oxide scale forms on a SiC matrix crack surface in proportion to the oxygen concentration at a given position; the closure of transport pathways due to oxide formation inhibits the ingress of oxidant, resulting in spatial variations of the oxide scale thickness. An analysis was performed using the model up until the time that inlet was sealed by the oxide scale. At high temperatures, oxygen consumption due to rapid oxide scale growth near the inlet was significant, resulting in significant decrease in oxygen concentration in the interior. As a result, the amount of interior oxidation scale was smaller at high temperatures than at mid-temperatures. The model illustrates that the degree of interior oxidation can be smaller at high temperatures, due to oxygen consumption near the inlet that starves interior regions of oxidant and limits oxide scale formation.

9:20 AM

(ICACC-S1-013-2024) Mechanical and Physical Properties of CVI-based UHTCMCs (Invited)

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

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

This presentation will focus on the mechanical and physical properties of ultra-high temperature ceramic composites based on carbon fibre, Cf, preforms impregnated with zirconium (ZrB_2) and/or hafnium diboride (HfB_2) powder and then densified with carbon by chemical vapour infiltration, CVI.

9:50 AM

(ICACC-S1-014-2024) Stress rupture mechanisms of an orthogonal 3-D woven amorphous SiC fiber/SiC/YSi_2-Si Matrix Composites at elevated temperature in air

S. Kanazawa*[1]; T. Matsumoto[2]; N. Yamazaki[3]; Y. Asakura[3]; T. Aoki[4]; T. Ogasawara[2]; F. W. Zok[5]

1. IHI Americas Inc., USA
2. Tokyo University of Agriculture and Technology, Japan
3. IHI Corporation, Japan
4. Japan Aerospace Exploration Agency, Advanced Composite Research Center, Institute of Aeronautical Technology, Japan
5. University of California, Santa Barbara, USA

Stress rupture behavior of an orthogonal 3-D woven composite comprising amorphous SiC fibers and a low-melting temperature Si-Y alloy matrix was investigated under constant tensile loading at 700 and 1100 °C in air. On the basis of the Larson-Miller parameter, the composite exhibits rupture strength at 700 °C that is similar to that of composites with other amorphous SiC fibers; in contrast, its rupture strength at 1100 °C was somewhat better. Fractographic observations show that internal oxidation of the fiber coatings occurs more extensively at 700 °C than at 1100 °C. The difference is attributable to the filling of near-surface matrix micro-cracks with oxides at the higher temperature, thereby retarding ingress of oxidants to the fiber coatings within the composite interior. This crack-healing process is believed to contribute to increasing the rupture lifetime.

10:30 AM

(ICACC-S1-015-2024) Environmental Degradation Mechanisms of SiC/SiC Composites with SiC/PyC Bilayer Interphase in Wet Oxygen

W. Wu*[1]; Y. Song[1]; G. Yu[1]; X. Gao[1]

1. Nanjing University of Aeronautics and Astronautics, China

The interface between fibers and the matrix plays a crucial role in determining the environmental performance of ceramic matrix composites (CMCs). This study investigates the environmental degradation behavior of SiC/SiC composites featuring a SiC/PyC bilayer interphase. The assessment is based on microstructural and mechanical changes observed under various wet oxygen atmospheres over a 10-hour period. Our findings reveal that these multi-interphase composites exhibit their highest strength retention in environments characterized by elevated temperatures and low levels of water vapor (≤10%). Conversely, both low temperatures and excessive moisture levels lead to significant mechanical property deterioration. The thin silica layer formed at low temperatures is insufficient to compensate for the loss of interfacial properties due to PyC consumption. Meanwhile, the oxide scale formed under excessive moisture introduces a multitude of defects.

10:50 AM

(ICACC-S1-016-2024) Influence of Temperature on Mechanical Behavior of Unidirectional SiC/SiC Composites under Monotonic Tensile Loading

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

1. Oklahoma State University, Materials Science & 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 the damage mechanisms and predict the durability of these systems, in-depth knowledge of their behavior at ambient and high temperatures is necessary. A method to conduct monotonic tensile testing of SiC/SiC minicomposites at elevated temperatures to investigate their thermomechanical behavior will be presented. The goal is to employ nondestructive health monitoring techniques in-situ at high temperatures, including acoustic emission (AE), electrical resistance (ER), and digital image correlation (DIC) to evaluate damage initiation and evolution in the minicomposite specimens during tensile testing. Results from mechanical testing and nondestructive techniques will be presented. Temperature dependence of specimen mechanical properties including elastic moduli, ultimate tensile strength, and AE-based onset of matrix cracking will be presented and discussed. Electron microscopy and micro-computed tomography were used to further characterize specimen damage after testing.

11:10 AM

(ICACC-S1-017-2024) Evaluation of mechanical behaviour at high temperature and damage caused by thermal cycling of C_f/SiC with multilayered interphase and modified matrix

R. Krishna*[1]; P. Wilson[2]; M. A Williams[2]; P. Sreerangam[2]; U. Andi[3]; R. Mitra[1]

1. Indian Institute of Technology Kharagpur, Department of Metallurgical and Materials Engineering, India
2. University of Warwick, WMG, United Kingdom
3. Council of Scientific and Industrial Research - National Aerospace Laboratories, Materials Science Division, India

In the present study, the mechanical behaviour of Chemical Vapour Infiltration (CVI) processed C_f/SiC composites (2.5D (woven) and 3D NOOBed) with multilayered $(PyC/SiC)_{n=4}$ interphase and a modified SiC/Si-B-C matrix has been investigated at both ambient and service conditions. Observation by TEM along with EDS analysis of the sample has revealed the presence of ~50 nm thick PyC interphase, which is responsible for the weakening of the fibre-matrix interfacial bond. Using X-ray computed micro-tomography to quantify the CVI-induced porosities, it is found that the arrangement of inter-bundle pores is more ordered in the 3D composite in comparison to 2.5D composites, which influences the crack propagation and failure of these composites. The tensile strengths of 2.5D and 3D composites at 1250°C have been found higher than those at 27°C by 22% and 17%, respectively. Furthermore, the effect of thermal cycling (30 cycles between 80°C and 1250°C) on the composites has shown 99% and 78% of strength retention for 2.5D and 3D composites, respectively. Measurements of elastic constants of the composites by ultrasonic phase spectroscopy before and after thermal cycling, have indicated that along with fibre architecture, volume and orientation of pores also influence the stiffness of these composites.

11:30 AM

(ICACC-S1-018-2024) High-Temperature Mechanical Behavior of Ceramic-Matrix Composites: Strength and Lifetime (Invited)

L. Li*[1]

1. Nanjing University of Aeronautics and Astronautics, College of Civil Aviation, China

Ceramic-matrix composites (CMCs) possess high specific strength and specific modulus at elevated temperatures and have already been applied in hot-section components in aerospace. To ensure the reliability and safety of the components during operation, it is necessary to understand the damage mechanisms under different loading conditions and establish the models and tools to predict the strength and lifetime especially at elevated temperatures. This paper focuses the on the strength and lifetime of C/SiC and SiC/SiC composites at elevated temperatures. The temperature-dependent of damage mechanisms, i.e., matrix multiple cracking, interface debonding and oxidation, and fibers fracture and pullout, were analyzed and considered in the micromechanical models. The micromechanical time-dependent constitutive model was developed to predict the composite's tensile stress-strain curves. The composite's lifetime under cyclic fatigue loading and stress-rupture loading at elevated temperature was predicted based on the Global Load Sharing criterion, and the effect of stochastic load spectrum on the lifetime was also considered. The developed micromechanical constitutive models and approaches were adopted to predict the strength and lifetime of C/SiC and SiC/SiC composites at elevated temperatures.

S3 21th Intl Symp on Solid Oxide Cells Materials Science & Technology

SYMPOSIUM 3: Air electrode

Room: Ballroom 1-2
Session Chairs: Kevin Huang, University of South Carolina; Dimitrios Niakolas, FORTH/ICE-HT

8:30 AM

(ICACC-S3-009-2024) Enhancing Low-Temperature SOFC Performance and Durability via Surface Modification and Scaling High Power Cells (Invited)

E. D. Wachsman*[1]

1. University of Maryland, USA

Low temperature operation (≤650 °C) enables high surface area nano-catalysts with enhanced activity and high durability. A number of nano-catalysts were evaluated with PrO_x being the best. The activity was in fact so high that SOFCs fabricated with a GDC scaffold and impregnated with PrO_x, without a separate "cathode" phase, obtained exceptionally high-power densities at low temperature. This high-power density is demonstrated not only in button cells, but do to readily scalable processing in full format commercial scale cells.

9:00 AM

(ICACC-S3-010-2024) Optimization of LSCF Air Electrodes for Infiltration

J. Liu*[1]; T. Yang[2]; B. Guan[2]; Y. Picard[2]; R. Pineault[1]; T. Kalapos[2]; H. W. Abernathy[1]

1. National Energy Technology Laboratory, Thermal Sciences, USA
2. LRST-NETL, USA

Surface modification of solid oxide cell (SOC) air electrodes through nano-catalyst infiltration has demonstrated its effectiveness. This method enhances performance by boosting both electrochemical activity and the density of active reaction sites. However, when dealing with the intricate oxygen exchange reaction on widely used LSCF-based electrodes, the enhancement of electrode performance depends not only on catalyst selection but also significantly on the underlying initial microstructure of the electrode backbone. This study focuses on the fundamental understanding and optimization of the LSCF based SOC air electrode specifically for infiltration. Electrodes with various microstructure properties were prepared with and without catalyst infiltration. Changes of electrode performance and initial degradation under reversible operating conditions are analyzed. Multiphysics simulations allowed for a deeper understanding of the interplay between the catalyst and the microstructure of the electrode backbone. Through this investigation, a comprehensive quantitative analysis of resistance and various losses originating from materials and microstructural variations was obtained. This study unveiled that the presence of a highly active catalyst through infiltration leads to comparable electrode performance, regardless of the initial microstructure and backbone performance.

9:20 AM

(ICACC-S3-012-2024) Synthesis, Performance analysis and Investigation of $NdBaCo_2O_{5+x}$ as an auspicious oxygen electrode material for reversible solid oxide fuel cells

A. Sreelakshmi*[1]; M. Rushabh[1]; S. Senthilkumar[1]; S. Aruna[1]

1. CSIR-National Aerospace Laboratories, Bengaluru, Surface Engineering Division, India

The reversible solid oxide fuel cells (RSOFC) can operate in electrolyzer and power generation modes. To have better durability and cell performance, an efficient material should be developed. The $NdBaCo_2O_{5+x}$(NBCO) oxygen electrode shows lower delamination comparitively [1]. In the present work, the double perovskite oxygen electrode has been utilized for oxygen ion conducting electrolyte 8 mol % ytrria stabilized zirconia (8YSZ). NBCO has been synthesized using combustion synthesis and the cell was fabricated with Fuel electrode(Nickel oxide – 8YSZ), Electrolyte(8YSZ), and Oxygen electrode(NBCO) with thickness 23.06, 529.9, and 20.12 μm respectively. The power density achieved was 73.5 and 48 mW cm^{-2} at 845 and 789°C respectively. The ohmic resistance of the cell decreases with temperature, and it was 3 Ωcm^2, 2.8 Ωcm^2, 2.7 Ωcm^2 at 775, 794, and 840°C. Further a $Gd_{0.1}Ce_{0.9}O_{1.95}$(GDC) interlayer was introduced along with the $NdBaCo_2O_{5+x}$ oxygen electrode so as to decrease the resistance of the electrode- electrolyte interface. It is expected that the interface can match the Thermal Expansion Coefficient(TEC) and reduce the delamination issue. Based on the encouraging results reported on NBCO for proton conducting fuel cells[2,5] the oxygen ion conducting fuel cells with double perovskite Electrode expected to have improved performance.

9:40 AM

(ICACC-S3-013-2024) Heterostructured Cr Resistant Oxygen Electrode for SOECs (Invited)

X. Liu*[2]; A. Kalu[3]; G. Liu[1]; C. Klemstine[2]; S. Yang[1]; W. Li[3]; E. M. Sabolsky[2]; Y. Zhong[1]

1. Worcester Polytechnic Institute, Mechanical and Materials Engineering, USA
2. West Virginia University, Mechanical & Aerospace Engineering, USA
3. West Virginia University, Department of Chemical and Biomedical Engineering, USA

High oxygen content in the air electrode of the high temperature solid oxide electrolysis cell (SOEC) presents a challenge to achieve rapid oxygen evolution and resistance to Cr poisoning. In this study, we employ an interstitial oxygen-conducting material, $La_2NiO_{4+\delta}$ (LNO), as the backbone for the air electrode, coated with a thin layer of a high-entropy perovskite (HEP) to form a heterostructured air electrode, enhancing performance and resistance to Cr. The exceptional Cr resistance of LNO is confirmed through electrical conductivity relaxation tests, showing no degradation after exposure to 80 ppt Cr for 200 h at 700°C, along with low oxygen evolution reaction kinetics. The HEP coating significantly improves the performance, increasing the surface exchange coefficient from 2x10^{-3} cm/s for LNO to 1.1x10^{-2} cm/s for the LNO sample coated with HEP $(La_{0.2}Sr_{0.2}Pr_{0.2}Y_{0.2}Ba_{0.2})(Co_{0.2}Fe_{0.8})O_3$. Although the coated sample exhibits initial degradation due to microstructural adjustments, it maintains a high surface exchange coefficient of 8.5x10^{-3} cm/s after exposure to Cr for 700 h. The HEP materials used demonstrate high Cr resistance due to the absence of Sr surface segregation and exhibit excellent surface exchange characteristics attributed to the active elements at the A site. In addition, systematic ab initio simulations were carried out on the LNO, HEP, and their interface on the stability and electrical conductivities.

10:30 AM

(ICACC-S3-014-2024) A novel high performance Cobalt-free air electrode for Solid Oxide Electrolysis Cell technology (Invited)

C. J. Ferchaud*[1]; F. F. van Berkel[1]; M. Stodolny[1]; L. Berkeveld[1]; M. Heijink-Smith[1]; M. Langerman[1]; J. Veldhuis[1]; X. Lu[1]; D. Montinaro[2]; C. Curzel[2]

1. TNO, Netherlands
2. SolydEra, Italy

Reduction of Critical Raw Materials (CRM) in the manufacturing of Solid Oxide Cell (SOC) is a key requirement to achieve low cost and large-scale commercialization of high temperature electrolysis and fuel cell systems. Cobalt is state-of-the-art compound in SOC electrodes, requiring costly safety management during manufacturing and recycling processes due to its carcinogenic properties. In addition, cobalt recognized since 2017 as CRM by the European community, presents an economical risk for SOC manufacturers regarding its supply. In the European project NewSOC, TNO has developed a novel concept of Co-free air electrode, based on the perovskite material $La_{0.6}Sr_{0.4}FeO_{3-\delta}$, to reduce the overall CRM content and cost in the SOC manufacturing and to improve performance and lifetime for electrolysis operations. Operations at the current density of 1.30 A/cm^2 with a fuel utilization of 80% has been demonstrated at 750°C at the thermal neutral voltage of 1.3V during single cell testing on 10x10 cm cell (81 cm^2 surface area) under steam electrolysis conditions (90% H_2O, 10%H_2). Integrated in the cell design of the EU commercial SOC stack supplier SolydEra, the Co-free air electrode is currently tested on short-stack level (6-9 cells) at SolydEra. The Co-free air electrode development made by TNO and the on-going short-stack test results at SolydEra will be the core of this communication.

11:00 AM

(ICACC-S3-015-2024) A High Performing Intermediate Temperature ZrO_2-Based Reversible Solid Oxide Cell Achieved by a New Barrier Layer Free Oxygen Electrode

J. Lu[1]; K. Huang*[1]

1. University of South Carolina, USA

Developing high performance oxygen electrodes (OEs) is a key to capitalizing intermediate temperature (IT) solid oxide cells. Here we report on a highly oxygen-active OE for IT ZrO_2-based SOECs without a CeO_2 barrier layer required. The new barrier layer free (BLF) OE consists of a high-conductivity oxide-ion conductor ($(Bi_{0.75}Y_{0.25})_{0.93}Ce_{0.07}O_{1.5\pm\delta}$, denoted as BYC) and a high-conductivity electronic conductor ($La_{0.8}Sr_{0.2}MnO_3$, denoted as LSM) to achieve fast oxygen reduction/evolution reaction (ORR/OER). Featuring a microscale porous BYC scaffold decorated with high-surface-area LSM nanoparticles (NPs), the new BLF-OE exhibits a low area-specific-resistance (ASR) of 0.10 Ω cm^2 at 650 °C in air. With 50%H_2-50%H_2O as a feed to hydrogen electrode (HE) and air to OE, the single-cell performance achieves 588 mA cm^{-2} at 0.80 V in the fuel cell mode and 688 mA cm^{-2} at 1.30 V in the electrolytic mode at 650 °C. The long-term stability testing under alternating fuel cell and electrolytic modes shows a low degradation rate of 0.10 mA cm^{-2} h^{-1} over 550 hours. These encouraging results promise the newly developed BYC-LSM to be an excellent OE for intermediate temperature SOECs.

11:20 AM

(ICACC-S3-016-2024) Improvement of air-side contacting in SOCs

Y. S. Ayhan*[1]; O. Guillon[1]; N. H. Menzler[2]

1. Forschungszentrum Juelich, IEK-1, Germany
2. Forschungszentrum Jülich GmbH, IEK-1, Germany

Energy transition worldwide can be realized with the contribution of promising and advanced technologies such as solid oxide cells (SOCs). Even though commercial products can be seen in the market, this ceramic-based technology can be still improved to increase both working efficiency and lifetime and decrease the cost of the end product. Nonoptimal air-side contacting in SOC stacks causes cell performance loss compared to single-cell measurements and may be prevented via a novel design which is the direct printing of an air-side electrode contact layer in a rib-channel form. This ceramic layer provides also gas distribution that eliminates the need for machined or stamped gas channels on the metallic interconnector. Since the machining / pressing process is costly and time-consuming, printing of this ceramic layer with the novel design might be advantageous in terms of performance increase and cost-effectiveness in SOC stacks. In this work, pastes were prepared, characterized via rheometer, and printed via stencil printing. The main challenge is to print relatively thick layers as stripes thereby conserving a kind of ideal rectangle-like form. Thus, ca. 500 μm thick ribs with appropriate surface flatness were successfully printed and characterized. In addition, this work aims at finding the best-performing design, microstructure and material combination of the rib-channel form which will be investigated.

S5 Next-Generation Bioceramics and Biocomposites

SYMPOSIUM 5: Next Generation Bioceramics and Biocomposites

Room: Coquina B
Session Chair: Hui-suk Yun, Korea Institute of Materials Science

8:30 AM

(ICACC-S5-009-2024) Antimicrobial/virucidal composite coatings for different applications (Invited)

C. Balagna*[1]; A. Luceri[1]; F. Gattucci[1]; S. Perero[1]; M. Ferraris[1]

1. Politecnico di Torino, DISAT, Italy

The emergence of new bug/virus-related diseases and the growing problem of antimicrobial resistance pose significant risks to human wellbeing. In response to these global concerns, researchers have been driven to find innovative solutions. The authors developed antimicrobial/virucidal composite coatings using two distinct methods: the co-sputtering technique and polymer-ceramic precursors. These coatings consist of silver nanoparticles embedded within a glass or ceramic matrix, enabling their application to various surfaces. The silver nanoclusters exhibit a controlled release of metallic ions, which effectively inhibits the adhesion and proliferation of various bacterial strains and fungi. Additionally, the coatings demonstrate potent virucidal activity against several airborne viruses, including SARS-CoV-2. The co-sputtering technique is not only promising but also industrially scalable, versatile, and environmentally friendly. Moreover, the technique allows for precise control over the coating's composition and thickness, enhancing its adaptability for different applications. Alternatively, the use of pre-ceramic polymeric precursors has been explored to design hybrid coatings with enhanced efficacy. These precursors facilitate the incorporation of silver nanoparticles into the matrix, thereby imparting specific antimicrobial properties to the composite coatings.

9:00 AM

(ICACC-S5-010-2024) Development of nanostructured composite coatings containing silver nanoclusters for air filtration systems

A. Luceri*[1]; S. Perero[1]; M. Donalisio[2]; R. Francese[2]; D. Lembo[2]; M. Ferraris[1]; C. Balagna[1]

1. Politecnico di Torino, Department of Applied Science and Technology, Italy
2. Università di Torino, Department of Clinical and Biological Sciences, Italy

In recent years, the world has witnessed the rapid spread of a serious infectious diseases: Coronavirus. This global health crisis has also led to severe economic consequences, prompting extensive research into new methods to prevent the transmission of viruses and subsequent infections. As infections can be transmitted through droplets or contaminated surfaces, and with many microorganisms developing resistance to antibiotics and therapies, finding a solution has become of paramount importance and is of worldwide interest. One potential solution involves inhibiting the proliferation of bacteria and viruses through the use of antimicrobial and antiviral composite coatings on various devices. This study focuses on air filters, where coatings composed of silica or zirconia matrix with silver nanoclusters were deposited using the co-sputtering technique. Preliminary characterization of the coatings assessed their morphological and compositional homogeneity. The effectiveness of these coatings was tested against S. Epidermidis, a Gram-positive bacteria, and against Coronavirus, Influenza A virus, Rhinovirus, showing promising results in terms of antibacterial and antiviral activity. Overall, these advancements in antimicrobial and antiviral composite coatings could play a crucial role in preventing the spread of infectious diseases and improving global health and safety.

9:20 AM

(ICACC-S5-011-2024) The effect of surface ion-doping on the bioactive glass cytocompatibility and antibacterial performance

M. Lallukka*[1]; A. Houaoui[2]; M. Miola[1]; Z. Najmi[3]; A. Cochis[3]; J. Massera[2]; L. Rimondini[3]; E. Vernè[1]

1. Politecnico di Torino, Department of Applied Science and Technology, Italy
2. Tampere University, Faculty of Medical Sciences and Technology, Finland
3. Università degli Studi del Piemonte Orientale, Italy

In orthopedic infection treatments, biofilm prevention while fostering bone growth is a critical challenge. Bioactive glasses doped with silver (Ag), or copper (Cu) ions offer a potential solution. However, the dopant toxicity must be addressed, as their presence may pose cytotoxic risks. This study evaluated cytocompatibility and antibacterial performance of Ag- and Cu-doped bioactive glasses (Ag-SBA2, Cu-SBA3). Human adipose stem cells (hASCs) underwent direct culture on glass discs, with and without pre-incubation, and on fibronectin-coated discs. In addition, the glasses' antibacterial activity against Staphylococcus aureus was assessed. Direct cell culture initially reduced cell viability but improved when cells were cultured on pre-treated discs. Fibronectin adsorption significantly enhanced Ag-SBA2 cytocompatibility, while Cu-SBA3 requires optimization. Regarding antibacterial activity, Cu-SBA3 reduced S. aureus metabolic activity after 24 hours, confirming its potential as a bioactive and antibacterial surface. However, further optimization is needed for Cu-SBA3 due to its cytotoxicity. Ag-SBA2 shows promise as a treatment against early infections while maintaining long-term cytocompatibility and bioactivity. This project is funded by the Eu Horizon 2020 research and innovation programme, Marie Sklodowska-Curie grant agreement No 860462.

9:40 AM

(ICACC-S5-012-2024) Control of Cell Recruitment by Surface Orientation of Hydroxyapatite/Collagen Bone-Like Nanocmposite

M. Kikuchi*[1]; T. Hasegawa[2]; N. Amizuka[2]

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

Hydroxyapatite/collagen bone-like nanocomposite (HAp/Col) having bone-like nanostructure and chemical composition is a synthetic material incorporating into bone remodeling process, i.e., the HAp/Col is resorbed by osteoclasts followed by bone formation by osteoblasts. Previous researches on bone tissue reactions of the HAp/Col are performed for the HAp/Col with random microstructure of the HAp/Col fibers. In the present study, the HAp/Col dense body was dewatered by a uniaxial pressing in the specially designed mold at a thickness of 3.6 mm. The HAp/Col dense body had a layered structure of randomly oriented fibers; thus, surfaces parallel to top surface (p-surface) exposed mainly m-surfaces (so-called a-surfaces) of HAp and long axes of Col molecules, and vertical to top surface (v-surface) were rich in end faces of the HAp/Col fibers, i.e., rich in c-surfaces of HAp and end faces of Col mokecules. After implantation of this layered HAp/Col of 3.6 mm in thickness and 2 mm in diameter into hole on tibia of Wistar rats, the p-surface showed recruitment of osteoblasts only followed by bone formation and the v-surface recruitment of osteoclasts only followed by osteoclastic resorption. These results indicates orientation of the HAp/Col control the recruitments of cells.

S6 Advanced Materials and Technologies for Rechargeable Energy Storage

SYMPOSIUM 6: Solid electrolytes for batteries I

Room: Ballroom 5
Session Chairs: Chongmin Wang, Pacific Northwest National Lab; Naoaki Yabuuchi, Yokohama National University

8:30 AM

(ICACC-S6-009-2024) High-entropy solid electrolytes with garnet framework (Invited)

N. Zettsu*[1]

1. Shinshu University, Department of Materials Chemistry, Japan

Since the ionic conductivity of solid electrolytes is optimized by their crystal structure and atomic arrangement, the conductivity at the heterointerface and the grain boundary region with the disturbance of the atomic arrangement decreases by 2 ~ 3 orders of magnitude compared with the bulk. In addition, at the dynamic electrode/solid electrolyte interface where an electric field is applied, new resistance layers are continuously generated by modulation and formation of distribution such as lithium-ion concentration and local atomic arrangement, and finally ion conduction is fully insulated. Our research group is working on the realization of high ionic conductivity by introducing the randomness of sublattices (high entropy) in multi-elemental substitution and the enhancement of the performance of electrochemical performance by stabilizing the grain boundaries and heterointerface structures. The nonlinear interactions among the many constituent atoms can be expected to give rise to electrical, mechanical, chemical, and electrochemical properties that cannot be realized by simple solution laws. In this paper, using a garnet-type crystalline solid electrolyte of $Li_7La_3Zr_2O_{12}$ as a model compound, we aim to realize high ionic conductivity by introducing the high entropy effect, and to improve the stability of heterointerface with $LiCoO_2$ cathode.

9:00 AM

(ICACC-S6-010-2024) Operando Optical Imaging Platforms to Study Battery Reactions (Invited)

X. Shan*[1]; G. Feng[1]; G. Thomas[1]

1. University of Houston, Electrical and Computer Engineering, USA

The traditional imaging and measurement techniques have experienced challenges to characterize these complicated interfacial chemical reactions in batteries. For example, most of the methods only provide rich information at a certain time point in the dynamic process or measure an average result over a large area during the reaction. On the other hand, these interfacial reactions are highly dynamic and spatially varied, and the signals at different time points or different locations could be totally different. We have developed a multimodal optical imaging platform to image the battery electrode reaction dynamics across the interfaces throughout the entire reaction process. First, we developed a dynamic, non-invasive, operando reflection interference microscope (RIM) to enable the real-time imaging of SEI and Li nucleation dynamics during its formation and evolution processes with high sensitivity. Secondly, we have built a 3D optical microscope, which allows us to image the morphology changes during the battery reaction dynamics. The Zn deposition process on 3D Zn-Mn alloy electrode has been studied, and the 3D morphology and the Zn nucleation have been imaged in situ. Thirdly, we also used optical microscope to study the solid state battery's reaction dynamics and image the Li diffusion process in the individual NMC particles.

9:30 AM

(ICACC-S6-011-2024) Polymer Electrolytes for Lithium Batteries (Invited)

H. Teng*[1]; H. Nguyen[1]

1. National Cheng Kung University, Chemical Engineering, Taiwan

Li-metal batteries (LMBs) are promising candidates for next-generation high energy-density batteries. Solid polymer electrolytes (SPEs) provide an intimate contact with electrodes and accommodate volume changes in the Li-anode, making them ideal for solid-state batteries; however, confined chain swing, poor ion-complex dissociation, and barricaded Li^+-transport pathways limit the ionic conductivity of SPEs. In the present study, we developed an interpenetrating polymer network electrolyte (IPNE) comprising O- and F-containing networked SPEs (O-NSPE and F-NSPE, respectively) and lithium bis(fluorosulfonyl) imide (LiFSI) with the following fuctions to achieve high performance of the resulting LMBs: (a) connect FSI^--aggregate domains for facile Li^+ diffusion; (b) dissociate complexed ions and expedite the Li^+ transport; and (c) prevent space-charge zone formation on the Li-anode surface for uniform Li deposition. In experiments, a $Li|IPNE|LiFePO_4$ battery outperformed liquid electrolyte-based batteries in terms of charge-discharge performance.

S8 18th Intl Symp on APMT for Structural & Multifunctional Materials & Systems

SYMPOSIUM 8: Microwave processing, SPS, flash sintering, high pressure assisted sintering III

Room: Coquina F
Session Chair: Michael Halbig, NASA Glenn Research Center

8:30 AM

(ICACC-S8-011-2024) Review of routes to sinter silicon nitride-based ceramics (Invited)

S. Hampshire*[1]

1. University of Limerick, The Bernal Institute, Ireland

Silicon nitride-based ceramics are major structural materials that have been the subject of many years of intensive research. These ceramics exhibit high flexural strength, good fracture resistance, high hardness and excellent wear resistance. These properties arise through the processing of the material which involves use of mixed oxide additives to provide conditions for liquid phase sintering and the development of microstructures in which high aspect ratio grains and intergranular glass phase lead to high fracture toughness and strength. This paper reviews the development of silicon nitride and the related sialons and their sintering and our understanding of the effects of grain boundary chemistry and structure on microstructure–property relationships. The use of unusual additive combinations and processes such as Spark Plasma Sintering (SPS) and other heat treatments on densification and $\alpha \rightarrow \beta$ transformation is discussed.

S2 Advanced Ceramic Coatings for Structural/Environmental & Functional Applications

SYMPOSIUM 2: Innovative Processing of Coatings

Room: Flagler C
Session Chair: Kuiying Chen, National Research Council Canada

8:40 AM

(ICACC-S2-019-2024) Microstructure and Scattering Coefficient Relationship in Thermal Barrier Coatings

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

1. Florida Institute of Technology, Mechanical Engineering, USA

Thermal barrier coatings (TBCs) are widely used to protect hot components in gas turbines. With the higher turbine inlet temperature, the thermal radiative transfer becomes important in the overall heat transfer in TBC. The radiative properties are closely related to the coating's microstructure. By changing the microstructure, radiative properties of TBC can be altered. It has been shown that mixing polyester powder into yttria-stabilized zirconia (YSZ) powder before thermal spraying is an effective way to control the microstructure of air plasma sprayed (APS) topcoat. In this study, several YSZ films are sprayed with different volumetric mixing ratios of YSZ and polyester. Samples are heat-treated in a kiln at 1200C for 5 hrs and 25 hrs, respectively, to investigate the changes in the radiative properties. The transmittance, and reflectance of the as-sprayed and heat-treated films are measured over the near-infrared wavelength range using an FTIR spectrometer. The radiative properties are inverted from the measurement via a hybrid four-flux and discrete ordinates method. The changes in radiative properties before and after the heat treatments are discussed. Based on the SEM images of YSZ films' cross sections, the morphology-based pore size distributions (MPSD) of both as-sprayed and heat-treated films are obtained and the relationship between the MPSD and radiative properties is investigated.

9:00 AM

(ICACC-S2-020-2024) An Novel Design for Nozzles for the Cold Spray and Micro-Cold Spray of Ceramic Coatings From Fine Particles

S. Bierschenk*[1]; D. Kovar[1]

1. University of Texas, Mechanical Engineering, USA

Micro-cold spray (MCS), also referred to as the aerosol deposition method, is the process of accelerating nanoparticles in a gas stream through a nozzle and impacting them at high velocity onto a substrate in vacuum to deposit metal and ceramic films. Cold spray (CS) is a similar process that accelerates particles using a high-pressure gas and deposits coatings into atmospheric pressure. A bow shock and repressurized stagnant gas above the substrate surface slow particles during deposition for both MCS and CS. This slowing results in minimum particle sizes to maintain high velocity during deposition of ~200 nm and 5 μm for MCS and CS, respectively. Due to the large particle sizes required, deposition of ceramics via CS is limited because ceramic particles >5 um typically erode the substrate instead of building a solid film. Recently developed pressure relief channel nozzles that reduce the pressure of the stagnant gas above the substrate can reduce particle slowing while maintaining high velocity gas flow, allowing for the deposition of finer particles for MCS and CS. The high velocity deposition of finer particles can improve the deposition efficiency of MCS and allow for the deposition of ceramic films via CS. Nozzle designs and resulting particle impact velocities are studied numerically, and films fabricated using the improved nozzle designs are examined.

9:20 AM

(ICACC-S2-021-2024) Comparison of the blocking behavior of APS, SPS and PS-PVD TBC systems on substrates with cooling holes

M. Rüßmann*[1]; E. Bakan[1]; S. Schrüfer[2]; R. Vassen[1]

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

High temperature turbine aerofoils and combustion chamber walls in jet engines rely on film cooling and thermal barrier coating systems (TBCs) to protect them from hot combustion gases. Cooling holes for the film cooling are typically produced after coating deposition by laser drilling. However, in case of additively manufactured components with more efficient cooling channels, subsequent coating processes should not block the sophisticated cooling structures, which is the case for conventional coating techniques such as Atmospheric Plasma Spraying (APS) and Electron Beam - Physical Vapor Deposition (EB-PVD). Therefore, new approaches must be developed to avoid the closing of the cooling holes during the deposition process. In this study Additively Layer Manufactured (ALM) substrates made of nickel base alloys with cooling holes were coated with CoNiCrAlY bond coats using the High-Velocity-Oxygen-Fuel (HVOF) process. Suspension Plasma Spraying (SPS) and Plasma Spray Physical Vapor Deposition (PS-PVD) were used afterward for YSZ topcoat deposition. The first experimental results revealing promising results in terms of avoiding cooling hole blockage. The surface characteristics of ALM parts and the effect of spray parameters on the coating microstructure like porosity, column density and blocking behavior will be discussed.

9:40 AM

(ICACC-S2-022-2024) Wear-resistant coatings for operation at 500 °C, obtained by vacuum-arc and magnetron sputtering of targets based on Ti(Nb, Cr,)-Al(Sn)-C MAX phases

T. Prikhna*[1]; O. Ostash[2]; O. Kuprin[3]; V. Podhurska[2]; B. Büchner[4]; D. Pohl[5]; P. Potapov[4]; T. Serbenyuk[1]; V. Sverdun[1]; V. Moshchil[1]; M. Karpets[6]; S. Ponomryov[7]

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2. Karpenko Physico-Mechanical Institute of the National Academy of Sciences of Ukraine, Ukraine
3. National Science Center Kharkov Institute of Physics and Technology, Ukraine
4. Leibniz-Institut für Festkörper- und Werkstoffforschung Dresden e. V., Germany
5. Dresden Center for Nanoanalysis (DCN), Technische Universität Dresden, Germany
6. National Technical University of Ukraine «Igor Sikorsky Kyiv Polytechnic Institute", Ukraine
7. Institute of Semiconductor Physics of the National Academy of Sciences of Ukraine, Ukraine

Many branches of industry interested in high temperature wear resistant coatings. For example, it is considered that further increase of efficiency of gas turbine aircraft engines can be attained via development of efficient high were resistant coatings on turbines blades of compressors and sliding mechanisms which are working in fritting corrosion modes at temperatures of 500 °C or higher. In the present study the composite coatings Ti(Nb, Cr,)-Al(Sn)-C 5-13 μm thick were deposited on heat resistant steel by vacuum-arc evaporation and magnetron spattering of 211- and 312-MAX phases targets. The coatings were polycrystalline (containing MAX phases) and amorphous. We studied friction coefficients and specific rate of wear of coatings in contact with steel at 25 and 500 °C, as well, as fritting corrosion at 25 °C. The specific wear rate parameter, v, of Ti-Al(Sn)-C coating with hardness 16.5 GPa at 0,5N load turned out to be almost 3 times lower at 500 °C for samples without coating ($v=6,1\times10^{-4}$ mm^3/N×m) than for sample with this coating ($W=1.94\times10^{-4}$ mm^3/N×m).

S11 Advanced Materials and Innovative Processing Ideas for Production Root Technologies

SYMPOSIUM 11: Sustainable energy concepts and applications

Room: Ponce de Leon

Session Chair: Chisung Ahn, Korea Institute of Industrial Technology

9:00 AM

(ICACC-S11-001-2024) Mechanical properties and self-healing mechanism of SiC dispersed $Yb_2Si_2O_7$ composite (Invited)

A. Okawa*[1]; S. T. Nguyen[2]; T. Nakayama[3]; T. Do[3]; H. Suematsu[3]; T. Goto[3]; K. Niihara[3]

1. Tohoku University, Institute of Multidisciplinary Research for Advanced Materials, Japan
2. National Institute of Technology Kushiro College, Department of Creative Engineering, Japan
3. Nagaoka University of Technology, Extreme Energy-Density Research Institute, Japan

This work describes the self-healing ability of SiC dispersed in $Yb_2Si_2O_7$. The $Yb_2Si_2O_7$/SiC samples with 5vol% and 10vol % of SiC were prepared by solid-state reaction and hot-press sintering at 1550°C for 1 h in Ar under 30 MPa. Scanning electron microscope observations, Vickers hardness measurements, and fracture toughness tests were carried out to evaluate the SiC distribution and mechanical properties of samples. To create cracks, a Vickers indentation was applied on the surface. The samples were then annealed at temperatures from 750°C to 1250°C for 1 h in air. X-ray diffraction (XRD) was used to characterize the crystallographic phase transformations before and after the annealings. Results showed that the strength recovery of 10vol% SiC sample continued up to 1150°C, whereas the 5vol% SiC sample showed the maximum strength at 1050°C. The fracture toughness was increased after the annealings, therefore compressive stress based on oxidation had a significant effect on strength recovery. SiC was still detected by XRD after annealing at 1150°C for 1 h, suggesting that it was not completely consumed. After annealings, the oxidation depth was affected by the amount of SiC, which led to the formation of compressive stresses. The obtained results suggest that an adequate addition of SiC can be an effective way to enhance the mechanical properties and extend the lifetime owing to self-healing.

9:30 AM

(ICACC-S11-002-2024) Valid Design of Cathode and Anode Materials for Aqueous Zinc Ion Batteries (Invited)

S. Mhin*[1]

1. Kyonggi University, Advanced Materials Engineering, Republic of Korea

There has been a growing interest in aqueous Zn-ion rechargeable batteries (AZIBs) as an alternative energy storage system to traditional Li-ion batteries. However, challenges such as dendrite formation on the Zn anode and instability of cathode materials have hampered their electrochemical performance, leading to limitations in rate capability and cycle stability. Also, the complexity of processing methods for cathode and anode materials has hindered their practical application. Therefore, it is required for a straightforward yet practical approach to improve the high-rate capability and cycle stability of AZIBs. In this study, we present a comprehensive examination of materials preparation and characterization for both cathodes and anodes, along with the associated processing methods and their impact on the electrochemical performance of ZIBs. Our focus is on the surface modification of the Zn anode to fine-tune its interaction with the electrolyte, addressing one of the key challenges in ZIB technology. The design of the proposed cathode and anode materials is not only straightforward and scalable but also holds significant promise for practical ZIBs with an extended lifecycle and enhanced safety features. This research offers valuable insights into the development of ZIBs, paving the way for more efficient and sustainable energy storage solutions.

S8 18th Intl Symp on APMT for Structural & Multifunctional Materials & Systems

SYMPOSIUM 8: Joining, integration, machining, repair, and refurbishment technologies

Room: Coquina F

Session Chair: Jerzy Lis, AGH University of Science and Technology

9:00 AM

(ICACC-S8-012-2024) Brazing of Ceramics and Ceramic Matrix Composites to Themselves and to Metals for High Temperature Structural Applications

M. C. Halbig*[1]; M. Singh[2]; R. Asthana[3]; A. S. Almansour[1]

1. NASA Glenn Research Center, USA
2. Ohio Aerospace Institute, USA
3. University of Wisconsin-Stout, Engineering and Technology, USA

Robust joining and integration solutions are essential for enabling the wider utilization of ceramic and ceramic matrix composite (CMC) components in aerospace applications. Joining and integration allows for the buildup of larger and more complex shaped ceramics and CMCs and their incorporation into metallic based systems such as support structures, thermal protection systems, manifolds and cooling systems, and multi-material and sub-element components. Brazing with active brazing alloy interlayers is a leading approach for ceramics and forming permanent joints with metals. However, each brazing solution must be specially tailored. In this presentation, considerations for materials selection, modified brazes, braze process optimization, material incompatibilities (e.g. chemical, thermal, and mechanical), and other requirements will be discussed. A brief overview of past brazing activities as well as current activities with silicon nitride and silicon carbide fiber/silicon carbide matrix CMCs (e.g. SiC$_f$/SiC$_m$) with emphasis on processing-microstructure-properties correlations and mechanical testing will be presented.

9:20 AM

(ICACC-S8-013-2024) Revisiting Wettability and Interfacial Phenomena in Processing and Joining of Ultra-High Temperature Ceramics

N. Sobczak[1]; R. Asthana*[2]

1. Institute of Metallurgy and Materials Science, Poland
2. University of Wisconsin-Stout, Engineering and Technology, USA

Refractory metal diborides and carbides are being developed for applications under extreme conditions. While these ultrahigh-temperature ceramics are generally processed in the solid state, liquid-phase processes such as infiltration and brazing also play a key role in manufacturing and joining of these materials. This paper revisits the scientific studies on high-temperature wettability of refractory metal diborides and carbides in context of liquid-phase processing and joining. The contact angle data were collected and critically evaluated vis-à-vis the structure of the interface. It was demonstrated that, during contact heating through melting and subsequent cooling, interactions that occur in the solid state and the liquid state govern the evolution of the contact angle and the formation of the interface. These interactions include solid-state diffusion; liquification and reaction product formation during heating; reactive wetting from substrate dissolution and reaction layer formation in the molten filler; and phase formation via dissolution-precipitation during cooling and solidification. Recent research on high-temperature wettability using an innovative capillary purification procedure that eliminates contact heating and erodes the surface oxide on liquid to yield pristine interfaces is also described.

9:40 AM

(ICACC-S8-014-2024) Study of Metal Elements for Insert Materials in Liquid Phase Diffusion Bonding of SiC Ceramics

T. Ozaki*[1]; H. Tsuda[2]; S. Mori[2]

1. Osaka Research Institute of Industrial Science and Technology, Applied Material Chemistry, Japan
2. Osaka Metropolitan University, Japan

SiC ceramics have excellent oxidation resistance, thermal stability, and high high-temperature strength, making them promising materials for thermal structure applications such as aerospace components. In order to achieve wide application of SiC ceramics as high-temperature structural components, it is important to develop effective bonding techniques. We tested diffusion-bonding using insert metals on SiC fiber-bonded ceramics, and found that by performing the bonding process using a combination of Ti and Cu foils as the interlayer, the bonding reaction progresses through the liquid phase. It was discovered that so-called transient liquid phase bonding (TLP bonding) was developed and a strong bond was obtained. In order to understand the mechanism of TLP bonding in SiC ceramics, it is important to explore metal elements other than Ti/Cu that cause TLP bonding and investigate their reactions. As a result of investigating the Ti/Cu-based joint structure, it has been found that a cermet-like structure is formed in which TiC particles, which are carbides, are precipitated in the matrix of copper or copper alloy. Therefore, an element that melts into metallic copper at a relatively low temperature and forms carbides should be a candidate. We fabricated bonded body by hot-pressing SiC ceramics using a combination of Hf and Cu foils as insert metals, and investigated the bonded interface.

S15 8th International Symposium on Additive Manufacturing and 3-D Printing Technologies

SYMPOSIUM 15: Additive Manufacturing Processing, Characterization, and Applications II

Room: Coquina H
Session Chair: Giorgia Franchin, University of Padova

10:20 AM

(ICACC-S15-016-2024) Additive Manufacturing – versatile but challenging process (Invited)

D. B. Kata*[1]; P. Rutkowski[1]; J. Lis[1]

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

Additive manufacturing (AM), which is also called three-dimensional (3D) printing is constantly and rapidly expanding. With structural and functional applications, 3D printing is widely used across the whole spectrum of industrial applications. Because of its relatively low cost, universal applicability and potential to reduce loss of raw material, it has started to be recognized as eco-friendly production methods, especially for ceramic and metal-based materials. The applicability of AM is shown by appropriate assessment of two systems, (1) AlN-graphene nanocomposite; (2) Inconel 625 – WC composite. The anisotropic AlN-graphene nanocomposites were shaped by AM technology. It is shown that additive manufacturing significantly influenced thermal anisotropy and microstructure appearance. The possibility of controlling anisotropy by graphene content and AM conditions is shown. Based on the example of thermal conductivity, the crucial parameters of aluminium nitride applicability are shown. Chemically and structurally homogenous Inconel 625 – WC composite coatings were prepared by Additive Manufacturing. Thus new class of coatings were implemented to high power turbined blades for improving their efficiency. Some new examples of AM technology for preparation high quality metal-ceramic composites are showed.

10:50 AM

(ICACC-S15-017-2024) Highly transparent/translucent polycrystalline ceramics made by SLA 3D printing

R. Svintsitski*[1]; R.Gaignon[1]; A. Roux[1]; M. Bourjol[1]; E. Louradour[1]

1. 3DCERAM SINTO, France

Alumina, spinel, zirconia transparent/translucent ceramics are appealing for many industrial sectors due to their specific properties: optical, mechanical, high melting point, high chemical inertness. These characteristics meet interests in many application fields as: lasers, vapor lamps, brackets for orthodontic brace, armored windows, sophisticated lens, catalysis… The growing demand for complex shaped products combined with the current transparent materials request make 3D printing ceramic the ideal technique to answer this need and confirm its industrial deployment as a new standard shaping process. As a result, 3DCeram is regularly solicited for the qualification and industrialization of its 3D printing processes for the production of transparent ceramics. The studies of two materials (alumina and spinel) from industrial requests will be presented in this presentation. The specificities of the raw materials, the shaping method but also the different heat treatments applied will be explained, as well as the best results obtained.

11:10 AM

(ICACC-S15-018-2024) Stereolithography 3D Printing of Complex Al_2O_3 Heat Exchangers

C. Weatherstone*[1]; T. Shoulders[1]; M. Kauf[1]; R. Manglik[2]

1. Technology Assessment and Transfer, USA
2. University of Cincinnati, Mechanical & Materials Engineering, USA

High-performance of Al_2O_3 heat exchangers have been fabricated by stereolithography additive manufacturing. To expand thermal-hydraulic performance, heat exchanger designs with enhanced features including offset-strip fins and wavy or corrugated fins were targeted for their exceptional heat transfer and low flow friction penalty. The additive manufacturing protocols were optimized (alumina resin rheology, build parameters, cleaning procedure, binder burn out, and sintering) for these advanced heat exchanger designs. These design and process changes allow the final product to meet 0.002in tolerances despite a 22-26% shrinkage. The resulting gaps between walls are as small as 0.5mm. The performance of 3D printed Al_2O_3 heat exchangers with advanced fin designs were measured in a unique setup at the University of Cincinnati. The simple uni-directional flow heat exchangers have since been modified for further performance improvements by the addition of cross-flow geometry with gas to gas and liquid to gas designs. Ceramic heat exchangers with the above-mentioned designs were evaluated and compared with modeling predictions and metal heat exchangers of same design. The results demonstrate consistent performance markers between materials, with the slight advantage going to 3D printed ceramics for enhanced temperature/environmental stability and the potential for finer features in ceramics.

11:30 AM

(ICACC-S15-019-2024) Additive Manufacturing of silicon carbide and boron carbide by means of CerAM technologies and possibilities of laser water jet cutting thereof

J. Abel*[1]; B. Matthey[2]; C. Berger[1]; U. Scheithauer[3]; T. Moritz[4]; M. Herrmann[5]

1. IKTS, Additive and Hybrid Manufacturing, Germany
2. Fraunhofer IKTS, Sintering and characterization, Germany
3. Fraunhofer IKTS, Shaping, Germany
4. Fraunhofer IKTS, Processes/Components, Germany
5. Fraunhofer IKTS, Germany

For more than 10 years, Additive Manufacturing (AM) of technical ceramics of complex and individualized components has been making its way into the landscape of modern production. Due to the property portfolio of ceramics like a high Young's modulus, high hardness and strength, these materials are known to be suitable for technical components but also for protective equipment and armor in defense sector. Of particular interest are lightweight equipment with high hardness, such as SiC and B_4C material for passive body protection. Additive manufacturing methods for the production of large-volume components, such as powder-based Binder Jetting Technology (BJT) and material-extrusion-based Fused Filament Fabrication (FFF), are used for this purpose. In addition, composite materials with e.g., diamond are developed as super hard materials within the framework of materials research and used in parallel in BJT as well as in FFF. Due to the high hardness of those ceramics, AM is particularly attractive since mechanical machining is almost impossible beside geomatrical advantages. The presentation will give an overview about the AM capabilities at IKTS, results as well as first approaches to cut super hard diamondlike materials by an innovative combined laser water jet cutting system.

11:50 AM

(ICACC-S15-020-2024) W-Cu complex structures obtained by investment casting into SiO_2-based moulds produced by stereolithography

G. Bianchi*[1]

1. SUPSI, MEMTi, Switzerland

A new indirect additive manufacturing technique is proposed to produce tungsten-copper (W-Cu) triply periodic minimal surface (TPMS) geometries by investment casting. The process combines stereolithography (SLA) and melt copper casting to realize complex architectures (i.e., gyroids) and to control the volume fractions of W and Cu, tailoring the composite properties. SiO_2-based moulds were produced by SLA to reproduce the negative of the object to be casted. The positive shape of the final object was obtained by filling the SiO_2 mould with a mixture of tungsten and corn starch (CS) powders, which was used as space holder. After a pyrolysis stage, the W porous structure was infiltrated by melted copper under vacuum. The leaching of the SiO_2 mould using a basic solution allowed for obtaining the final object. The produced samples retained the geometric properties of the CAD design.

S6 Advanced Materials and Technologies for Rechargeable Energy Storage

SYMPOSIUM 6: Solid electrolytes for batteries II

Room: Ballroom 5
Session Chairs: Mahalingam Balasubramanian, Oak Ridge National Lab; Robert Dominko, National Institute of Chemistry

10:20 AM

(ICACC-S6-012-2024) Solid-state NMR spectroscopy as a direct tool to monitor fast ion dynamics in materials for new energy storage systems (Invited)

M. Wilkening*[1]

1. Graz University of Technology, Chemistry, Austria

Fast, if not ultrafast, ion dynamics is a basic prerequisite for both electrolytes and active materials in ceramic batteries. Nuclear magnetic resonance serves as a direct tool to probe the irregular movement of the ions by taking advantage of the interactions of the spin-carrying ions with internal dipolar magnetic and quadrupole electric field fluctuations. In particular, nuclei-specific NMR relaxation experiments help us understand the dynamic features not only in materials with one mobile ion, it is also possible to separately study ionic motion in materials with two or more mobile species. By considering recent results on garnet-type oxides (LLZO, LLZTO (1,2)) and lithium titanate ($Li_4Ti_5O_{12}$, LTO (3)) with mixed cations, that is, Li and H in protonated LLZTO and Li and Na in sodiated LTO, the talk is giving an overview of the powerfullness of NMR relaxation measurements to probe Li as well as Na and H ion dynamics. (1) P. Posch, S. Lunghammer, S. Berendts, S. Ganschow, G.J. Redhammer, A. Wilkening, M. Lerch, B. Gadermaier, D. Rettenwander, H.M.R. Wilkening, Energy Storage Mater. 24, 2020, 20. (2) M. Gombotz, C. Hiebl, A. Wilkening, F. Stainer, H.M.R. Wilkening, J. Phys. Chem. C127, 2023, 10960. (3) P. Posch, S. Lunghammer, A. Wilkening, K. Hogrefe, H.M.R Wilkening, J. Phys.: Energy 5, 2023, 015001.

10:50 AM

(ICACC-S6-013-2024) Design strategies in multifunctional structural battery electrolytes (Invited)

D. Fam*[1]; D. Safanama[1]; J. Lim[1]; S. Goh[1]; N. Ding[1]; M. Tan[1]; S. Chien[1]; J. Cheong[1]

1. Institute of Materials Research and Engineering, Singapore

Electrification in transportation brings environmental benefits but comes with significant challenges in terms of the added weight and space that batteries take up. Multifunctional structural batteries that combine the functionalities of mechanical robustness and electrochemical energy storage properties aim to address these challenges. However, the development of multifunctional constituents for structural batteries is complex due to the conflicting material requirements for electrochemical energy storage and structural stability. In particular, the development of structural battery electrolytes is especially challenging considering ion conduction properties, electrochemical and mechanical stability, and processibility. In our work, we uncover non-saliant factors such as crystallisation kinetics and composition in developing design strategies for various structural battery electrolytes that possess good ionic conductivities below their melting points. We envision that these new structural battery electrolytes can pave the way towards the genesis of a new class of cheaper and easily processed solid state batteries.

11:20 AM

(ICACC-S6-014-2024) Enhanced Interfacial Stability of Sulfide Solid Electrolyte/ Li-metal Anode by N-GQD Coating

Y. Cho*[1]; D. Kim[1]; D. Kim[1]

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

Solid electrolytes have been regarded as an alternative to liquid electrolytes due to their better safety and higher energy density. Among the various solid electrolytes, sulfide solid electrolytes (SSE) show relatively high ionic conductivity and ductility. Sulfide solid electrolytes still suffer from serious side effects at the interface with Li-metal. Li dendrite and the resistive layer (Li_2S, Li_3P, LiCl) are formed at the interface of SSE and lithium metal. Two typical approaches were attempted to improve the compatibility between Li-metal and sulfide solid electrolyte. One is the element substitution of electrolyte, and the other is the introduction of an intermediate buffer layer. In this study, the interfacial stability of argyrodite-based sulfide electrolyte (Li_6PS_5Cl) with Li-metal anode was studied by the introduction of the nitrogen doped graphene quantum dot (N-GQD) coating. Ex-situ FT-IR, Raman, and XPS analyses were carried out to characterize N-GQDs. N-GQD layer enabled an uniform lithium stripping/plating and also effectively suppressed the lithium dendrites. The electrochemical performance of the symmetric cells with N-GQD were evaluated at a constant current density.

11:40 AM

(ICACC-S6-015-2024) 3D printing of $Li_{1.5}Al_{0.5}Ge_{1.5}P_3O_{12}$ electrolytes for All Solid Sate Lithium Batteries

A. Sabato*[1]; D. Ferreira[1]; A. Pesce[2]; M. Nuñez Eroles[1]; M. Casas-Cabanas[2]; P. Lopez-Aranguren[2]; M. Torrell[1]; A. Morata[3]; A. Tarancón[4]

1. IREC, Nanoionics and Fuel Cells, Spain
2. Center for Cooperative Research on Alternative Energies (CIC energiGUNE), Basque Research and Technology Alliance (BRTA), Electrochemical Energy Storage, Spain
3. Catalonia Institute for Energy Research (IREC), Nanoionics and Fuel Cells, Spain
4. IREC / ICREA, Spain

Many efforts in the field of Lithium batteries are focusing on the development and implementation of solid electrolytes in order to overcome the drawbacks related to the liquid ones. Li-conducting glass-based NASICON materials gained attention in the last years as solid state electrolytes, thanks to their good ionic conductivity at room temperature (10^{-3}-10^{-4} S cm^{-1}) and their wide electrochemical stability windows. Here we present concepts and results concerning disruptive ceramic 3D printing applied to full ceramic $Li_{1.5}Al_{0.5}Ge_{1.5}P_3O_{12}$ (LAGP) electrolytes. The use of 3D printing allowed the fabrication of simple as well as complex architectures, with enhanced contact area with the electrodes. The inks and the printing processes were both optimized in order to reach an accuracy up to ≈100 μm. The use of 3D printing allowed the production of 100 μm thick membranes with both flat and corrugated geometry. The corrugated shapes allowed the reduction of area specific resistance up to 20%. The printed material demonstrated a conductivity in line with conventional manufacturing techniques reported in literature (> 10^{-4} S cm^{-1} at RT). The produced electrolytes were tested also in plating-stripping cycles vs. metallic Li. The corrugation improved the behaviour of the electrolyte leading to a lower overpotential (35 mV) during 1000h of cycling in standard conditions (0.1 mA cm^{-2}, 2 h per cycle).

13th Global Young Investigator Forum

13th Global Young Investigator Forum: Design and processing

Room: Coquina D

Session Chairs: Oriol Gavalda Diaz, Imperial College; Qiance Zhang, University of Bristol

10:20 AM

(ICACC-GYIF-013-2024) Inkjet printing of cobalt ferrite and sodium potassium niobate films for magnetic and piezoelectric applications in the microelectronics (Invited)

M. Mariani*[1]; R. Bernasconi[2]; F. Maspero[3]; C. Galassi[1]; L. Magagnin[2]; N. Lecis[1]

1. Politecnico di Milano, Mechanical Engineering, Italy
2. Politecnico di Milano, Chemistry, Materials and Chemical Engineering “Giulio Natta”, Italy
3. Politecnico di Milano, Physics, Italy

Inkjet printing is a versatile additive manufacturing technology that can be used to create magnetic and piezoelectric films required in several applications, including sensors, actuators, biomedical devices and energy harvesters. This technique offers the potential for multimaterial direct-write fabrication, which eliminates the need for complex lithography or etching processes, in the production of MEMS with non-conventional designs. In this work the preparation of stable colloidal suspensions for jetting is studied and followed by the optimization of the printing conditions and thermal treatments for both $CoFe_2O_4$ and $(K_{0.5}Na_{0.5})NbO_3$. The printed and densified films are studied from a microstructural perspective using microscopy, surface roughness analysis, and x-ray diffractometry to determine the influence of the printing parameters and phase composition. In the case of cobalt ferrite, vibrating sample magnetometry is performed to assess the coercivity and saturation magnetization values by applying an external magnetic field from -2 T to 2 T both for the in-plane (IP) and the out-of-plane (OP) configurations. A full characterization of the niobate was performed using multiple setups to determine its piezoelectric behavior.

10:50 AM

(ICACC-GYIF-014-2024) Thermal engineering using photonic structures (Invited)

S. Shin*[1]

1. National University of Singapore, Department of Mechanical Engineering, Singapore

Control of thermal transport is of significant interest for a wide range of applications, such as thermoregulation of individuals, buildings, vehicles and batteries, thermo-electric and solar-thermal energy conversion, bio/chemical sensing, and micro/nanomanufacturing. However, heat transfer processes are often difficult to actively control: heat conduction is usually diffusive in nature owing to the incoherence of heat carriers (phonons and electrons) and thermal radiation is generally broadband or have wide energy distribution. If one could engineer the transport of thermal energy, arguably the most ubiquitous form of energy, with similar degree of controllability as electrical and optical energy, a variety of energy transport and conversion technologies can be improved or even revolutionized. In this talk, I will introduce a thermo-photonic engineering approach to manipulate nanoscale heat transport by using surface phonon polaritons (SPhP). I will mainly focus on how the SPhP can be utilized to tailor thermal radiation properties, especially to achieve a coherent, near-monochromatic far-field thermal emission, which is a big departure from the incandescent behaviour in the classic textbook as described by the Planck's law. I will show my experimental and theoretical work to quantify the far-field thermal radiation from these rationally-designed single nano-emitters.

11:20 AM

(ICACC-GYIF-015-2024) Phases, microstructures, and ionic conductivity of high-entropy Li-garnet

Z. Fu*[1]; S. Budur[1]

1. Penn State Harrisburg, USA

Ceramic-based solid-state electrolytes in solid-state batteries for energy storage have attracted much attention. High-entropy ceramics show promising properties in various fields. Here, we designed and fabricated a series of $Li_7La_3Zr_2O_{12}$ (LLZO) based high-entropy Li-garnet solid-state electrolytes. With properly designed chemical formulas, we studied the effects of several key factors, mainly defect energy and atomic radius of the substituting cations on the La and Zr sites on the phases, microstructures, and ionic conductivity. We synthesized a series of single-phase cubic Li-garnets with promising ionic conductivities (up to ~4.7 mS/cm). Samples with some secondary phases show significantly lower ionic conductivities, as expected. Microstructure observations reveal the effects of the substituting elements on the element distributions, grain structures, and grain boundary conditions. Correlations between the phases, microstructure, and ionic conductivity (bulk and grain boundary) are also studied and summarized.

11:40 AM

(ICACC-GYIF-016-2024) Scalable strategies for the recovery of ceramic materials from solid oxide cells (SOCs) and their re-manufacturing

S. Saffirio*[1]; S. Anelli[1]; D. Ferrero[2]; I. Schiavi[3]; M. Santarelli[2]; S. Pylypko[4]; F. Smeacetto[1]; S. Fiorilli[1]

1. Politecnico di Torino, Department of Applied Science and Technology, Italy
2. Politecnico di Torino, DENERG, Department of Energy, Politecnico di Torino, Italy
3. Environment Park, Turin, Italy
4. Elcogen, Estonia

As the SOCs market is foreseen to remarkably increase in the near future, the recovery and re-use of critical and high-cost materials is crucial to enable the large-scale production and the overall sustainability of SOC-based technologies. In this regard, we hereby present a single-step recycling process optimized for the Ni-YSZ electrode components of end-of-life (EoL) SOCs. Thanks to its adaptation from the bench scale to a larger scale, a 10-fold increase in the amount of processed powders was achieved, enabling to target the quantity required for the manufacturing of new SOCs containing 30 wt% of recycled material. The disaggregation of the Ni-YSZ composite and the selective extraction of Ni (recovered in the form of NiO) were achieved by treating the cell components inside a hydrothermal reactor in presence of low-concentrated (0.6-1.0 M) HNO_3 solutions, thus combining hydrothermal treatment and acid leaching. The processing parameters – including temperature, time, acid concentration and solid/liquid ratio – were optimized considering their mutual interactions to maximize the overall efficiency of the process, therefore minimizing waste flows, energy consumptions and costs. Re-manufactured cells containing 30 wt% of recovered ceramic powders were electrochemically and mechanically characterized for a comparison with standard virgin SOCs.

S2 Advanced Ceramic Coatings for Structural/Environmental & Functional Applications

SYMPOSIUM 2: Environmental Barrier Coatings I

Room: Flagler C
Session Chair: Kang Lee, NASA Glenn Research Center

10:20 AM

(ICACC-S2-015-2024) Xenotime-based Minerals as Environmental Barrier Coatings

E. Opila*[1]; P. Stack[1]

1. University of Virginia, USA

Xenotime minerals are rare earth (RE) phosphates of the type $REPO_4$ where RE is found to be primarily Y, with minor amounts of Gd, Dy, Er, Yb in approximate proportion to the elemental natural abundance in the earth. Prior work demonstrated that $YbPO_4$ had necessary properties for a viable environmental barrier coating (EBC) commensurate with state-of-the art $Yb_2Si_2O_7$. An additional developing focus of research is exploration of multicomponent rare earth silicates with reduced thermal conductivities attributed to phonon scattering of RE with varying mass and size, enabling EBCs to also act as thermal barriers or T/EBCs. Multicomponent rare earth compositions are typically synthesized via combinations of high purity single component rare earth silicates. However, rare earth separation into high purity single components is costly, slow, and generates significant amounts of waste. Thus, motivation exists to use multicomponent rare earth minerals such as xenotime directly without the separation and recombination of RE. In this work, properties of naturally occurring xenotime minerals, individual $REPO_4$ (RE=Y, Gd, Dy, Er, Yb), synthetic xenotime, and equimolar (Y, Gd, Dy, Er, Yb)PO_4 are explored to evaluate EBC properties. Phase purity, thermal expansion, thermal conductivity, high temperature steam resistance, and CMAS resistance were evaluated. Efficacy of the proposed xenotime mineral as T/EBCs is demonstrated.

10:40 AM

(ICACC-S2-016-2024) Quantitative microstructural analysis techniques in YbDS environmental barrier coatings

V. Mika*[1]; D. Smith[2]; G. Harrington[6]; R. A. Golden[3]; J. Shi[5]; M. Titus[1]; R. Trice[4]

1. Purdue University, Materials Science and Engineering, USA
2. Northwestern University, Materials Science and Engineering, USA
3. University of Virginia, Materials Science and Engineering, USA
4. Purdue University, Department of Materials Engineering, USA
5. Imperial College London, United Kingdom
6. Missouri University of Science & Technology, USA

Ytterbium disilicate (YbDS) environmental barrier coatings are crucial to the protection of ceramic matrix composite components in gas turbine engines from corrosive effects at working temperatures and atmospheres. When applied using atmospheric plasma spray, the coatings form a multitude of amorphous, crystalline, stable and metastable phases. In this study we investigate the effectiveness of XRD and Raman spectroscopy in determining phase contents of YbDS coatings, and possible biases that can arise. Raman spectroscopy and XRD were used to analyze the microstructure of

YbDS coatings, and industry data was investigated using the same techniques. The results suggest a correlation can be drawn between phase contents received through XRD and Raman spectroscopy analysis, but that special care must be taken during sample preparation and data review.

11:00 AM

(ICACC-S2-017-2024) Exploring aspects related to the search for new T/EBCs

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

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

Greater gas turbine engine efficiency is a major goal in aeronautics research often pursued through increased engine operating temperatures. However, it is necessary to replace the current hot-stage alloy components with more thermally robust parts, such as Silicon-based ceramics and composites. Unfortunately, these materials are still susceptible to the effects of oxidation, water vapor, and (Calcium-Magnesium-Alumino-Silicate) CMAS interaction, among other issues at high temperature. To mitigate these effects, thermal and environmental barrier coating (T/EBC) materials are employed to help control the rate of degradation to the underlying composite, but must also survive the corrosive environment. The results of CMAS and water vapor exposure to novel tantalates, aluminates, and gallates will be presented with several compositions showing promise for use as a T/EBC.

11:20 AM

(ICACC-S2-018-2024) Performance of EB-PVD Y-based EBC system under high temperature water vapor environment

C. Y. Guijosa Garcia*[1]; U. Schulz[1]; R. Naraparaju[1]

1. DLR - German Aerospace Center, Institute of Materials Research, Germany

Environmental barrier coatings (EBCs) based on RE2Si2O7 (REDS) are used to enhance the durability of ceramic matrix composites (CMC) in high-pressure, high gas, and velocity combustion atmospheres. While the use of RE-monosilicates (REMS), REDS, or multi-component for EBC or T/EBC is still under debate, efforts are required to produce dense, uniform, crack-free layers that have good adherence with the Si-bond coat. Atmospherically plasma spray (APS) provides a relatively dense coating, however, most REDS sprayed lose silica and lead to the presence of REDS and REMS mixture. Also, the CMC substrate requires grit blasting, which can damage the matrix and/or fibers. Physical vapor deposition, e.g., electron beam physical vapor deposition (EB-PVD), can provide good control of chemistry, adhesion, microstructure to improve the accommodation of CTE, without grit blasting the CMC, and the feasibility to coat the sharp edges. This study aims to deposit double layer YMS/YDS on SiC substrate using EB-PVD and evaluate their performance under water vapor (30% mass) at 1350 and 1400°C for longer times. Results have shown that EB-PVD technique offers the thick deposition of EBC system. The changes after the water vapor test will be discussed in terms of morphology, crystalline phase, and chemistry of the coating.

S11 Advanced Materials and Innovative Processing Ideas for Production Root Technologies

SYMPOSIUM 11: Coating, forming and shaping processes for industrial applications

Room: Ponce de Leon
Session Chair: Sungwook Mhin, Kyonggi University

10:20 AM

(ICACC-S11-003-2024) The Promising Progress of 2D Materials as a Sustainable Solid Lubricant (Invited)

A. Sumant*[1]

1. Argonne National Lab, Center for Nanoscale Materials, USA

There has been a great interest in developing a new class of solid lubricants based on graphene and other two-dimensional materials. These remarkable materials have played a pivotal role in achieving exceptionally low levels of wear and ultra-low friction, extending their influence to the remarkable phenomenon of superlubricity, even on a macroscopic scale. Their exceptional performance in minimizing wear and friction across a diverse range of systems has captivated researchers and engineers alike. What truly captivates our scientific and technological curiosity is the unique capacity of these two-dimensional materials to function as semi-liquids, giving rise to stable tribolayers with intricate tribochemical properties. From both scientific and practical standpoints, this feature is incredibly intriguing. I'll discuss the role of tribochemistry at the micro/nanoscale and how it impacts tribological performance at the macroscale with demonstrated examples that we have worked on in collaboration with industry, particularly in the automotive industry for metal stamping applications. This sets the stage for future breakthroughs and the realization of oil-free superlubricity in real-world applications. In doing so, it contributes significantly to the broader mission of decarbonization within the lubrication industry.

10:50 AM

(ICACC-S11-004-2024) Design and Demonstration of Metal Wire DED Type Metal 3D Printer System Incorporating Robot and Metaverse (Invited)

T. Nakayama*[1]; W. Mita[1]; M. Iwama[1]; H. Hiraga[1]; Y. Miyashita[1]; M. Ito[2]; H. Suematsu[3]; K. Niihara[1]

1. Nagaoka University of Technology, Japan
2. IMI inc., Japan
3. Nagaoka University of Technology, Extreme Energy-Density Research Institute, Japan

The promotion of DX in manufacturing is one of the most important initiatives in root technology. In particular, there is a need for manufacturing that does not require face-to-face work and rapid manufacturing methods in corona disasters. In this paper, we report on a new metal 3D printer system that incorporates a robot and a metaverse. The metal wire 3D printer system uses DED (Directed Energy Deposition). In this system, 1.5 mm diameter metal wire is used as the raw material. The metal 3D printer is mounted on the end of a robot arm. The base plate for modeling is also mounted on the 6-axis robot, allowing it to move freely. The accuracy of the modeling object is about 1 mm. Therefore, this method always requires post-processing of the surface of the molding object. This post-processing is also performed in conjunction with a robot system. The object formed on the base plate is attached to another robot, which first inserts its hand into the cutting machine to cut the surface, and then inserts its hand into the wire EDM machine to separate the object from the base plate. Furthermore, the interior of the room where these series of devices are installed is all scanned by LiDAR to construct a metaverse space. Some of the processes can be performed interactively in real space by giving instructions from the metaverse space.

11:20 AM

(ICACC-S11-005-2024) Experimental Study on Optimization of Plasma Process for High Density Ceramic Slurry Manufacturing

C. Ahn*[2]; B. Nah[1]

1. Hanyang University, Division of Materials Science & Engineering, Republic of Korea
2. Korea Institute of Industrial Technology, Heat & Surface Technology R&D Department, Republic of Korea

As one of the key element technologies constituting the ceramic material based additive manufacturing (3D printing) process, the various kinds of customizing studies has been performed to optimize the form of raw materials supplied to the feeding tool. In particular, securing a platform capable of processing feed materials into a high-density slurry form have considered as an important issues because it is one of the practical ways to minimize shrinkage and deformation during post-heat treatment process required on printed structure which comprise by ceramic materials. Meanwhile, forming a functional group on the particle surfaces by physical process such as ball mill is applying mainly as a pretreatment step to enhance the powder content in the slurry. However, there is a limitation to establish the RM (Rapid Manufacturing) system significantly for ceramic 3D printing process due to the disadvantage that it takes a long time and may occur powder shape deformation. In this study, the innovative plasma process is proposed to increase hydroxyl functional groups (OH^-) efficiently inside slurry which composed of alumina powders (D: 300 nm) and deionized water resulting in high dispersibility and particle contents. Additionally, the tendency of slurry dispersion by plasma process variables is observed, and the optimal conditions are discussed to maximize the powder content in slurry.

11:40 AM

(ICACC-S11-006-2024) Flexible Laser Processing of SiC-CMC by Hybrid ArF Excimer Laser

T. Onose*[1]; Y. Kamba[1]; O. Konda[1]; H. Motosugi[1]; T. Miura[1]

1. Gigaphoton Inc., Research & Development Div., Japan

Deep ultra-violet (DUV) lasers, in the spectral range of around 200 nm to 400 nm, are attracting attention to laser micromachining, since, due to its high photon energy, DUV light can cut atomic and molecular bonds in various wide bandgap materials directly and reduce thermal affects. Hybrid ArF excimer laser, consisting of a solid-state laser including high harmonics generation process as a seed light source and an ArF excimer amplifier, can generate DUV light at the wavelength of 193 nm with several millijoules pulse energy. Since the ArF photon (6.4 eV) can cut the Si-C bond (3.2 eV) directly, no cooling gases or fluids were required. In addition, using galvanometer scanner system, one can make flexible shaped laser processing. We demonstrated 20° diagonal holes on 2-mm-thick SiC ceramics matrix composite (CMC) plate. Thanks to the high beam quality of hybrid ArF laser, one can make diagonal holes with steep angle. The effective thickness of SiC plate in case of the 20° diagonal hole drilling is 5.5 mm. Since the exit shaping to the film holes is the key technology of film cooling of gas turbine engines with greater surface coverage, we also made a shaped diagonal hole on SiC-CMC. The hole shape of input side was 1 mm squared, and the output side was circular shape. The diagonal angle was also 20°. Total processing time was 249 seconds. Details of laser processing and quality of drilled hole will be reported.

S13 Development & Applications of Adv Ceramics & Composites for Nuclear Fission/ Fusion Energy Sys

SYMPOSIUM 13: Advanced characterization techniques and methods

Room: Ballroom 4
Session Chair: David Sprouster, Brookhaven National Laboratory

10:20 AM

(ICACC-S13-013-2024) Neutron Scattering Analysis of Nuclear Materials (Invited)

E. O'Quinn*[1]

1. The University of Tennessee, Knoxville, Nuclear Engineering, USA

A major obstacle to progress in nuclear technology is the fabrication of durable materials that can withstand the extremes that operating conditions entail (e.g., high-temperature and energetic particle irradiation). Nuclear materials can structurally degrade in a variety of ways, ranging from accumulation of point defects to a complete loss of crystallinity. In order to better understand how materials are modified these effects must be comprehensively analyzed, which is challenging. For example, techniques such as X-ray diffraction have limited sensitivity to the local defect structure and low-Z elements. This presentation summarizes recent efforts of characterizing defective nuclear materials by means of neutron total scattering experiments. The key to this research strategy is the use of neutron probes from the world's most intense Spallation Neutron Source at Oak Ridge National Laboratory that are coupled with highly energetic ions from the GSI Helmholtz Centre for Heavy Ion Research (Darmstadt). Neutron total scattering overcomes many limitations of other techniques as shown by several case studies, covering the topics of nuclear fuels and wasteforms. New insights are provided into the atomic-scale oxygen clustering of oxidized UO_2, the amorphization process of crystalline ceramics, and the short-range order of nuclear waste glasses.

10:50 AM

(ICACC-S13-014-2024) Nondestructive Examination of AGR TRISO Particles and Compacts Using X-ray Computed Tomography (Invited)

W. Chuirazzi*[1]; R. Kancharla[1]; J. Stempien[1]

1. Idaho National Lab, USA

Next-generation nuclear reactors, a crucial emerging energy source to meet growing worldwide energy demand, require materials that can withstand the harsh environments of a reactor core. Certain next-generation reactor designs utilize tristructral isotropic (TRISO) coated fuel particles to generate power. However, these ceramic particles must be studied extensively for qualification before approval for commercial use. This work showcases how micro X-ray computed tomography (XCT) has been used to nondestructively examine irradiated TRISO fuel particles and compacts from the Advanced Gas Reactor (AGR) program. By providing quantitative data from XCT datasets, this nondestructive technique can be used to complement traditional destructive analyses by enabling targeted destructive analysis as well as providing input for computational models. In this talk we give an overview of previous work on irradiated AGR-3/4 compacts and AGR-5/6/7 particles, include imaging and post-processing technique development. These methods allow for nondestructive examination of both the TRISO particle fissile fuel kernel and coating layers. We conclude this presentation by presenting recent XCT results on specimens from the AGR program.

11:20 AM

(ICACC-S13-015-2024) Deformation of a SiC-based nuclear-fuel cladding under C-ring compression at 1200 °C with X-ray tomography imaging

G. Yuan*[1]; P. Forna-Kreutzer[2]; J. Ell[3]; H. Barnard[3]; S. Gonderman[4]; C. Deck[5]; E. J. Lahoda[6]; R. O. Ritchie[3]; D. Liu[2]

1. University of Bristol, Physics, United Kingdom
2. University of Bristol, United Kingdom
3. Lawrence Berkeley National Laboratory, USA
4. General Atomics, NTM, USA
5. General Atomics, USA
6. Westinghouse Electric Company, USA

Compared with Zircaloy, SiC fibre reinforced SiC matrix composites (SiC_f-SiC_m) have improved performance and safety in loss of coolant accident condition. And thus been considered as candidate accident tolerant fuel cladding for light water reactors. Various toughening mechanisms, e.g., crack deflection in outer coating, crack deflection and bifurcation in matrix and fibre bundles, and fibre pull-out occur in SiC_f-SiC_m under deformation. These mechanisms drive overall material performance. Applying in situ microstructural characterization techniques enables a comprehensive insight into SiC_f-SiC_m's failure processes, which is critical to optimize performance of SiC_f-SiC_m for nuclear applications. In this work, crack initiation and propagation in a SiC_f-SiC_m cladding under load was studied at RT and 1200°C. The material has a ~600 μm thick composite layer and a ~260 μm final outer chemical vapor deposited (CVD) SiC coating layer, deposited by several thinner SiC layers in series. In situ synchrotron X-ray micro-computed tomography (XCT) imaging with C-ring compression was applied. The hoop strength was derived at both temperatures. Before XCT experiments, local property and residual stress were estimated by nanoindentation and Raman spectroscopy. The results are discussed of the correlation between microstructures and macro-scale performance of the SiC_f-SiC_m material.

11:40 AM

(ICACC-S13-016-2024) Multiscale, Multimodal Characterization and Safety Testing of Silicon Carbide Cladding

P. Xu*[1]

1. Idaho National Lab, USA

SiC is being pursued as one of the ATF cladding candidate materials for light water reactors (LWRs). It has excellent high-temperature oxidation properties and superior irradiation resistance. However, SiC exhibits nonlinear mechanical behavior governed by microcracking under different conditions, such as the stress-state induced by irradiation, and PCMI under accident conditions. Morphology of defects is one of the key factors which impact cladding performance and safety. Therefore, quantification of defects and leak paths are critical to determining SiC cladding in-core performance. This research work provides qualitative insight into the defect distribution before and after safety tests in the Transient Test Reactor (TREAT). 3D X-ray imaging generates critical microstructural defect information, followed by advanced microstructure characterization using electron microscopy techniques. The automated workflow developed in this work enabled identification of the defects in the irradiated samples that were not present in the unirradiated sample. The statistics of the defects and characteristics provide information regarding the mechanical responses of SiC cladding under reactor accident conditions and can be later correlated to manufacturing for processing and performance improvement, as well as modeling and simulations. The plan for safety testing in TREAT will be outlined.

S18 Ultra-High Temperature Ceramics

SYMPOSIUM 18: Response in Extreme Environments

Room: Coquina A
Session Chairs: Yue Zhou, Missouri University of Science & Technology; Samuel Humphry-Baker, Imperial College London

10:20 AM

(ICACC-S18-013-2024) Tungsten diboride under thermal and irradiation damage (Invited)

S. A. Humphry-Baker*[1]; J. Davidson[1]; T. Zagyva[1]; M. Hasegawa[1]; X. Liu[1]

1. Imperial College London, Materials, United Kingdom

Tungsten diboride (WB_2) ceramics are candidate neutron shielding materials for fusion reactors. In this application the material will be exposed to high thermal and radiation fluxes, posing an extreme materials design challenge. This presentation will focus on recent progress in evaluating the properties of WB_2 ceramics reinforced with a minor volume fraction of free boron precipitates. The material shows an allotropic phase transformation at ~1000 °C that results in a volumetric expansion of ~0.05% and a corresponding localised decrease in the thermal conductivity at the transformation temperature. The flexural strength is also reported, we observed a small but monotonic increase up to ~1500 °C, followed by a brittle to ductile transition, and corresponding loss in strength, at a homologous temperature that is broadly in line with the literature on ZrB_2 ceramics. Indentation hardness anisotropy effects are also reported. Finally, we report the microstructure and hardness evolution of samples irradiated by helium ions at 300 and 700 °C. The irradiation induced hardening is correlated with bubble structures observed by transmission electron microscopy and structural defects characterised by grazing incidence X-ray diffraction. Materials design strategies including compositional and grain size effects will be discussed.

10:50 AM

(ICACC-S18-014-2024) Oxidation resistance of UHTCMCs up to 1700°C: Influence of Nb-coating (Invited)

A. Vinci*[1]; J. E. Förster[2]; D. Sciti[1]; R. Naraparaju[2]

1. CNR - ISSMC, Italy
2. DLR - German Aerospace Center, Institute of Materials Research, Germany

Ultra-High Temperature Ceramic Matrix Composites are a new class of materials that combine the good mechanical properties of CMCs with the oxidation and ablation resistance of UHTCs. Preliminary studies on the oxidation resistance of C-ZrB_2/SiC composites have shown the ability of these materials to survive harsh conditions with negligible erosion. The oxidation mechanism of the C-ZrB_2/SiC system involves the oxidation of the outer fibres and the formation of a borosilicate layer that provides a barrier against further oxidation up to 1650°C. Above this temperature, B_2O_3 actively evaporates, leaving behind a ZrO_2/SiO_2 layer that becomes unstable at higher temperatures. In order to improve the oxidation resistance of UHTCMCs and allow reusability of these materials, reactive coatings have been investigated to delay the oxidation kinetics. Thanks to the chemical compatibility between the coating and the UHTC substrate, these materials do not suffer from typical CMCs-related issues, such as coating spallation. In this work, niobium-based coatings were deposited on the surface of C-ZrB_2/SiC composites via magnetron sputtering. Two compositions with varying SiC amounts were investigated and oxidation was carried out from 1500 to 1700°C in air. The results show how 10 μm of Nb coating is already beneficial in stabilizing and preserving B_2O_3 even above 1650°C, considerably slowing down oxidation kinetics.

11:20 AM

(ICACC-S18-015-2024) Transforming UHTC Metal Ceramic Multilayer Composites for Hypersonics

J. C. Stotts*[1]; C. R. Weinberger[2]

1. Colorado State University, School of Advanced Materials Discovery, USA
2. Colorado State University, Department of Mechanical Engineering, USA

In this talk we will introduce metal ceramic multilayer composites with the ability to phase transform into a single phase ultra-high temperature ceramic. These materials offer enhanced low-temperature fracture toughness and reliability without sacrificing high-temperature mechanical properties by eliminating the metal reinforcement layers upon reaching temperatures sufficient for the activation of carbon diffusion. The presence of ductile metal layers increases the fracture toughness and reliability of the material making them less susceptible to catastrophic failure during component construction and assembly. At low temperatures, these materials are practically indefinitely stable because of the high activation energy for carbon diffusion in the carbide layers that control the phase transformation kinetics. Here, knowledge of the overall thermodynamics, i.e., carbon content, is combined with a coarse-grained model to predict phase transformation kinetics as well as toughening estimates in the limit of small-scale yielding to characterize the design space of these novel composites. It is proposed that the ability to control thermodynamic and mechanical properties of the post-transformation ceramic via selection of metal-to-ceramic layer ratios making these novel composites an excellent alternative to current methods for reinforcement of ultra-high temperature ceramics.

11:40 AM

(ICACC-S18-016-2024) The Response of ^{11}B Enriched ZrB_2 Ultra-high Temperature Ceramic to Neutron Irradiation at Elevated Temperatures

Y. Lin[1]; T. Koyanagi*[1]; D. Sprouster[2]; W. Fahrenholtz[3]; G. Hilmas[3]; Y. Katoh[1]

1. Oak Ridge National Laboratory, USA
2. Brookhaven National Laboratory, Nuclear Science and Technology, USA
3. Missouri University of Science & Technology, Dept. of Materials Science and Engineering, USA

ZrB_2 is an ultra-high temperature ceramic (UHTC) potentially useful in fusion reactors but little is known about its response to irradiation. A concern is the ^{10}B transmutation to helium will cause swelling and catastrophic cracking. We used advanced TEM and synchrotron XRD to investigate the microstructural evolution and lattice expansion of ^{11}B enriched ZrB_2 under mixed-spectrum neutron irradiation. Irradiation in HFIR at 220 and 560°C, to 2.2×10^{25} neutron/m^2 ($E > 0.1$ MeV) decreased the thermal diffusivity by a factor of 5-10. Cavities were observed in the 560°C irradiated samples, but not in the 220°C samples, while the 560°C samples had significantly fewer microcracks than the 220°C samples. X-ray diffraction showed expansion of the a-axis and contraction of the c-axis after irradiation. The abundance of dislocation loops on prism planes suggests that the anisotropic a-axis lattice swelling, which decreases as the irradiation temperature increases, is the primary cause of grain boundary microcracking. The dominance of prism planar defects and $a > c$ lattice swelling in ZrB_2 may be related to the lower c/a ratio of 1.12 for ZrB_2. It is suggested that the formation of matrix and grain boundary cavities in the 560°C irradiated samples was caused by the production of helium and an increase in temperature due to neutron absorption from residual ^{10}B transmutation.

S19 Molecular-level Processing and Chemical Engineering of Functional Materials

SYMPOSIUM 19: Energy-Related Matters I

Room: Ballroom 3
Session Chair: Christina Birkel, Arizona State University

10:20 AM

(ICACC-S19-012-2024) Actinide Oxide Nanomaterials Synthesized from Molecular Single-Source Precursors – Promising Materials for Energy Harvesting, Storage and Catalysis

A. Lichtenberg*[1]; S. Mathur[1]

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

Actinide (An = Th, U) molecular complexes have been successfully synthesized and employed as single source precursors (SSPs) for generating nanostructured actinide oxide materials. As a result, a variety of thin films comprising thorium and uranium oxides have been generated using sophisticated methods, such as metal organic chemical vapor deposition (MO-CVD), plasma-enhanced chemical vapor deposition (PE-CVD), and magnetic field chemical vapor deposition (MF-CVD). Furthermore, the SSPs have been effectively employed in the microwave-assisted synthesis of actinide oxide nanoparticles. Actinide oxide-based nanomaterials exhibit intriguing catalytic potential in both theoretical considerations and practical applications. Notably, some of the actinide oxide materials we've synthesized have found application in the realms of energy generation and storage. Furthermore, thorium- and uranium oxides have been assessed as highly suitable for some catalytic applications, one of which includes the synthesis of ammonia (NH_3) from nitrogen (N_2) and hydrogen (H_2).[1] (1) G. Wang G, E.R. Batista, P. Yang, Front. Chem. 2023, 10, 1051496.

10:40 AM

(ICACC-S19-013-2024) Porous Monolithic Perovskite Structures for High-Temperature Thermochemical Cycles (Invited)

M. Pein*[1]; A. Eltayeb[1]; C. C. Agrafiotis[1]; L. Klaas[1]; M. Roeb[1]; C. Sattler[1]

1. DLR - German Aerospace Center, Institute of Future Fuels, Germany

High-temperature thermochemical cycles based on redox metal oxides offer great potential to utilize heat from renewable sources such as concentrated solar irradiation, excess renewable electricity and industrial waste heat. Thermochemical cycle concepts and applications investigated at DLR include water and carbon dioxide splitting, heat storage and air separation. In contrast to conventional packed-bed reactor concepts using granulated materials, the implementation of monolithic porous structures, such as foams or honeycombs, can be beneficial in terms of surface area and corresponding reaction kinetics, as well as lower pressure drop. However, these monolithic structures pose additional challenges related to long-term mechanical stability, as metal oxides undergo significant dimensional changes during the reduction and oxidation steps in such thermochemical cycles. Besides the choice of material, the manufacturing route and post-treatment of these monolithic 3D-structures is of utter importance. In this work, recent progress in fine-tuning the perovskite composition of the general formula ABO_3, which were shown to outperform other candidate redox oxides for such processes and the optimization of mechanical and thermodynamic stability of reticulated and 3D-printed porous monolithic perovskite structures are presented and discussed.

11:10 AM

(ICACC-S19-014-2024) Molecular-level processing of chalcogenide-based 2D materials by unique building block concepts (Invited)

V. Brune*[1]; S. Mathur[1]

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

Chalcogenide-based 2D materials possess tunable and defined crystallographic structures and elemental compositions which offer a broad portfolio of potential applications in (opto)electronic as well as energy storage and conversion devices. Unique building block pathways following simple synthetic protocols for molecular precursors enable target molecule-to-material transformation, resulting in chalcogenide-based MX_y (X=S,Se;Y=1,2;M=metal) materials. The need of innovative and smart chemical processing methods to obtain specific material compositions integrating the advancements in materials processing techniques motivated us to establish a reliable synthesis approach of molecular building blocks to form stable precursor classes $[M\{X(C_2H_4)_2NMe\}_y]$ (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;X=S,Se) for functional material processing. The reaction of tridentate donor ligand system with suitable metal compounds resulted in (air)stable molecular precursors, which have been characterized on an atomic scale. These precursors enabled the formation of homogeneous assemblies of inorganic nanostructured materials by thermal decomposition methods (CVD, solvothermal). Appropriated molecular building blocks provide an extraordinary synthetic concept to unique molecular precursor classes delivering an economic approach for sustainable large scale 2D material synthesis.

S5 Next-Generation Bioceramics and Biocomposites

SYMPOSIUM 5: Next Generation Bioceramics and Biocomposites

Room: Coquina B
Session Chairs: Roger Narayan, North Carolina State University; Katalin Balazsi, Centre for Energy Research HAS

10:20 AM

(ICACC-S5-013-2024) Chitosan-derived hydroxyapatite hollow microspheres as drug-laden cell carriers (Invited)

S. Chen[2]; A. Osaka*[1]

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

Microspheres with several hundred micrometers have attracted considerable attention in the development of injectable cell carriers. In particular, microspheres with a hollow cavity exhibited an enhanced drug delivery property. Here, hydroxyapatite hollow microspheres were prepared by in situ biomimetic deposition of hydroxyapatite on chitosan microspheres followed by a sintering process. The resultant hydroxyapatite microspheres exhibited a spherical morphology and a hollow structural feature. They showed a good biocompatibility and served as supporting matrices for osteoblast MC3T3-E1 cells. Moreover, they adsorbed tetracycline hydrochloride (model drug, one type of antibiotics) and well maintained its antibacterial property against the growth of bacteria.

10:50 AM

(ICACC-S5-015-2024) Porous, transparent model minerals for in-situ geobacterial imaging

L. Quinn*[1]; K. Sharma[2]; R. Wipfler[1]; S. Parra[1]; V. Orphan[1]; K. Faber[1]

1. California Institute of Technology, USA
2. University of California, Santa Cruz, USA

Geobacteria such as geobacter sulfurreducens can perform complex chemical transformations of great interest to sustainability research, such as methane oxidation and heavy metal remediation from wastewater. As these bacteria traditionally inhabit opaque sediments with complicated pore networks, studying their interactions with each other and with their habitats is difficult. As a result, these bacteria can be colonized in transparent soil substitutes like Nafion™ or the naturally-occurring mineral cryolite (Na_3AlF_6). However, these materials can be expensive, and ideal transparent soil substitutes would allow for control over pore characteristics such as size and morphology. To address these challenges, this work turns to freeze casting, a facile pore-forming technique that allows for easy tuning of pore morphology by changing solvents, solids loading, and freezing rate. Freeze casting and sintering synthetically-produced cryolite powder produces a biocompatible, transparent, porous solid well-suited to bacterial colonization and imaging. In this work, we explore different pore morphologies accessible through this technique and the effect that pore characteristics have on transparency, permeability, and bacterial colonization. The transparent porous cryolite created using this technique could further allow in-situ, realtime monitoring of bacterial growth and spatial dependence.

S8 18th Intl Symp on APMT for Structural & Multifunctional Materials & Systems

SYMPOSIUM 8: Aqueous synthesis, colloidal processing, bio-inspired synthesis and processing

Room: Coquina F
Session Chair: Michael Halbig, NASA Glenn Research Center

10:20 AM

(ICACC-S8-015-2024) Hydrogen ceramic industry- breakthrough or alternative (Invited)

J. Lis*[1]; D. B. Kata[1]

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

Hydrogen should ultimately become the significant component to generate energy to all practical industry needs in form of electricity or heat, which is used to the production of ceramic and metallic materials. Despite its undoubted advantages, hydrogen as a green fuel has undesirable features also. During its combustion in tunnel kilns, a significant amount of water vapour appears. Given the temperatures of firing at about 1250°C: ceramic tiles, sanitary ceramics, refractory ceramics, noble ceramics or building materials, the steam generated has a detrimental effect on the finished products. Its reactivity at temperatures above 1200°C leads to a number of destructive physico-chemical phenomena causing secondary porosity of the products, uncontrolled phase composition, undesirable microstructural features and degradation of the kiln lining. Controlling these processes is a challenge for current material technologies using H_2 as a heat source. Despite these disadvantages, green hydrogen is widely recognised as a fuel of the future that will be implemented on a large scale in the EU by 2030 with the possibility of expanding the technology to other countries of the world by 2050. In this presentation, a number of aspects relating to the advantages and difficulties of using hydrogen as an alternative source for heating industrial ceramic tunnel kilns are presented.

S16 Geopolymers Inorganic Polymers and Sustainable Construction Materials

SYMPOSIUM 16: Conversion to ceramics

Room: Coquina C
Session Chair: Waltraud Kriven, University of Illinois at Urbana-Champaign

10:40 AM

(ICACC-S16-005-2024) Ceramic synthesis using organic base geopolymers and base-catalyzed hydratable aluminate powders

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Geopolymers were synthesized using some organic bases and then were fired to see the development of crystalline phases and the microstructure. Additionally, organic bases were found to be effective set accelerators for hydratable alumina binders. The reaction products when using the bases guanidine, tetramethylguanidine, and tetramethylammonium hydroxide were characterized at room temperature and after firing. We found differences in reactivity and reaction product due to the amount of five-coordinated aluminum in the initial powder and the base being used. In order to expand the number of possible binder systems, we explored the use of organic bases to induce setting of synthetic alumina-containing powders (e.g., amorphous analogues of mullite, yttrium aluminum garnet, and magnesium aluminum spinel) rather than pure alumina along with the formation of crystalline phases after firing.

11:00 AM

(ICACC-S16-006-2024) Cold Sintered laterites based geopolymers: Densification, microstructure and micromechanic (Invited)

E. Kamseu*[1]; J. Nouping Fokoua[1]; M. Biesuz[2]; A. Akono[3]; S. Rossignol[4]; C. Leonelli[5]; V. M. Sglavo[6]

1. MIPROMALO, Research, Cameroon
2. University of Trento, Department of Industrial Engineering, Italy
3. Northwestern University, Civil and Environmental Engineering, USA
4. Laboratoire SPCTS, France
5. University of Modena and Reggio Emilia, Department of Engineering Enzo Ferrari, Italy
6. University of Trento, Italy

The consolidation of the natural iron-rich aluminosilicate, i.e. laterite, is enhanced using geopolymerisation, external pressure and temperature in an ultra-low energy sintering context. Optimized formulations of laterite and Rice Husk Ash (RHA) are used to investigate the effects of the external pressure and temperature on the porosity, phase's development, micromechanics and microstructure. FT-IR spectroscopy, SEM micrograph, Mercury Intrusion Porosimetry (MIP), Transmission Electronic Microscopy (TEM) and Archimedes Porosimetry method were used to characterize the final densified products. Results showed that ultra-low energy sintering process allows the reduction of ~ 99% of capillary porosity thank to the enhancement of the antiparticles bonding and the formation of nanosize of ferrisilicates. The indentation modulus > 50 GPa, the hardness modulus of 3,18 GPa and the fracture toughness > 1 $MPam^{0.5}$ are in agreement with the compact microstructure, high bulk density (> 2.00 $g.cm^{-3}$) and low water absorption. The interlocking of globular units of H-N-(A, Fe)-S with nanosize of ferrisilicates conducts to high performance ceramics.

11:30 AM

(ICACC-S16-007-2024) Geopolymer route to the synthesis of ultra high temperature ceramic powders (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

Geopolymers are chemically charge-balanced, inorganic polymeric ceramics made from a mixture of an aluminosilicate source and an alkali metasilicate solution. Geopolymers can be used for a wide variety application from low technology to high technology materials by controlling its chemical composition. The new trend is that geopolymers can be considered as precursors to ceramic formation. Thus, the XRD amorphous structure of pure geopolymers at room temperature can be transformed into their refractory analogues (i.e. nepheline, leucite or pollucite) at high temperature over ~950°C. The other approach is that geopolymers can be filled with reactive carbon nano-powders before or after curing at room temperature. Following additional curing and powdering, they can be converted into high temperature tailored SiC, Si_3N_4, SiAlON in the form of nanoparticles by carbothermal reduction and nitridization processes at high temperature over ~1400°C. The geopolymer route has an advantage over traditional synthesizing methods of these materials, having a high volume fraction of nanoporous microstructure and using a higher amount of nano-sized carbon filler as well as increased reaction between the reactants and therefore transformed amount of materials. Three types of high temperature ceramics, SiC with high yield over 95 %, Si_3N_4 in two a and b polymorphs and SiAlON type compounds, were synthesized.

S8 18th Intl Symp on APMT for Structural & Multifunctional Materials & Systems

SYMPOSIUM 8: Polymer-based processing

Room: Coquina F
Session Chair: Jerzy Lis, AGH University of Science and Technology

10:50 AM

(ICACC-S8-016-2024) Challenges using Polymer Derived Ceramics for Thermoplastic Material Extrusion based Additive Manufacturing (Invited)

F. Clemens*[1]; F. Sarraf[1]; S. Churakov[2]

1. Empa, Swiss Federal Laboratories for Materials Science and Technology, Switzerland
2. University of Bern, Switzerland

Preceramic polymers (PCPs) are used to produce SiO_2, $Si_xO_yC_z$, silicate or non oxide ceramics. T hermogravimetry analysis (TG) was used to demonstrate that the yield of methyl-silsesquioxane SILRES MK after pyrolysis is influenced under air atmosphere by the heating rate. The yield was reduced from 81 wt% to 75 and 69 wt% using 0.6 and 0.3 K/min heating rates, respectively. The reduced SiO_2 yield of PCP at slow heating rates can be explained by the high volatility of cage structured POSS spices in the PCP and was confirmed by GC-MS and FTIR analysis. The results are highly relevant for thermoplastic material extrusion based additive manufacturing (MEX-AM) because slow heating rates are needed to avoid structural defects like blisters, cracks or pores, and therefore, a variation of the PCP yield during post-printing processes generates a problem in obtaining defined chemical composition of silicate ceramics, glass ceramics before sintering as expected. By XRF it could be confirmed that for low heating rates (0.6k/min) a mullite composition with 60/ 40 wt% Al_2O_3/ SiO_2 ratio resulted in a 61.70/37.70 wt% of Al_2O_3/ SiO_2 ratio due to the lower yield of PCP. However, we could demonstrate, that by using compounding at higher temperatures the residue can be stabilized even at low heating rates and the defined chemical composition of silicates can be achieved successfully.

11:20 AM

(ICACC-S8-017-2024) On the Design of High Performance Bioplastics by Using Advanced Blend Design and Solvent Casting (Invited)

E. Oloo[1]; S. Gupta*[1]

1. University of North Dakota, Mechanical Engineering, USA

The growing interest in sustainability has made sustainable practices significant aspects of materials research and engineering. A current major global issue that requires sustainable solutions is the issue of plastic wastes disposal associated with the production and usage of petroleum-based (traditional) plastics. Along with recycling, bioplastics have emerged as a potential solution. However, the weaker mechanical and physical properties of bioplastics in comparison to traditional plastics have limited their applications. As a result, numerous studies on polymer blending/mixing are being investigated in efforts to develop novel bioplastic composites with enhanced properties. This study explores the blending of renewable and biodegradable bioplastics including PLA, PHA, and CA to create tertiary bioplastic composites. The study also analyzes the utilization of lignin as a reinforcement additive to improve the properties of bioplastics. The control sample consist of 50 PHA-50 PLA. A solvent casting process is the fabrication method utilized for all the samples studied. Thermal, microstructure characterization, and mechanical testing are performed on all samples to analyze the effects of the polymer blending on different properties. The highest ultimate tensile strength was observed in the 1 dissolved lignin-49.5 CA-PHA-PLA at (37.289 MPa, 52.9% increase).

11:50 AM

(ICACC-S8-018-2024) Compositionally Complex Ultra-High Temperature Ceramics via Metal Functionalized Preceramic Polymers

J. Ponder*[2]; H. Hackbarth[1]; N. D. Posey[2]; J. Delcamp[3]; N. Bedford[1]; M. B. Dickerson[3]; T. Pruyn[3]

1. University of New South Wales, Australia
2. Air Force Research Lab/UES Inc., Materials and Manufacturing Directorate, USA
3. Air Force Research Laboratory, Materials and Manufacturing Directorate, USA

Materials capable of withstanding ultra-high temperatures are becoming increasingly important for various aerospace applications. While silicon-based ceramics have proven to be a key class of materials for harsh environments, transition metal-based ceramics are required for ultra-high temperatures conditions. Preparation of ultra-high temperature ceramics (UHTCs) via powder sintering limits the structural designs possible and is challenging for preparing composite components. Preceramic polymers (PCPs) have been used to address these challenges via the synthetic tunability and processing properties of polymers. Following pyrolysis of the PCP, a polymer derived ceramic (PDC) is obtained. By tuning PCP structure and curing/pyrolysis conditions, the composition of the PDC can be manipulated. We report that functionalizing a polysilazane PCP with transition metal complexes yields polymer-metal complexes that convert to UHTCs composites when pyrolyzed. Individual metal complexes and blends were studied to understand how the resulting PDC phases blend. Following pyrolysis under different conditions, various diffraction, spectroscopic, and microscopy methods were utilized to understand the ceramization process and final PDC composition. Ultimately, we report a route to polymer derived compositionally complex UHTC nanocomposites suitable for various high temperature applications.

13th Global Young Investigator Forum

13th Global Young Investigator Forum: Design and processing

Room: Coquina D
Session Chairs: Zhezhen Fu, Penn State Harrisburg; Marco Mariani, Politecnico di Milano

1:30 PM

(ICACC-GYIF-017-2024) Stability and CMAS Resistance Optimization of Rare Earth Disilicates for Environmental Barrier Coatings via High Entropy Design (Invited)

L. Sun*[1]; J. Wang[1]

1. Shenyang National Laboratory for Materials Science, Institute of Metal Research, Chinese Academy of Sciences, Advanced Ceramics and Composites Division, China

Environmental barrier coatings (EBCs) have been developed to improve the durability of SiC_f/SiC CMC components against harsh combustion environment. Rare-earth (RE) disilicates attract attentions for their low thermal expansion coefficient, excellent high temperature water vapor and good thermal and chemical compatibility with silicon-based ceramics and composites. Herein, we summarized the recent progress on the optimizations of critical properties of rare-earth silicates through strategic high entropy design to modify the current performance deficiencies of rare-earth silicates like thermal properties (coefficient of thermal expansion and thermal conductivity), CMAS corrosion resistance and high temperature phase stability in Institute of Metal Research, Chinese Academy of Sciences. The present advancements demonstrate the merits of high entropy engineering for advanced EBCs for the improvement of crucial properties in engine applications.

2:00 PM

(ICACC-GYIF-018-2024) Compositional design of multicomponent rare-earth disilicates environmental barrier coating materials for SiC_f/SiC composite

Y. Luo*[1]; L. Sun[1]; J. Wang[1]

1. Institute of Metal Research, Chinese Academy of Sciences, China

Multifunctional environmental barrier coating (EBC) is necessarily used to protect the turbine components made of SiC_f/SiC composites from environmental attacks. A prime strategy of designing multifunctional EBC is doping multiple rare-earth principal components into rare-earth disilicates to achieve synergetic optimization on multiple properties. However, controlling the phase formation of multicomponent rare-earth disilicates $(nRE_x)_2Si_2O_7$ remains a crucial challenge, due to the complex polymorphic phase competitions and transformations led by different RE^{3+} combination. Herein, the mechanisms of phase formation for $(nRE_x)_2Si_2O_7$ materials are investigated via high-throughput modeling and experiments. From the viewpoint of configuration ensembles, it is found that their phase formation capability is governed by the ability to accommodate sufficient configurational randomness of multiple RE^{3+} cations into the lattice, which could be described by the configurational entropy of mixing. The dependence of phase formation and stabilization of $(nRE_x)_2Si_2O_7$ on the effective multi-RE^{3+} radius is discussed. A guideline is proposed to precisely control the phase formation capability and high-temperature stability of $(nRE_x)_2Si_2O_7$. The results can accelerate the design of multicomponent EBC materials with tailored compositions and controlled polymorphic phases.

2:20 PM

(ICACC-GYIF-019-2024) High-Char Preceramic Composite Resin for Infiltration-free Ceramic Matrix Composite Manufacturing

A. Thukral*[1]; K. Bhattacharyya[1]; R. Pandey[1]; G. Iftime[1]; B. Karki[1]; J. Wei[1]

1. Palo Alto Research Center, USA

The economic viability of current manufacturing process for fiber-reinforced ceramic matrix composites (CMCs) is severely constrained by the highly resource and cost intensive nature of repeated infiltration cycles. The fundamental limitation stems from non-availability of high-char preceramic resins. The lower char-yield results in substantially high porosity (~25%) in ceramic matrix upon pyrolysis, necessitating implementation of multiple resin-infiltration cycles to fill the pores. In this work, we will showcase our novel preceramic resin and its synthesis route which enables a scalable, infiltration-free approach to manufacturing CMCs. The chemical linking between inorganic ceramic particles and organic monomer is designed to facilitate high loading of inorganic particles within the resin, while maintaining a sufficiently low melt viscosity for effective polymer impregnation. We will present comprehensive chemical, physical, and morphological characterization of our preceramic resin and functionalized particles. Our resin exhibits a suitable processing temperature window, temperature-viscosity correlation, gel time, and well-dispersed network of inorganic particles within the resin matrix. Furthermore, we will demonstrate its practical application through the infiltration-free fabrication of carbon-fibers reinforced ceramic matrix (Cf/C) composite.

2:40 PM

(ICACC-GYIF-020-2024) Development of regenerative spinel oxide catalysts for biomass utilization

S. Yamaguchi*[1]; T. Ozaki[1]; T. Suyama[1]; S. Ikawa[1]; T. Kobayashi[2]; T. Kobayashi[2]; M. Ootani[3]

1. Osaka Research Institute of Industrial Science and Technology, Applied Material Chemistry, Japan
2. Bigbang Co., Ltd., Japan
3. Kansai Catalyst Co., Ltd., Japan

To attain carbon neutrality, power generation from biomass has attracted attention recently. Biomass is converted to gasified gas containing hydrogen and carbon monoxide via gasification. Gasification is complex reactions, combustion, pyrolysis, and reduction induced by heat source at ca. 400-1100 deg. C. Despite of attractive power generation using efficient engines or solid oxide fuel cells (SOFCs) with a supply of gasified gas, tar in the gasified gas provokes clogging on filters, flow channels, engines, and electrodes of SOFCs. For converting tar to hydrogen and carbon monoxide efficiently, stable and active catalyst for is demanded. We have previously elucidated that Ni-Al spinel oxide catalyst (NAO) possessed high stability owing to regenerative function through reduction and oxidation and high activity for reforming tar. In this study, we investigated the spinel oxide catalyst in a practical system for its regenerative function, mechanical strength, and activity for reforming tar. As a result, rolling granulated NAO and zirconia added NAO (NAZO) was found to serve high mechanical strength, activity for tar removal, and permeability in the catalyst bed. Additionally, a heat resistant bacterium suspended in mist water was confirmed to be sterilized by passing through the catalyst bed in a reformer. These results are useful for an efficient gasification of materials contaminated with bacterium.

S2 Advanced Ceramic Coatings for Structural/Environmental & Functional Applications

SYMPOSIUM 2: Environmental Barrier Coatings III

Room: Flagler C

Session Chairs: Jie Zhang, Institute of Metal Research, Chinese Academy of Sciences; Bryan Harder, NASA Glenn Research Center

1:30 PM

(ICACC-S2-007-2024) Evolution of residual stresses during metastable crystalization of rare-earth (di) silicate environmental barrier coatings on SiC CMC substrates (Invited)

R. Sarrafi-Nour*[1]; D. Dunn[1]; Y. Gao[1]

1. GE Aerospace, Research Center, USA

Owing to their attractive properties, rare-earth silicate material system is now well-established as the material system of choice for environmental barrier coating (EBC) on Si-based ceramics and ceramic matrix composites (CMC) components of advanced gas turbines and propulsion engines. Thermal spray methods, atmospheric plasma spray (APS) in particular, have been a common platform for application of both thermal and environmental barrier coatings at industrial scale. Deposition of rare-earth silicate materials via APS often encourages formation of amorphous and metastable phases. The deposited coatings then assume their equilibrium phase structures on heat treatment or exposure to high temperatures. This presentation will discuss the effect of metastable crystallization of APS EBC systems during post-deposition heat treatment on a SiC CMC substrate. In-situ synchrotron X-ray diffraction stress measurements of coated SiC CMC substrates are used as a means to monitor the evolution of residual stresses associated with the EBC layers. The results track the sequential response of the coating-substrate system to increasing temperature after the EBC deposition and before the system achieves equilibrium conditions. The observations are coupled and compared with residual stress measurements using beam curvature methods.

2:00 PM

(ICACC-S2-008-2024) Thermal Properties of Plasma Spray-Physical Vapor Deposition and Suspension Plasma Sprayed HfO_2 Coatings

B. J. Harder*[1]; M. Slizik[1]; M. J. Presby[1]; J. L. Stokes[1]; L. C. Hoffman[1]; A. Vozar[2]; N. Antolino[2]; J. Wan[2]; A. Setlur[2]; R. Sarrafi-Nour[2]

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

Si-based ceramics such as SiC/SiC ceramic matrix composites require environmental barrier coatings (EBCs) for operation in turbine engine environments. However, for future turbine engines with combustion gas and turbine inlet temperatures in excess of 3000°F (1650°C), EBCs will likely require additional thermally tolerant oxide topcoats. These topcoats need to be stable in the turbine environment in addition to possessing low thermal conductivity to provide thermal barrier functions yet offer acceptable mechanical properties. One prospective material system is HfO_2, which can be incorporated into high temperature EBCs as topcoats via various processing methods. In this work, monoclinic HfO_2 was applied on bulk α-SiC substrates via two methods: suspension plasma spray (SPS) and plasma spray-physical vapor deposition (PS-PVD). These processes deposit materials in psuedocolumnar structures for increased in-plane compliance to better manage the thermal expansion mismatch stresses with Si-based ceramics to allow for better adhesion to Si-based ceramic substrates and standard EBC materials. HfO_2 coatings deposited using both processing methods were evaluated in the as-deposited and heat treated conditions up to 2800°F (1538°C). The resulting microstructure and phase of both coating processes are compared and evaluated using laser thermal conductivity and preliminary CMAS testing.

2:20 PM

(ICACC-S2-009-2024) Mechanical Testing of Suspension Plasma Spray and Plasma Spray-Physical Vapor Deposition HfO_2 Coatings

A. Vozar*[1]; B. J. Harder[2]; R. Sarrafi-Nour[1]; M. J. Presby[2]; J. Salem[3]; N. Antolino[1]; A. Setlur[1]; J. Wan[1]

1. GE Research, USA
2. NASA Glenn Research Center, Environmental Effects and Coatings, USA
3. NASA Glenn Research Center, Materials and Structures, USA

The introduction of silicon carbide/silicon carbide (SiC/SiC) ceramic matrix composites (CMCs) into aviation turbine engine hot section enabled higher inlet temperature and reduced cooling requirements. Environmental barrier coatings (EBCs) are required to achieve CMC aerospace turbine components durability. Future EBC systems will require operation in environments with combustion gas and turbine inlet temperatures in excess of 3000°F (1650°C). Oxide topcoats of EBC architectures are required to provide sufficient EBC thermal barrier function and mechanical properties. Hafnium oxide (HfO_2) has been evaluated as a prospective oxide topcoat material deposited in a pseudocolumnar structure for increased in-plane compliance to better manage thermal expansion mismatch stresses with Si-based ceramics, thus, help adhesion to Si-based ceramics. Coatings were applied using suspension plasma spray (SPS) and plasma spray-physical vapor deposition (PS-PVD) on bulk α-SiC substrates and on a proprietary bondcoat or sublayer system to compare the properties of the two deposition methods. Samples were exposed to aging heat treatment from 2500-2800°F (1371-1538°C) in air and mechanical pull testing was conducted at room temperature to track adherence with aging. Microstructure effects and failure mechanisms and the prospective for these systems as a part of advanced EBC systems is discussed.

2:40 PM

(ICACC-S2-010-2024) Modeling oxidation kinetics of oxide-silicon composites

J. Wan*[1]; A. Setlur[1]; R. Sarrafi-Nour[1]; C. Henderson[1]; M. Barber[1]; K. Bryce[2]

1. GE Aerospace, Research Center, USA
2. Rensselaer Polytechnic Institute, USA

In order to evaluate the potential of oxide/non-oxide composites for application in engine combustion environments, a first-principle analytical model was developed to account for the oxidation kinetics of composites of refractory oxides and silicon. Under normal oxidation situations, diffusion of oxidants, either oxygen or water vapor, through a bi-continuous network of metal oxides and silica controls the rate of oxidation. This leads to a parabolic relationship between oxidized layer thickness versus time. Oxidation tests were conducted over different temperature and oxygen pressure levels. Oxides considered include mullite, hafnon, a rare earth silicate and hafnia. The model was validated in cases where diffusivity and/or permeability data is available. Rate constants were calculated over different temperature ranges. For example, in the mullite-silicon system at higher temperatures, diffusion of oxygen through mullite matrix controls the oxidation rate. At lower temperatures, oxygen or water diffusion though silica dominates. This model provides a preliminary picture how the oxidation kinetics can be predicted through basic diffusion parameters for such composites.

3:20 PM

(ICACC-S2-011-2024) Architectural development of Ytterbium Disilicate Environmental Barrier Coatings (Invited)

J. Zhang*[1]; H. Wang[1]; Z. Luo[1]; J. Wang[1]

1. Institute of Metal Research, Chinese Academy of Sciences, Shenyang National Laboratory for Materials Science, 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 strong/ductile layered structure composed of dual-phase layer and $Yb_2Si_2O_7$ layer significantly reduced the tendency for mud crack initiation during thermal cycling. 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.

3:50 PM

(ICACC-S2-012-2024) High-Temperature Slurry Environmental Barrier Coating with Graded HfO_2-$HfSiO_4$ Topcoat

R. I. Webster*[1]; K. Lee[1]; B. J. Harder[1]; B. Puleo[1]

1. NASA Glenn Research Center, USA

Environmental barrier coatings (EBCs) have enabled the use of silicon carbide (SiC)-based ceramic matrix composites (CMCs) in gas turbine engines by protecting the underlying CMC from corrosive combustion species. Current-generation EBCs consist of a rare earth silicate topcoat and a silicon bond coat. The relatively low melting point of the silicon bond coat (1414°C) limits the upper use temperature of these coatings. To protect SiC-based CMCs at temperatures beyond that achievable by the current state-of-the-art, an oxide-based bond coat capable of withstanding temperatures of up to 1482°C has been developed at NASA Glenn Research Center. Hafnia (HfO_2) is a promising EBC topcoat material due to its stability in high-temperature steam; however, its coefficient of thermal expansion (CTE) is highly anisotropic and much larger than that of SiC. In this study, a graded HfO_2-$HfSiO_4$ topcoat was deposited via a slurry process on the NASA-developed oxide-based bond coat. The oxidation resistance of this EBC system was evaluated at 1482°C in a steam cycling environment. The durability and stability of this slurry-deposited HfO_2-$HfSiO_4$ topcoat was compared to that of HfO_2 deposited by plasma spray physical vapor deposition (PS-PVD).

4:10 PM

(ICACC-S2-013-2024) Foreign Object Damage and Oxidation in a Modified $Yb_2Si_2O_7$ Environmental Barrier Coating

L. C. Hoffman*[1]; M. J. Presby[1]; B. J. Harder[1]; J. L. Stokes[1]

1. NASA Glenn Research Center, Environmental Effects & Coatings, USA

Foreign object damage (FOD) is one of the key damage/failure modes in environmental barrier coatings (EBCs) developed for gas turbine engines. A limiting factor of EBC development is the growth of a SiO_2 thermally grown oxide (TGO) layer which exacerbates spallation. Previous work studied FOD in NASA's Generation II Ytterbium Disilicate EBC and recently it has been shown that the addition of oxide modifiers to the Ytterbium Disilicate EBC resulted in significant reduction of TGO growth. The present work will compare FOD in a modified Ytterbium Disilicate EBC to that of

the baseline Ytterbium Disilicate EBC. The effects of steam exposure prior to impact will also be explored. 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 a normal incidence angle. The impact damage was characterized by optical profilometry and scanning electron microscopy (SEM) of the cross-sections.

4:30 PM

(ICACC-S2-014-2024) Solid State Reaction Kinetics of Yttria- Alumina Diffusion Couple Produced by Spark Plasma Sintering

C. S. Witharamage*[1]; E. J. Opila[1]

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

High entropy rare earth oxides (HERO)-based environmental barrier coatings (EBC) have been developed to protect refractory alloys from rapid oxidation in high-temperature environments. However, if the HERO coating contains cracks or defects, the alloy will be exposed to elevated temperatures. Adding an Al_2O_3-forming interlayer between the HERO coating and the alloy has been proposed as an oxygen diffusion barrier. In addition, the HERO compositions are potential EBC candidates for Al_2O_3/Al_2O_3 ceramic matrix composites, which are also used for hot section components in gas turbines. Therefore, understanding the reaction kinetics between RE_2O_3 and Al_2O_3 is crucial. The solid-state reaction kinetics between Y_2O_3 and Al_2O_3 have been studied as representative of the HERO coating/Al_2O_3 interface. Y_2O_3-Al_2O_3 diffusion couples were produced by spark plasma sintering (SPS) at 1350 C, 1425 C, and 1500 C. SPS diffusion couples were heat-treated in the air at their consolidation temperatures for 0, 24, and 96 hours. Scanning electron microscopy revealed the formation of yttrium aluminum garnet, yttrium aluminum perovskite, and yttrium aluminum monoclinic reaction product layers at the interface at all temperatures. The thickness of each layer was determined as a function of exposure temperature and time, which determined the reaction kinetics and formation enthalpy of each layer.

S13 Development & Applications of Adv Ceramics & Composites for Nuclear Fission/ Fusion Energy Sys

SYMPOSIUM 13: Ceramic fuel materials, technologies, and characterization

Room: Ballroom 4
Session Chair: Peng Xu, Idaho National Lab

1:30 PM

(ICACC-S13-017-2024) Beyond TRISO: Development of coated particle fuel (Invited)

E. Lopez Honorato*[1]; R. Heldt[1]; A. Diaz[1]; E. Dominguez[1]; F. Dal Forno Chuahy[1]; T. J. Gerczak[1]; J. Hunn[1]

1. Oak Ridge National Lab, USA

Traditional tristructural-isotropic (TRISO) fuel particles are made of three layers of pyrolytic carbon (PyC) and one of SiC produced by fluidized bed chemical vapor deposition (FBCVD). Although originally designed for high-temperature gas-cooled reactors, other coated particle fuels have been proposed for several future micro reactor designs for terrestrial use and space exploration. The expanded envelope of operating conditions introduced by some of these new applications requires the fabrication of coated particle fuels with a wide variety of kernel compositions (oxides, carbides, or nitrides), kernel sizes (200–800 μm), coater kernel charges (from tens of grams to kilograms), and differences in the number of coating layers, coating thicknesses, and coating compositions (e.g., PyC, SiC, ZrC, W, ZrN, etc.). Each of these variables requires not only modifications to the deposition conditions but also the FBCVD system to produce coatings with optimal performance. In this work, we discuss addressing the challenges associated with the development of new coated particle fuels using the experience from the Advanced Gas Reactor Fuel Development and Qualification (AGR) program in the US, as well as the use of additive manufacturing, image analysis, computational fluid dynamics, data mining, and machine learning to accelerate the development of new coated particle fuels.

2:00 PM

(ICACC-S13-018-2024) Sintering and densification of gel-cast ceramic nuclear fuels

P. Makurunje*[1]; S. Middleburgh[1]

1. Nuclear Futures Institute, Bangor University, United Kingdom

Droplet formation techniques contribute to the shaping of microspherical nuclear fuel particles, typically of uranium oxide. In the present study, gel-casting, a wet granulation colloidal extrusion technique, was used to manufacture microspherical kernels. A colloid of uranium dioxide feedstock was mixed with a gelatinising organic polymer capable of solidifying by ion exchange. The colloid-polymer system was extruded through a nozzle and cast to form a macromolecular network that hardened into microspherical kernels. Sintering analyses were performed. Microstructural studies on the residual phases of sodium and calcium revealed a peculiar sintering behaviour of the gel-cast kernels. Alternating patterns of limited and exaggerated grain growth were observed across grain clusters. Sodium-rich crystallites with a core-shell microstructure showed that residual sodium acts as a sintering aid. Thermal conductivity and expansion studies were important in understanding sodium's effect on fuel performance against its contribution to fuel particle sintering.

2:20 PM

(ICACC-S13-019-2024) 3D porosity characterisation of TRISO coatings using plasma focused-ion-beam tomography

E. White*[3]; S. Waters[2]; M. Jiang[1]; Q. Zhang[3]; M. Davies[4]; D. Goddard[5]; N. Tzelepi[6]; D. Liu[3]

1. University of Bristol, United Kingdom
2. United Kingdom Atomic Energy Authority, Materials Research Facility, United Kingdom
3. University of Bristol, Physics, United Kingdom
4. Ultra Safe Nuclear Corporation, United Kingdom
5. National Nuclear Laboratory, Preston Laboratory, United Kingdom
6. National Nuclear Laboratory, Central Laboratory, United Kingdom

TRistructural ISOtropic nuclear fuel (TRISO) is a type of ceramic coated nuclear fuel that is under consideration for the next generation of nuclear reactors. TRISO particles consist of a central fuel kernel, a porous carbon 'buffer' coating and a SiC coating that is sandwiched between two pyrolytic carbon (PyC) coatings. Fuel performance models use assumptions about the porosity distribution and density of the coatings, therefore it is important to evaluate these coatings post-manufacturing to quantify and understand the porosity distribution to better inform these models. Plasma focused ion beam scanning electron microscopy (PFIB-SEM) has been applied to these coatings because, unlike conventional FIB-SEM, PFIB-SEM can run at much higher currents, allowing for much faster milling speeds. This allows for larger, more representative images of coating microstructure. This is important when carrying out 3D segmentation to extract out the porosity distribution. For this work, the segmentation will focus on the buffer and IPyC coatings, and their comparison to FIB-SEM segmentations. The comparison of FIB-SEM to PFIB-SEM is an important exercise to carry out, not only does it allow for validation of the segmentation method, but by comparing pore volume distributions, future users can be better informed on the microstructure length scales covered specifically by each technique.

2:40 PM

(ICACC-S13-020-2024) Peridynamics Modelling of TRISO Coated Particle Fuel, a Comparison between 2D and 3D Models

A. Battistini*[1]; T. A. Haynes[2]; L. Jones[3]; M. Wenman[1]

1. Imperial College London, Materials, United Kingdom
2. University of East Anglia, United Kingdom
3. National Nuclear Laboratory, United Kingdom

TRISO coated particle fuel is an advanced technology fuel developed as a solution for conventional and high temperature gas nuclear reactors, with the latter offering the possibility of nuclear co-generation powered industrial facilities and hydrogen production, both technologies that could provide important contributions towards net-zero. A bond-based Peridynamics model has been developed and implemented in the finite element code "Abaqus" in order to simulate the behaviour of TRISO during thermo-mechanical transients. Thanks to the non-local formulation of Peridynamics, this model can autonomously predict crack formation and propagation without the need for fundamental modifications to the solution strategy, usually required by most of the available codes to deal with fracture mechanics. The available model has been updated to account for residual stresses and the effect of the spherical geometry on the simulations, which until now have been restricted to 2D plane stress and strain approximations. Preliminary results from a 3D model have also been included, focusing on the outer layers (pyrolytic carbon and silicon carbide) of the TRISO particle and the interface between them to reduce the computational requirements. These layers have been chosen as the correct prediction of their behaviour is paramount for the safety assessment and lifetime performance prediction of this new fuel.

S15 8th International Symposium on Additive Manufacturing and 3-D Printing Technologies

SYMPOSIUM 15: Binder Jetting and Powder Bed Fusion Processes I

Room: Coquina H
Session Chair: Soshu Kirihara, Osaka University

1:30 PM

(ICACC-S15-021-2024) Ceramics Binder Jetting by Granulated Particle and Nanoparticle Containing Ink; Particle Homogenizing Modeling (Invited)

K. Kamoda*[1]

1. Ricoh Company, Ltd., Advanced technology R&D Division, Japan

Ceramic additive manufacturing is expected to use for manufacturing structural ceramics parts, but the modeling quality (e.g., accuracy, density) is limited when manufacturing thick parts because of debinding problem. This study presents a novel binder jetting (BJ) method called particle homogenizing modeling (PHM) for manufacturing structural ceramics. The PHM method is characterized by the ceramic granulated particles used for the recoating powder, and ink which contains ceramic nanoparticles and a solvent that collapses the granulated particles. In this study, the relative density of the green body is evaluated as a function of the ink saturation to verify the effectiveness of the proposed PHM method. Homogenization occurs due to increased saturation, and consequently, the highest value of the relative density of the sintered body is 95 vol.%. Furthermore, less deformation or cracking is observed when compared to previous studies, and the shrinkage rate is constant when green bodies with a thickness of 10 mm to 30 mm are sintered. Building parts 10 mm or thicker without deformation or cracking would be a groundbreaking advancement in the ceramics additive manufacturing field. The novel PHM method can potentially be used to manufacture large, high-density, high-precision ceramic parts, and can also be applied in various industrial fields in the future.

2:00 PM

(ICACC-S15-023-2024) Layerwise Slurry Deposition (LSD-Print) of silicon carbide

N. H. Schubert*[2]; A. Zocca[1]; J. Guenster[2]

1. BAM Federal Institute for Materials Research and Testing, Ceramic Processing and Biomaterials, Germany
2. BAM Federal Institute for Materials Research and Testing, Germany

The Layerwise Slurry Deposition (LSD-Print) is a slurry-based variation of binder jetting that combines a highly packed powder bed with the precise ink printing of the binder jetting method. In comparison to most additive manufacturing (AM) techniques the layerwise slurry deposition (LSD-print) realizes a high green density and good interconnection of the single layers by spreading and drying thin layers of ceramic slurry. Through the high capillary forces developed during drying a green density of up to 60 % is possible. In combination with the jetting of ink by means of a printhead, which prints the 2D- cross-sections of the wanted 3D-Part, complex geometries with features up to 1 mm are realizable. After heat treatment, the printed ink hardens and generates 3D parts that are stable in water, while the rest of the powder bed is washed out. The current presentation focuses on the LSD-print of silicon carbide ceramics, discussing the possibility to produce silicon infiltrated silicon carbide (SiSiC) parts with density and mechanical properties close to traditional SiSiC. Additionally, the challenges and recent developments in developing slurries for the LSD-print of sintered silicon carbide (SSiC) will be discussed.

2:20 PM

(ICACC-S15-024-2024) Depowdering of an Additively Manufactured Ceramic Heat Exchanger with Narrow and Turned Channels

M. Du*[1]; W. Yu[1]; D. France[2]; D. Singh[1]

1. Argonne National Lab, USA
2. Univeristy of Illinois Chicago, Department of Mechanical and Industrial Engineering, USA

Depowdering is a critical step in powder bed-based additive manufacturing (PBAM) to remove the unbound powder around/inside the printed parts. This step can be challenging, especially to internal unbound powder, for designs with complex inner geometries (e.g., narrow and turned channels). In this study, a one-piece silicon carbide heat exchanger with multiple layers of internal channels was printed using the binder jetting additive manufacturing process. The channels have a semi-elliptical geometry (with cross-section dimensions of 5.9×2.9 mm) and multiple turnings along the length. To depowder this design, two methods were systematically tested, including compressed air and vortex motion. Effects of air pressure and motion power on the structural and surface integrity of the exchanger body and channel surface were examined. It was found that vortex motion or compressed air alone could only partially depowder the internal unbound powder of the printed heat exchanger. Consequently, a combination of vortex motion and compressed air blowing with multiple cycles was developed and tested for this purpose. A study of the effect of vortex time in each depowdering cycle was conducted, and results showed that an optimized depowdering cycle could completely depowder the narrow and turned channels while maintaining structural and surface integrity and an efficient number of cycles.

S16 Geopolymers Inorganic Polymers and Sustainable Construction Materials

SYMPOSIUM 16: Conversion to ceramics II

Room: Coquina C
Session Chair: Waltraud Kriven, University of Illinois at Urbana-Champaign

1:30 PM

(ICACC-S16-008-2024) Investigations of CO_2-sorption and sequestration in zeolites and as produced lime-sand bricks (Invited)

C. Rüscher*[1]; B. Hagel[1]; N. Och[1]

1. Leibniz University Hannover, Mineralogy, Germany

In the first part it is shown that as prepared hydro sodalites (FAU type zeolite) like commercially available zeolite 13X and 4A dehydrates completely during heating up to 400°C, i.e. about 13 wt%, 23 wt% and 20 wt%, respectively. Rehydration occurs at room temperature at open (atmospheric) conditions in certain time depending on the humidity. Data are collected for the mass gain of thermally dehydrated samples during cooling (and at room temperature) under CO_2/N_2 gas-mixtures. Dehydrated hydro sodalites gain up to 6 wt % and Z13X, Z4A gain up to about 15, 10 wt% when exposed to CO_2/N_2 > 20wt% CO_2 (flowing gas 30 ml/min, 1 bar). Leaving the CO_2 sorbed samples at open conditions the CO_2 contents of Z13X and Z4A becomes completely replaced by H_2O, whereas the CO_2 entrapped in the sodalite attract H_2O forming $(H_3O)^+HCO_3^-*2(H_2O)$ type species. For comparison for CO_2-sorption experiments on geopolymer samples and geopolymer/zeolite composites we may refer to known literature. In the second part it is shown that the crystalline CSH-phases (C=CaO, S=SiO_2, H=H_2O) formed during hydrothermal production of lime-sand bricks becomes rather slowly carbonated at atmospheric conditions: $CSH+CO_2 \Rightarrow CaCO_3+SiO_2+H_2O$. It is often overlooked, that here SiO_2 forms an amorphous polymer acting as a glue between the sand-grains. Conditions for accelerated carbonation and polymer formation are worked out.

S17 Advanced Ceramic Materials and Processing for Photonics and Energy

SYMPOSIUM 17: Multi-functional materials and Advanced and nanostructural materials for photo-voltaics and solar fuels

Room: Coquina G
Session Chairs: Farid Akhtar, Lulea University of Technology; Fiorenzo Vetrone, Institut National de la Recherche Scientifique, Université du Québec

1:30 PM

(ICACC-S17-016-2024) Advancements in Photoelectrochemical Hydrogen Production based CdS Photoanodes (Invited)

M. Siaj*[1]

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

Photoelectrochemical (PEC) water splitting, leveraging photoactive materials to convert solar energy into hydrogen, has garnered substantial attention. The challenge lies in designing semiconductor photoanodes with exceptional light harvesting, photochemical stability, charge carrier separation, and redox ability. In our study, we investigated innovative strategies to enhance PEC performance. Firstly, a unique CdS/MoS_2 heterojunction photoanode was constructed using chemical vapor deposition (CVD), exhibiting remarkable efficiency due to optimal junction thickness and synergistic effects between MoS_2 and CdS. Secondly, a pure Z-scheme heterostructure, CdS/Au/SnO_2-S, was designed, significantly boosting charge carrier separation through an ingenious incorporation of Au nanoparticles, resulting in improved photocurrent density and conversion efficiency. Lastly, a plasmonic hierarchical nanorod array, CdS/CPDA/Au, was developed, demonstrating outstanding photoconversion efficiency and stability due to Schottky rectification and localized surface plasmon resonance-enhanced light absorption. These advancements mark significant progress in the field, offering promising avenues for the development of highly efficient and stable PEC devices, crucial for the future of sustainable energy production.

2:00 PM

(ICACC-S17-017-2024) Advanced Ceramic Materials and Processing for Photonics and Energy (Invited)

R. Narayan*[1]

1. North Carolina State University, USA

Two photon polymerization offers significant benefits over conventional processes for scalable mass manufacturing of medical devices, particularly those with small-scale features. The raw materials (e.g., inorganic-organic hybrid materials and acrylate-based polymers) used in two photon polymerization are obtained at low cost and are readily accessible. Two photon polymerization can be established in a typical “dirty” manufacturing environment; no cleanroom facilities are needed. Two photon polymerization is a straightforward and single-step method for producing complex structures with small-scale features. Two photon polymerization was shown to be able to create microneedles with a larger range of shapes and dimensions than commonly used microneedle fabrication techniques. For example, a hollow microneedle created out of an inorganic-organic hybrid material was used to create pores in porcine skin for drug delivery. Several types of microneedles and other types of medical devices that were fabricated out of inorganic hybrid materials using two photon polymerization will be discussed.

2:30 PM

(ICACC-S17-019-2024) Silicon-based Thin Film Structures for Photonic and Photovolatic Applications (Invited)

P. Mascher*[1]; P. Bhattacharyya[1]; B. Ahammou[1]; F. Azmi[1]; R. Kleiman[1]

1. McMaster University, Engineering Physics, Canada

Silicon nitride (SiNx) based films have been recognized as essential dielectric films in the microelectronics and optoelectronics industry due to their desirable properties, such as high electrical insulation, excellent thermal stability, and compatibility with integrated circuit fabrication processes. They are also a potential candidate for fabricating wavelength-selective reflective coatings and surface passivation layers in building-integrated photovoltaics (BIPV) technologies. SiNx-based films are one of the popular choices for anti-reflective coatings in photovoltaics as well. Recently, SiNx and oxynitride (SiOyNx) based thin film optical filters have been explored to provide distinct color rendering to solar-charged EVs. In this talk, we will present studies of the influence of processing and fabrication conditions on the optical and mechanical properties of SiNx and SiOyNx films, using an ECR-PECVD. reactor, which allows tuning of the refractive index and mechanical properties of the films. We will also demonstrate the design and fabrication of multi-layer notch filters for photovoltaic applications, with a reflection peak of around 73% from 350 to 467 nm with a FWHM of 79 nm along with less than 10% reflection for longer wavelengths of the remaining high transmission region of 300–1200 nm.

3:20 PM

(ICACC-S17-020-2024) Advances in the Development of Ceramics for Solar Fuel Generation (Invited)

O. K. Varghese*[2]; D. Waligo[2]; D. Rana[2]; J. Napagoda[2]; L. Schaffer[2]; M. Paulose[1]

1. University of Houston, Department of Physics, USA
2. University of Houston, Department of Physics & Texas Center for Superconductivity, USA

Ceramics are key materials for the renewable fuel generation strategies such as solar thermochemical, photocatalytic/photoelectrochemical (PEC) and photovoltaics assisted electrochemical water splitting and carbon dioxide reduction. High thermal, mechanical and chemical stability, abundance of constituent elements and low cost make many ceramic materials highly sought after for these applications. Solar thermochemical processes use these ceramics to absorb concentrated solar energy and undergo redox cycles, solid-oxide electrolyzers consist of ceramic electrolytes to transport charges for electrochemical reactions and PEC devices use semiconducting metal oxides to absorb solar energy and perform redox reactions. In principle, solar PEC process is the most promising as it permits direct conversion of solar energy to value added products at ambient conditions; however, it is not a mature technology. Development of novel nanostructured photocatalysts that are stable and efficient is crucial for solving the problems with the technology. This presentation is aimed at giving an overview of the major solar fuel generation technologies employing ceramics and discussing in detail the experimental and simulation approaches to develop new ceramic photocatalysts for PEC water splitting.

3:50 PM

(ICACC-S17-021-2024) Lead-sulfur interaction to induced water stability in formamidinium lead triiodide (Invited)

S. Ahmad*[1]; M. P. Usman[1]; E. Ruiz[2]; S. Kazim[1]

1. BCMaterials, Basque Center for Materials, Applicaiton and Nanostructures, Spain
2. Universitat de Barcelona, Spain

Formamidinium lead triiodide perovskite (FAPI) is now endorsed as a material of choice to avoid thermal degradation and phase segregation due to its higher thermal and phase stability. Sincere research efforts are being laid to stabilize thin films of FAPI, which are free of methylammonium (MA) and bromine (Br). It is the most promising perovskite photoactive material for solar cell fabrication. However, despite these significant efforts, pure FAPI thin film exhibits phase transition issues due to its thermodynamically stable non-perovskite phase (2H). To address this issue, we employed a multifunctional additivisation technique, in which the sulfur heteroatom changes from the 2H phase to an intermediary phase that is similar to the cubic phase. The produced micron-sized grains of FAPI display higher stability against high relative humidity, water intrusion, and shelf life aging. Our findings from theoretical and experimental studies support the notion that Pb...S interaction is essential for preserving the cubic perovskite phase and the stoichiometric distribution of elements.

4:20 PM

(ICACC-S17-022-2024) A Photonics Framework for Thermophotovoltaics (Invited)

M. Leite*[1]

1. UC Davis, Materials Science and Engineering, USA

Thermophotovoltaics (TPV) represent a promising route for converting heat 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. Thus, we focused on dual-layer emitters that could be scaled up. We screened the optical response of >2,800 material combinations with melting point >2,000 °C, comprising refractory silicides, borides, carbides, nitrides, etc. The mismatch in permittivity allowed for emission control, key for the development of high performing TPV. We found emitters for TPV with theoretical efficiency >50% for GaSb solar cells [1]. To complement our analysis, we also identified optimal material combinations for InGaAsSb, InGaAs, Ge, GaSb, and Si solar cells. We also quantify the response of B4C/AlN using in situ, high-temperature optical measurements [2]. The discussion of the best material options will be accompanied by the optical characterization of selected materials up to 1500 °C, using in situ reflection and transmission measurements.

4:50 PM

(ICACC-S17-023-2024) Novel carbon-ceramic electrospun fibers combining a high oxidation stability with low thermal and high electrical conductivities

J. Denk*[3]; X. Liao[1]; T. Tran[2]; S. Agarwal[1]; S. Schafföner[3]; G. Motz[3]

1. University Bayreuth, Chair of Macromolecular Chemistry II, Germany
2. University Bayreuth, Physical Chemistry I, Germany
3. University of Bayreuth, Ceramic Materials Engineering, Germany

In the present study, fibers and nonwovens in the nanometer scale composed of polymer blends of polyacrylonitrile (PAN) and different amounts of a commercially available organosilazane were processed via electrospinning. After the stabilization of the polymer fibers, the subsequent pyrolysis led to ceramic C/SiCON fibers. Various analytical methods such as FTIR, WAXS and solid state NMR were used to investigate the microstructure of the resulting material. It was mainly composed of a amorphous carbon phase, with embedded amorphous SiC_xO_y, SiN_xO_y, and SiC_xN_y ceramic nanophases. With HAADF and STEM-EDS the elemental distribution and the nanostructure was confirmed. The ceramic nonwoven had an excellent foldability, mechanical flexibility and oxidation stability. Additionally, it had unique electrothermal properties with a simultaneously high electrical conductivity of 4.2 S/cm combined with a low thermal conductivity of 19.8 mW / (mK). Whereas the carbon phase was attributed to the high electrical conductivity, the ceramic phases were responsible for the low thermal conductivity by boundary scattering in the obtained “sea-island” nanostructure. This special combination of properties makes this material unique and thus attractive for applications such as smart textiles, heating systems or oxidation and flame stable reinforcing fibers.

S18 Ultra-High Temperature Ceramics

SYMPOSIUM 18: Novel Processing Methods

Room: Coquina A
Session Chairs: Jon Binner, University of Birmingham; Adam Peters, Johns Hopkins University

1:30 PM

(ICACC-S18-017-2024) Reactive laser synthesis of ultra-high-temperature ceramics HfC, ZrC, TiC, HfN, ZrN, and TiN for additive manufacturing (Invited)

A. B. Peters*[1]; C. Wang[1]; D. Zhang[1]; A. Hernandez[1]; D. Nagle[1]; T. Mueller[1]; J. B. Spicer[1]

1. Johns Hopkins University, Materials Science and Engineering, USA

Ultra-high-temperature ceramics (e.g HfC, ZrC, TiC, HfN, ZrN, and TiN) are optimal materials for applications that require extreme temperature resilience (M_p>3000°C), resistance to chemically aggressive environments, wear, and mechanical stress. Processing UHTCs with laser-based additive manufacturing (AM) has not been fully realized due to a variety of obstacles such as slow diffusion, poor densification, and laser-induced microcracking. To circumvent the issues, we present an alternative net-shaped approach to producing AM-UHTCs by synthesizing the non-oxide material in-situ (during laser processing), rather than processing a UHTC feedstock material directly. By employing selective laser reaction sintering (SLRS), we demonstrate how laser-induced reactions between reactive precursors (metals and oxides) and a gaseous atmosphere (e.g. CH_4, NH_3) can be used to simultaneously synthesize and reaction-bond UHTC-AM layers. We examine experimental results and computational models of C and N diffusion in host transition metal lattices to elucidate how laser processing parameters might be optimized to favor reaction bonding for robust layer formation with >99.9 wt% yield. While SLRS presents a host of processing considerations, we demonstrate how SLRS may be viable for the AM of near net-shape UHTC materials that are not readily produced using current methods.

2:00 PM

(ICACC-S18-018-2024) Pressureless sintering of chopped carbon fiber reinforced zirconium diboride from additive manufacturing

J. Kaufman*[1]; L. M. Rueschhoff[2]; C. Wyckoff[1]

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

Ultra-high temperature ceramics (UHTC) are a critical class of materials for aerospace applications because of their high operating temperatures and survivability in extreme environments but suffer from inherent brittle behavior. Chopped carbon fibers have been explored as a reinforcement phase but careful consideration must be given to sintering conditions to avoid damage to the fibers. This work focuses on pressureless sintering of zirconium diboride with chopped carbon fibers made via additive manufacturing to obtain near-net shapes with aligned fibers. Characterization of microstructure (X-ray computed tomography, SEM), and mechanical properties will be presented and compared to samples made via conventional processing (pellet pressing). Deep learning-based auto-segmentation of X-ray computed tomography (XCT) of the carbon fiber in the ZrB_2 to study fiber alignment as a function of fiber loading and quantification of fiber spacing will also be presented.

2:20 PM

(ICACC-S18-019-2024) Synthesis of monodispersed ZrC nanoparticles derived from MOF-801

Y. Zou*[1]; H. Lee[1]; S. Lee[1]

1. Korea Institute of Materials Science, Republic of Korea

Zirconium carbide (ZrC) is a promising ultrahigh temperature ceramic (UHTC) due to its high melting point, strength and thermal conductivity, making it an important material for harsh environment applications. Commercial ZrC powders are conventionally synthesized via carbothermal reduction of ZrO_2, which requires high temperature and long reaction time. However, these powders often exhibit large particle size and irregular shape, impeding the availability of high-performance ZrC components. Thus, exploring novel approaches for the synthesis of ZrC particles with controlled size and shape is significant. As a Zr-based metal-organic framework, MOF-801 offers control over particle size and shape through simple solvothermal reactions. Its porous structure enables effective absorption and uniform distribution of the carbon source at a molecular scale, beneficial to reducing the diffusion distance, dwell temperature and duration. Consequently, nanosized ZrC powders with enhanced properties may be available. In this study, we successfully synthesized monodispersed ZrC nanoparticles via carbothermal reduction using MOF-801 and sucrose. The effects of the sucrose amount, synthesis temperature and holding time on resulting ZrC powders were investigated. The results show that ZrC nanoparticles ranging from 100 to 150 nm were achieved at 1500 °C. The sintering behavior of synthesized ZrC powders was also discussed.

2:40 PM

(ICACC-S18-020-2024) Polymer-Derived High and Ultra-High Temperature Ceramic Matrix Composites (Invited)

J. Binner*[1]; M. Younas[2]; E. Zancan[2]

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

Polymer derived ceramics (PDCs) are an advanced class of ceramics that can be used to manufacture both high and ultra-high temperature ceramic fibre reinforced, ceramic matrix composites (CMCs). This talk will present two different aspects of the work, one focused more on resistance to high temperatures – in the range of 1600 – 2000°C; and the other for ultra-high temperature resistance – over 2000°C. The first relies on incorporating a hydrogel-based PDC that will act as an 'internal environmental barrier' so that a coating, which can be damaged, can be avoided. The second is based on the incorporation of hafnium-doped silicon carbonitride precursors to a carbon fibre / zirconium diboride-based composite.

3:30 PM

(ICACC-S18-021-2024) Infiltration of Porous Ultra-High Temperature Ceramics for Active Cooling

A. J. Kaplan*[1]; C. Tallon[1]

1. Virginia Tech, Materials Science and Engineering, USA

Space and hypersonic travel create extreme thermal and ablative conditions. One of the classes of materials that can survive these conditions is ultra-high temperature ceramics (UHTCs) due to their uniquely high melting temperatures, thermal resistance, and mechanical strength. With recent developments in the manufacturing processes of UHTCs allowing for the formation of complex geometries and intricate microstructures/architectures, interest in UHTC-based TPS and active cooling strategies has grown. 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. In this work, homogenous aligned porous structures in UHTC materials were developed with sample porosities up to 70%, and pore sizes ranging from 5 to 17 μm by controlling the freezing front formation at -80°C at various solid loadings. The microstructure was characterized using SEM and the porosity connectivity was assessed using infiltration of a liquid phase. The effect of porosity on the mechanical properties of UHTCs was assessed both experimentally and computationally.

3:50 PM

(ICACC-S18-022-2024) Processing of Nb-coatings on ZrB_2 and C-ZrB_2/SiC composites

J. E. Förster*[1]; A. Vinci[2]; D. Sciti[2]; R. Naraparaju[1]

1. DLR - German Aerospace Center, Institute of Materials Research, Germany
2. CNR - ISSMC, Italy

Ultra-high temperature ceramics (UHTCs) are a highly investigated group of materials, due to its combination of a high melting point and a high electrical/thermal conductivity. Therefore, these materials have a high potential for the space- and aerospace industry as thermal protective systems (TPS) and leading edges of hypersonic vehicles. Their mechanical properties were largely improved by introducing C fibers in to the matrix. It was also known that the addition of certain amount of SiC in to the ZrB_2 matrix will also help in improving their oxidation resistance at elevated temperatures. The formation of a borosilicate layer at the surface of C-ZrB_2/SiC provides reduced oxidation kinetics up to 1650°C. Unfortunately, the evaporation of B_2O_3 from the borosilicate leaves a less protective layer of ZrO_2/SiO_2 behind for longer exposure times/elevated temperature regimes. Previous results on the functionality of metallic Nb-coatings on baseline ZrB_2 have proven that a dense mixed oxide scale of $Nb_2Zr_6O_{17}$ formation on the surface and a stable B_2O_3-based liquid solution has improved the overall oxidation behavior. This work focuses on the application of thin metallic Nb-coatings on ZrB_2 and C-ZrB_2/SiC by means of magnetron sputtering. Firstly, the requirements for coatings on top of ZrB_2-based UHTCs and UHTCMCs will be presented. Secondly, all process steps for the application of reliable Nb-coatings will be discussed.

4:10 PM

(ICACC-S18-023-2024) Optimized reactive melt infiltration approaches for preparing multiphase ceramic composites

P. Makurunje*[1]; S. Middleburgh[1]

1. Nuclear Futures Institute, Bangor University, United Kingdom

Multiphase ultra-high temperature ceramic composites have an important role in enhancing the oxidation resistance of aerospace and nuclear composites. Reactive melt infiltration is a technique for preparing ceramic composites by directing molten silicon, transition metals and/or alloys into the voids of a preform and effecting reaction. However, high processing temperatures present challenges for the reactive melt infiltration process. The present study highlights four material selection approaches for preparing state-of-the-art multiphase ceramic matrix composites while offsetting the high-temperature demands of the process. The four techniques utilise material combinations of immiscible phases, miscible phases, silicide phases and/or silicide eutectics to lower the temperature required for reactive melt infiltration. Current efforts aim at preparing complex and homogeneous microstructure preforms prior to reactive melt infiltration, minimising damage to reinforcing phases, applying rapid heating techniques, and developing in situ real-time monitoring systems. Reactive melt infiltration demands optimization of the composition of the phases incorporated and the processing temperature to be used.

S19 Molecular-level Processing and Chemical Engineering of Functional Materials

SYMPOSIUM 19: Additive Manufacturing

Room: Ballroom 3
Session Chair: Jijeesh Nair, Fraunhofer Gesellschaft

1:30 PM

(ICACC-S19-015-2024) Two-Photon Polymerization Enabled 3D Printing of Ceramics and Composites (Invited)

A. Gurlo*[1]; M. Bekheet[1]; H. Yang[1]; X. Wang[1]

1. Technical University of Belin, Germany

This work demonstrates a novel approach to 3D printing of high-performance ceramic micro-structures with sub-500 nm resolution. We have succeeded to fabricate objects with excellent shape fidelity from yttria-stabilized tetragonal zirconia polycrystals (TZP), cubic YSZ as well as high entropy $Sr(Ti_{0,2}Zr_{0,2}Hf_{0,2}Mn_{0,2}Sn_{0,2})O_3$ perovskites (HEP). The proposed approach additive manufacturing of carefully synthesized organic-inorganic hybrid resins, followed by specially designed debinding and sintering procedure. The essential aspect of this approach is the synthesis route of the hybrid resin. Here we applied a molecular-level processing approach for mixing metal cations (precursors) at a molecular level, followed by providing additives to render photopolymerization functionality. Consequently, the resin mixed with a carefully selected photoinitiator manifests readily two-photon absorption activity, forming cross-linked network while attaining sub-wavelength printing resolution. The microstructures of fabricated TZP, YSZ and HEP ceramics, i.e., phase and elemental composition and crystallite size, are characterized after various sintering temperatures. TZP, YSZ and HEP micro-objects sintered at 1200 °C demonstrate attractive optical transparency and excellent mechanical properties.

2:00 PM

(ICACC-S19-016-2024) Light as a processing tool for complex-structured polymer-derived ceramic materials (Invited)

T. Konegger*[1]; J. Eßmeister[1]; L. Schachtner[1]; K. Rauchenwald[1]

1. TU Wien, Institute of Chemical Technologies and Analytics, Austria

The controlled use of light has emerged as a highly versatile tool in processing of ceramic materials, as it allows for both spatial and temporal control over chemical reactions. This is especially true in case of polymer-derived ceramics (PDCs), owing to the straightforwardness to implement photopolymerization functionality directly within the primary starting constituent. In this contribution, two distinct pathways for the use of photopolymerization-based reactions in the structuring of PDCs will be shown. First, vat photopolymerization (VPP), which has emerged as a method of choice for 3D structuring of many ceramic materials, will be highlighted. A distinct focus will be set on exploiting distinct advantages of preceramic polymer systems, including their use in 3D-printed structural composites as well as their decoration with metal nanoparticles for heterogeneous catalysis. Special attention will be paid to photopolymerization characteristics, as they strongly define outcome and success of the whole process. The second part will focus on photopolymerization-assisted solidification templating, an innovative technique facilitating the generation of PDC-based monoliths with highly controlled and oriented pore structures. The versatility of these monolithic materials will be demonstrated in distinct current application examples, with a primary focus on the utilization of CO_2.

2:30 PM

(ICACC-S19-017-2024) Direct Ink Write Additive Manufacturing of Preceramic Polymers for Nano Ceramics (Invited)

J. J. Bowen[1]; C. Clarkson[1]; J. Ponder[1]; H. Koerner[1]; L. M. Rueschhoff[1]; C. Wyckoff[1]; J. Lewis[2]; M. B. Dickerson*[1]

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

Direct ink write (DIW) additive manufacturing is a facile technique, allowing for the production of ceramics with complex shapes and unique microstructures. Ceramic components protyped or produced by DIW have potential for use in a number of automotive and aerospace applications including brake discs. In this presentation, we will discuss the use of liquid carbon and silicon carbide precursors in the DIW process to print SiC/Si_3N_4, C/C, and porous SiOC materials. DIW ink chemistries and formulations, with respect to UV-induced cross-linking, rheology, and printability will be discussed. The interdependence of ink constituents, including block copolymer templating agents, on the final nanostructure and properties of the printed materials will be explored. Of particular note is the mechanical and thermal properties of the printed materials.

S1 Mechanical Behavior and Performance of Ceramics & Composites

SYMPOSIUM 1: Fracture mechanics, failure analysis and fractography

Room: Coquina E
Session Chairs: Michael Jenkins, Bothell Engineering and Science Technologies; Jonathan Salem, NASA Glenn Research Center

1:30 PM

(ICACC-S1-019-2024) Reflections on the NIST Guidebook: Fractography of Ceramics and Glasses (Invited)

G. D. Quinn*[1]

1. National Institute of Standards and technology, Materials Measurement Sciences Division, USA

Fractography is a powerful but underutilized tool for the analysis of fractured glasses and ceramics. The 1st edition of the NIST Guidebook on Fractography of Ceramics and Glasses was published in 2007. Its goal was to make fractographic analysis of brittle materials less an art and more an engineering practice for scientists and engineers. The 3rd edition was printed in 2020. It was 16% larger than the 2nd edition of 2016, and has 1130 figures and illustrations. It added new developments in our field, new equipment and techniques, and new case studies. It expanded some topics that were glossed over (e.g., terminal crack velocities, thermal shock) in the earlier editions. This presentation discusses how the Guide has evolved and how a 4th edition is in preparation.

2:00 PM

(ICACC-S1-020-2024) Crack Propagation in Calcium Fluoride Single Crystals

J. Salem*[1]

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

Calcium fluoride exhibits multiple failure modes: At mildly elevated temperatures, slip deformation can occur; in humid environments, slow crack growth can occur; and due to very low fracture toughness, fast fracture due to handling damage is a continuous concern. Structural design of optical components launched into space requires fracture mechanics properties. To perform component design of a calcium fluoride (CaF_2) prism, the fracture toughness and slow crack growth parameters were measured on the {100}, {110} and {111} low index planes. The fracture toughness is lowest on the {111} plane at 0.35 ± 0.01 MPaÖm with a very flat cleavage surface exhibited during both fracture toughness and strength testing. Slow crack growth was significant on the {111} plane with a power law exponent of $n = 30 \pm 8$. For engineering purposes, slow crack growth was insignificant on the {100} and {110} planes with $n > 75$. The facture surfaces have distinct patterns that are indicative of the cleavage planes. Biaxial testing with disks implies that design for general multiaxial states should be based on {111} strength and crack growth properties.

2:20 PM

(ICACC-S1-021-2024) Laminates of porous ceramic layers fabricated via SiC foaming and dense ceramic layers

T. Sawada*[1]; Y. Maki[2]; S. Ikari[2]; K. Yamamoto[1]; S. Kawai[1]; W. Nakao[2]

1. LIXIL Corporation, Japan
2. Yokohama National University, Graduate School of Science and Engineering, Japan

Laminated ceramics containing layers of pottery materials with high and low Young's moduli were developed to mimic the nacre structure of abalone shells with high resistances against static and dynamic fractures. The layers with the low Young's modulus moderated crack deflection and impact, thereby exhibiting a high fracture resistance. The ceramic pores were formed by the CO_2 gas generated through the oxidation of SiC during firing. Three-point bending strength and falling-ball impact tests were performed to evaluate the static and dynamic fracture resistances of the laminated ceramics, respectively. The dynamic fracture resistance was enhanced by elastic wave scattering owing to the difference between the Young's moduli of the dense and porous layers. The static fracture resistance was improved by the smaller curvature of the bottom layer deformation owing to the difference in Young's modulus between the dense bottom layer and porous layer above it. These findings may provide new design guidelines for pottery with high resistances to both dynamic and static fractures.

2:40 PM

(ICACC-S1-022-2024) Advanced ceramic composites reinforced with 2D nanomaterials: Unraveling interfacial shear resistance and toughening mechanisms

C. Lopez Pernia*[1]; X. Liu[2]; C. Athanasious[2]; J. Lou[3]; H. Gao[4]; N. P. Padture[1]; B. Sheldon[1]

1. Brown University, Engineering Department, USA
2. Georgia Institute of Technology, USA
3. Rice University, USA
4. Nanyang Technological University, Singapore

The capacity to develop cutting-edge ceramic-matrix materials with bespoke properties paves the way for their utilization across an extensive spectrum of sectors, including aerospace, energy, and electronics. In this sense, the field of ceramic composites reinforced with two-dimensional (2D) nanomaterials has witnessed significant attention in recent years due to their potential for enhancing and tailoring mechanical and functional properties. The main challenge that persists in this field lies in effectively controlling toughening mechanisms. Several attempts have been reported, involving the incorporation of graphene-based nanomaterials with variations in size, chemical composition and content. More recently, there has been a proposition to explore the addition of boron nitride nanosplatelets (BNNs): another 2D nanomaterial commonly referred to as "white graphene". This study delves into understanding the crack propagation behavior, unveiling the underlying mechanisms that govern fracture toughness in ceramic composites reinforced with BNNs and rGO. The work explores the role of interfacial shear resistance, along with the size and type-dependent impacts of the 2D nanomaterials, in facilitating load transfer between nanosheets and the ceramic matrix in order to highlight its critical importance in enhancing mechanical properties.

3:20 PM

(ICACC-S1-023-2024) Statistical strength characterization of metal/ceramic joints (Invited)

S. Grutzik*[1]; T. Diebold[2]; K. T. Strong[2]

1. Sandia National Laboratories, Materials and Failure Modeling, USA
2. Sandia National Laboratories, Materials Mechanics and Tribology, USA

Adhesive joining of metal/ceramic interfaces via soldering or brazing is common in fuel cells, microelectronic components, and other applications. Characterizing or predicting the strength of such joints can be very complicated. Brittle failure may occur in the ceramic due to stress concentrations from the solder or braze process, ductile failure may occur in the filler material, or failure may occur along the filler/ceramic interface. Each of these failure modes need to be addressed in a different way. Here, the focus is on the first case of brittle failure in the ceramic in the vicinity of the joint. A modified ring-on-ring specimen was developed to characterize soldered copper/ceramic interfaces and stress due to processing and loading was calculated with a finite element model. It quickly becomes apparent that a standard application of Weibull strength-scaling of neat ceramic strength data to the soldered specimen is not sufficient to accurately predict the measured failure loads. An alternative is to perform strength scaling in terms of the asymptotic stress field at the metal/ceramic interface corner. The two approaches to statistical strength scaling in various scenarios will be discussed as well as other issues such as the impact of whether or not the filler material is explicitly included in the computational model.

3:50 PM

(ICACC-S1-024-2024) Delamination in CFRP: Experimental Approach

K. Jribi*[1]; B. Azizi[1]; A. Mello[1]

1. Embry-Riddle Aeronautical University, Aerospace Engineering, USA

Barely Visible Impact Damage (BVID) in Carbon Fiber Reinforced Polymer (CFRP) composites is a critical issue in material engineering. These low-energy impacts may appear harmless but can cause hidden damage, such as tiny fissures or delaminations, compromising the material's structural integrity, reducing its load-bearing capacity, and shortening its lifespan. Our research focuses on delamination in CFRP composites, specifically investigating the stress levels required for initiation. We follow a systematic approach: Characterization: Non-destructive X-ray radiography helps detect internal delaminations, invisible through visual inspection. Severity Assessment: Detected delaminations are evaluated, assigned severity scores, and categorized by perimeter to assess their impact on composite performance. Compression After Impact (CAI) Testing: Composite samples undergo CAI testing, applying compressive forces in controlled laboratory settings to monitor delamination progression under stress conditions, leading to failure when delamination initiates. This data provides a comprehensive understanding of CFRP composites' response to diverse loading conditions. Analyzing patterns and trends aids in predicting their behavior and establishing standardized measures for evaluating composite resilience to damage, ensuring their reliability and longevity.

4:10 PM

(ICACC-S1-025-2024) Effect of Lamellar Structure Alignment on the Compressive Behavior of Freeze-Casted Ceramics

S. Sattar*[1]; O. Kravchenko[2]

1. University of Minnesota Duluth, USA
2. Old Dominion University, USA

In this study, a novel multiscale homogenized computational model is proposed to predict the effective mechanical properties and the failure behaviour of the freeze-casted ceramics under the off-axis compressive loading, and the results are validated against experimental results. The continuum damage modeling was employed to describe various failure modes. The homogenized model was developed based on the global orientation of the lamellar wall structure, taking into consideration the morphology of the material. Additionally, the local orientation of the lamellar microstructure was applied to investigate its impact on the material's effective properties and failure mode. Compression tests were conducted on freeze-casted alumina samples to determine their effective compressive moduli and strength at various loading angles. The Digital Image Correlation method was employed to measure the strain field during the experiment, attributing it to the effective misalignment of the porous structure, and validating the formation of strain localization patches in the macroscale modeling. The effective stiffness and strength obtained from the computational model compared well with experimental results. The proposed computational framework was used to analyze the effect of lamellar wall alignment within the freeze-casted ceramic on the effective compressive properties and progressive failure behaviour.

4:30 PM

(ICACC-S1-026-2024) Understanding fracture in tough alumina-based ceramic inspired by nacre

V. Vilchez[1]; S. Lightfoot[2]; P. Withers[2]; F. Bouville*[1]

1. Imperial College London, Department of Materials, United Kingdom
2. University of Manchester, Department of materials, United Kingdom

Millions of years of evolution have gifted natural materials with sophisticated hierarchical structures that enable them to resist fracture despite being primarily composed of brittle ceramics. Nacre stands out as having one of the simplest natural microstructures, resembling a brick-and-mortar wall, yet exhibiting many toughening mechanisms leading to a pseudo-ductile behaviour in tension. Today's nacre-like composites exhibit higher values of fracture toughness and improved crack-resistance curves compared to bulk ceramics. However, characterizing fracture in such materials remains a challenge, and understanding the link between the microstructure and the crack resistance is necessary to improve these materials further. We worked on characterizing fracture in nacre-like ceramics composed of alumina bricks with various ceramic interface, with the aim of determining the role of microstructure and composition on the mechanical response. We have developed analytical tools validated by finite element analysis to measure crack resistance curves of highly deflected and branched cracks. Supported by in-situ synchrotron tomography fracture testing and wedge splitting, we assessed the stress intensity mode-mixity at the crack tip and quantified the damage evolution in 3D. This analysis reveals important lessons on the effect of the microstructure and mortar properties to guide the improvement of these materials.

4:50 PM

(ICACC-S1-027-2024) The effect of oxidation on the flexural strength of AlN - A Weibull analysis

A. Klein*[1]; J. Zhu[2]; M. J. Whiting[1]; R. A. Dorey[1]

1. University of Surrey, School of Mechanical Engineering Sciences, United Kingdom
2. Dyson Technology Ltd, United Kingdom

A high thermal conductivity, low thermal expansion and high electrical resistivity make aluminium nitride (AlN) suitable for use in high temperature heater applications. However, in air, AlN oxidises at temperatures above 800 °C. While this should not significantly impact the electrical performance of the AlN, where the oxide layer is thin with respect to the dimension of the bar, the impact on mechanical properties, and hence long-term resistance to mechanical and thermal loading is less well understood. AlN bars, manufactured from laminated tape-cast AlN (containing yttria and alumina sintering aids), were pressurelessly sintered at 1790 °C in an inert atmosphere. Sets of 30 bars were oxidised at temperatures between 800 °C and 1200 °C, for up to 12 hours. After oxidation, the flexural strength was determined via 4-point bend testing. Weibull analysis was used to describe the variation in the strengths within a given thermal oxidation condition. A non-oxidised sample set had a characteristic strength of 259 MPa. With oxidation at 1200 °C, a decrease in strength with oxidation time is reported, down to 216 MPa after 12 hours. A maximum strength was observed in samples oxidised for 12 hours at 900 °C, of 287 MPa. The Weibull moduli of samples subjected to different thermal oxidation conditions varied between 5 and 10, with no discernible trends in the moduli with respect to oxidation parameters.

S3 21th Intl Symp on Solid Oxide Cells Materials Science & Technology

SYMPOSIUM 3: Proton conducting ceramic cells

Room: Ballroom 1-2
Session Chairs: Eric Wachsman, University of Maryland; Xingbo Liu, West Virginia University

1:30 PM

(ICACC-S3-017-2024) Improving the faradaic efficiency in protonic ceramic electrolysis cells (Invited)

S. Ricote*[1]; H. Zhu[1]; R. J. Kee[1]

1. Colorado School of Mines, Mechanical Engineering, USA

The faradaic efficiency, which is proportional to the ratio of the net hydrogen molar flux over the imposed current density, is an important performance metric for Protonic Ceramic Electrolysis Cells (PCECs). It is commonly accepted that the faradaic efficiency increases with increasing steam partial pressure, decreasing oxygen partial pressure on the steam electrode, and decreasing the operating temperature. These variations in operating conditions reduce the electronic contribution to the protonic-ceramic membrane conductivity. However, despite general agreement on the definition, there are significant (even qualitative) discrepancies in the literature about how the faradaic efficiency depends on current density. After an overview of the literature on PCECs, a physics-based Nernst-Planck model will be presented to explore the faradaic efficiency dependence on operating parameters such as temperature, pressure, and gas compositions. Finally, challenges with measuring experimentally the faradaic efficiencies will be discussed.

2:00 PM

(ICACC-S3-018-2024) Recent advances in the development of proton-conducting ceramic cells for electrolysis and co-electrolysis (Invited)

M. E. Ivanova*[1]

1. Forschungszentrum Juelich, Solid Oxide Cells, Germany

The energy transition targets amplify the need for economically viable technologies for hydrogen production and CO2 valorization. Proton conducting ceramic cells (PCCs) as a novel and challenging technology have the potential to operate at temperatures lower than that of the conventional solid oxide cells (SOCs) and additionally offer further advantages in terms of techno-operational and integration perspective. Cells based on the class of proton-conducting ceramics, such as for example $BaZr_{1-x}(Ce,Y)_xO_{3-d}$ (x=0.3-0.5, BZCY) have been developed by industrially relevant processing techniques and their performance was tested in electrolysis and fuel cell modes. Their viability as co-electrolysers for CO2 valorization to added value products was explored as well.

2:30 PM

(ICACC-S3-019-2024) 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.

3:20 PM

(ICACC-S3-020-2024) Multiscale structuring for performance enhancement of protonic ceramic fuel cell (Invited)

K. Bae*[1]

1. KENTECH, Republic of Korea

The high operating temperatures (700–1000 °C) of solid oxide electrochemical cells (SOCs) cause many problems in their practical use such as fast thermal degradation and consequent limited-material use. Protonic ceramics (PCs) have drawn much attention in this regard: their higher ionic conductivity and lower activation energy in the ion transport than those of SOC materials were expected to effectively decrease the temperature without loss of performance. The current works also emphasize the promising aspects of PCs with an example application of fuel cells. Unlike the expectation in performance enhancement at lowering temperatures, protonic ceramic fuel cells (PCFCs) are still suffering from much low power outputs. It is mainly due to poor chemical stability and/or difficult manufacturability. To overcome these notorious characteristics of PCs, thin film deposition was introduced into the fuel cell fabrication, along with a multiscale anode support structure is suggested, which is considered as more suitable to grow and to uphold the thin film elements. Under the suggested cell structures, thin and single-grain columnar electrolytes were grown where the impeding grain-boundary is minimized. Details of the fabrication process and the electrochemical performance are going to be discussed in the presentation.

3:50 PM

(ICACC-S3-021-2024) Advancements in Additive Manufacturing of Protonic Ceramic Fuel cells

M. Asghar*[1]

1. Tampere University, Renewable Energy Technologies Group, Faculty of Engineering and Natural Sciences, Finland

Protonic ceramic fuel cell (PCFC), alternatively referred to as proton-conductive solid oxide fuel cell, is a promising low-temperature (400-650°C) fuel cell technology. Additive manufacturing has the potential to revolutionize PCFC manufacturing, as it enables fabrication of both dense and porous structures with desirable mechanical and electrochemical properties. In this study, we employed extrusion-based 3D printing to fabricate PCFCs, employing $BaZr_{0.3}Ce_{0.5}Y_{0.15}O_{3-\delta}$ and $BaZr_{0.4}Ce_{0.4}Y_{0.1}Yb_{0.1}O_{3-\delta}$ as electrolyte materials, NiO as the anode, and $BaCo_{0.4}Fe_{0.4}Zr_{0.1}Y_{0.1}O_{3-\delta}$ and $Ba_{0.5}Sr_{0.5}Co_{0.8}Fe_{0.2}O_{3-\delta}$ as the cathode materials. For comparative analysis, we also 3D printed fuel cells using oxide-conductive materials with doped-ceria as the electrolyte. We investigated the rheological properties of the printable pastes using various methods, including dynamic light scattering, viscometry, tensiometry, differential scanning calorimetry, and thermal gravimetric analysis. We also characterized the cells using current-voltage measurements, electrochemical impedance spectroscopy, and various spectroscopic and microscopic techniques to understand the underlying mechanisms. Finally, we conducted a systematic study to optimize the sintering temperature during cell fabrication for optimal fuel cell performance and investigated degradation mechanisms to improve stability of the cells.

4:10 PM

(ICACC-S3-022-2024) Alternative production processes for proton conducting SOC

A. Bartoletti[1]; a. Sangiorgi[1]; E. Mercadelli[1]; A. Gondolini[1]; A. Sanson*[1]

1. CNR-ISSMC, Italy

Solid Oxide Cells (SOCs) are considered key devices for pushing the development on the hydrogen economy as a consequence of their high efficiency, fuel flexibility and modularity. In this area proton conductors represent a fore-front materials for both fuel cell and electrolyzers. One key point of the entire technology is the production process of the cells. Alternative shaping processes able to considerably reduce the manufacturing costs and time, increase reliability are however required to widespread the technology. The work addresses the main issues connected to the production of a half protonic cells using direct ink writing (DIW) or a combination of screen printing and freeze casting examining the main parameters important for the realization of a porous/dense bilayer. For the DIW technique, different inks preparation methods were evaluated to obtain high solid loading (> 40 vol%) "green" suspensions (water based or solvent-free). The optimization of drying, thermal post printing treatments leaded to BCZY-based supports with a high degree of engineered porosity after sintering. On the other hand, an ad-hoc development of water-based BCZY suspensions suitable for freeze-casting as well as a proper screen printing ink, leaded to the production of a robust porous/dense BCZY backbone with high level of unidirectional porosity and a gas-tight electrolyte.

4:30 PM

(ICACC-S3-023-2024) Joining and integration of protonic ceramic electrolysis cell

S. Anelli*[1]; D. Ferrero[2]; D. Schmider[3]; J. Dailly[3]; M. Santarelli[2]; F. Smeacetto[1]

1. Politecnico di Torino, DISAT, Italy
2. Politecnico di Torino, DENERG, Italy
3. European Institute for Energy Research (EIFER), Germany

Protonic ceramic electrolysis cells (PCECs) operate advantageously at an intermediate temperature range (400-600 °C), and they can produce pure and dry H_2 during operation, potentially at elevated pressure. The scale-up of this technology is still under development as only a few studies report PCEC operations beyond the lab-scale level. Aiming to the gas tightness of a PCEC stack assembly, the utilization of glass-ceramic materials as sealants is a viable solution. However, the thermomechanical compatibility between sealants and the cell materials is a critical issue, due to the thermal and chemical expansion/contraction of the $Ba(Ce,Zr)_{1-y}M_yO_{3-\delta}$ (BCZM) electrolyte in dehydrated and hydrated conditions. The thermochemical compatibility of different glass-based systems has been studied with a $BaCe_{0.7}Zr_{0.2}Y_{0.1}O_{3-\delta}$ (BCZY721) electrolyte and with two typical ferritic stainless steels used as interconnects in SOEC (i.e., Crofer22APU and AISI441). The joined samples have been thermomechanically characterized in both dry and moist conditions. Postmortem analyses (FESEM and XRD) on the joined samples, to assess their interfacial behavior, are presented and discussed.

4:50 PM

(ICACC-S3-024-2024) Shrinkage analysis of free-standing tapes used for BZCY-based proton-conducting electrolysis cells

L. Schäfer*[1]; R. Muecke[1]; Y. Zeng[1]; M. E. Ivanova[1]; N. H. Menzler[1];
O. Guillon[1]

1. Forschungszentrum Jülich GmbH, IEK-1, Germany

Proton-conducting electrolysis cells hold promise for efficient green hydrogen production. The successful transition from lab-scale research to large-scale cell production necessitates a comprehensive understanding of their sintering behavior. This understanding is pivotal for consistently achieving the desired microstructure and mitigating defects such as warpage, delamination, and cracks. This study investigates sintering and shrinkage in tape-cast half-cells featuring a BZCY ($BaZr_{0.7}Ce_{0.2}Y_{0.1}O_{3-\delta}$) electrolyte and a BZCY-Ni(O) cermet electrode. Traditionally, shrinkage analysis has predominantly focused on pressed pellet samples despite substantial differences in microstructure and geometry to tape-cast samples. This disparity has left the sintering behavior of the tape configurations understudied. This research fills this gap by conducting an in-depth examination of shrinkage in free-standing tapes, including both bilayers (comprised of an electrolyte and an electrode) and single-layer tapes, offering valuable insights into this less-explored area.

S6 Advanced Materials and Technologies for Rechargeable Energy Storage

SYMPOSIUM 6: All-solid-state batteries III

Room: Ballroom 5
Session Chairs: Jelena Popovic-Neuber, University of Stavanger;
Martin Finsterbusch, Forschungszentrum Juelich

1:30 PM

(ICACC-S6-016-2024) NASICON-based materials, the perfect match for the next generation of batteries? (Invited)

J. Chotard*[1]; P. Canepa[2]; V. Seznec[1]; E. Mahayoni[1]; A. Tieu Jue Kang[2];
K. Choudhari[1]; S. Park[1]; C. Masquelier[1]

1. University de Picardie Jules Verne, LRCS, France
2. National University of Singapore, Singapore

NASICON-based materials have a regain interest in the last decade. Firstly known for their high ionic conductivity, their chemical versatility allow them to be used as electrode materials as well as as coating materials for different battery technologies (Li-ion, Na-ion, multivalent-ions and all-solid-state batteries) by tuning its composition. Indeed, the NASICON crystal structure of general formula $A_xMM'(XO_4)_3$, allows a wide range of chemical substitutions. Among them, the vanadium phosphate $Na_3V_2(PO_4)_3$ (NVP) is of particular interest because of its fantastic rate capacity and thermal stability, with a theoretical capacity of 117.6 mAh/g at 3.4 V and an energy density of 396 Wh/kg. Because of the wide oxidation range of Vanadium, NVP can be used either as an anode or a cathode. NASICON materials have also proven to be among the best solid electrolyte material for sodium All-Solid-State-Batteries (ASSBs). As an example, $Na_{3.4}Zr_2Si_{2.4}P_{0.6}O_{12}$ attains ionic conductivity as high as 10 mS/cm with 3D diffusion path. Because of those electrochemical properties, their chemical stability and compatibility, NASICON-type are perfectly matching for building All-Solid-State-Batteries. In this presentation, we will discuss the astonishing diversity of the crystal chemistry of the NASICON materials (both as electrode or electrolyte) mostly studied through operando X-Ray Diffraction as well as its use in ASSBs.

2:00 PM

(ICACC-S6-017-2024) Materials and microstructural parameters governing the mechanical stress and conductivity of all-solid-state lithium-ion-battery cathodes

O. Guillon*[1]; F. Al-Jaljouli[1]; R. Muecke[1]; P. Kaghazchi[1]

1. Forschungszentrum Juelich, IEK-1, Germany

All-solid-state lithium ion batteries are considered a promising future battery concept due to their high safety and energy density. However, they might suffer from mechanical damage induced by stresses due to volume changes of the electrode active materials constrained by the solid electrolyte, reducing available capacity during cycling. Also optimized ionic and electronic conductive pathways are required for functionality of the cathode layer. Using microstructure-based simulations, several cases are investigated in order to better understand and rationally design robust solid-state batteries. First, the impact of grain size, solid volume fraction and relative density of a LLZO/LCO composite is studied. We define governing factors for performance optimization. Second, the orientation and shape of the particles is modified, leading to additional degrees of optimization. Third, it is possible to screen combinations of electrolyte (oxide, sulfide or polymer) as well as cathode active materials (including “zero-strain cathodes”) highlighting the differences and most suitable couples. Finally, a method is introduced to determine the stresses easily from the free macroscopic strain mismatch.

2:20 PM

(ICACC-S6-018-2024) Controlled Engineering of Composite Electrode and Electrolyte for All-solid-state Na-ion Batteries

S. Dwivedi[1]; S. Vasudevan[1]; P. Balaya*[1]

1. National University of Singapore, Department of Mechanical Engineering, Singapore

Extending utilization of all-solid-state batteries in smart electronic applications necessitates the exploration of conformal, safe, and cost-effective solid cell components. However, the development of solid-state batteries faces major challenges, such as high interfacial resistance, electrode-electrolyte compatibility, and abridged electrode-electrolyte-additive particle connectivity. Therefore, rigorous analysis and modifications on the electrode and electrolytes are required to achieve cell performance comparable to traditional non-aqueous batteries. Herein, all solid-state Na-ion cell with a high storage capacity of ~105 mAh/g at 0.1C is demonstrated at 60 °C with a decent rate performance and cyclic stability. The cell comprises a polymer-ceramic composite electrolyte and a modified $Na_3V_2(PO_4)_3$ based composite electrode. The electrolyte was fabricated by incorporating the NASICON-type active ceramic filler in a Polyethylene Oxide (PEO) based matrix. To obtain compatible catholytes and analytes, various combinations of the electrode, electrolyte and additive materials were thoroughly investigated. The superior electrochemical performance of the all-solid-state Na-ion cell presented in this talk enables their use in low-volume conformal devices, maintaining critical safety standards for practical applications by avoiding flammable liquid electrolytes and metal anodes.

2:40 PM

(ICACC-S6-019-2024) Proton H^+ self-diffusion in Li-H exchanged garnet-type $Li_6La_3ZrTaO_{12}$ as directly probed by solid-state ^1H NMR relaxation

F. Stainer*[1]; M. Gombotz[1]; C. Hiebl[1]; M. Wilkening[1]

1. Graz University of Technology, Institute for Chemistry and Technology of Materials, Austria

Ceramic proton conductors are attracting more and more attention as they can be used in various applications such as fuel cells or electrolysers. For many materials, high temperatures are, however, needed (> 400 °C) to ensure a sufficiently high proton diffusivity. $Li_6La_3ZrTaO_{12}$ (LLZTO) is known for its rapid Li^+ diffusivity as has been directly revealed by ^7Li NMR measurements. By exchanging parts of the highly mobile Li^+ ions by protons through treatment of a single crystal in water or glacial acetic acid, we obtained a mixed proton–lithium ionic conductor. Here, H^+ proton diffusivity and Li^+ diffusivity have separately been studied with element specific ^1H and ^7Li NMR spectroscopy. While long-range ^7Li diffusion is slightly slowed in Li–H exchanged LLZTO, we directly observe rather high H^+ self-diffusivity, which is, however, significantly slower than Li^+ dynamics. With the help of NMR spin–lattice relaxation measurements we were able to measure local (and long-range) energy barriers (0.20(1) eV vs 0.45(3) eV) as well as the self-diffusion coefficient D_H of H^+ dynamics (1.2×10^{-15} m^2 s^{-1} at 125 °C). These encouraging results are assumed to open new directories in designing ceramics offering fast transport pathways for protons at low temperature.

S7 18th Intl Symp on Functional Nanomaterials & Thin Films for Sustainable Energy Harvesting

SYMPOSIUM 7: Nanomaterials for energy conversion, storage and catalysis

Room: Coquina B
Session Chair: Muhammet Toprak, KTH Royal Institute of Technology

1:30 PM

(ICACC-S7-001-2024) Sodium-Sulfur Batteries with Unprecedented Performances Enabled by a $CoFe_2O_4$ Catalytic Additive under an External Magnetic Field (Invited)

A. Cabot*[1]

1. Catalonia Institute for Energy Research, Spain

The electrochemical performance of room-temperature sodium-sulfur batteries (SSBs) is limited by slow reaction kinetics and sulfur loss in the form of sodium polysulfides (SPSs). Here, we demonstrate that through electron spin polarization, at no additional energy cost, an external magnetic field (M on) generated by a permanent magnet can significantly improve the SPSs adsorption capacity and reaction dynamics of a ferrimagnetic sulfur host. More specifically, we detail the preparation of a carbon nanofiber/$CoFe_2O_4$/S (CNF/$CoFe_2O_4$/S) cathode with unprecedented performance and stability at ambient temperature when M on. CNF/$CoFe_2O_4$/S cathodes with spin polarization provide unprecedented decay rates down to 0.0039% per cycle at 1.0C for 2700 cycles. We further test the performance of SSBs, which has 248 mA h g^{-1} under 1.0C after 100 cycles when M on. Furthermore, we evidence that even when removing the external magnetic field, the magnetic polarization effect persists, opening the door for practical applications. This work not only demonstrates an effective strategy to improve electrochemical performance in SSBs, but also contributes to the enrichments of spin effects in the fields of electrocatalytic.

2:00 PM

(ICACC-S7-002-2024) Towards ceramic thin-film solid-state lithium ion batteries (Invited)

A. Morata*[1]; J. Gonzalez-Rosillo[1]; F. Monteiro-Freitas[1]; B. Laurenti[1]; K. Castelló[1]; A. Tarancón[2]

1. Catalonia Institute for Energy Research (IREC), Nanoionics and Fuel Cells, Spain
2. IREC / ICREA, Spain

Inorganic thin-film solid-state Li-ion batteries (SSLBs) are attracting increasing attention as compact, stable and sustainable solution for powering small portable electronics. To date, ASSLBs based on Lithium phosphorus oxynitride (LiPON) remain unbeaten in combination with Li-metal, since their glassy nature prevents the spread of Li-dendrites. However, LiPON’s low ionic conductivity (c.a. 1 μScm^{-1}) lowers cells performance and its difficult processability hinders large-scale production. Among the next generation of ceramic electrolytes, NASICON superionic solid electrolyte $Li_{1+x}Al_xTi_{2-x}(PO_4)_3$ (LATP) is very promising due to its good ionic conductivity (c.a. 1 $mScm^{-1}$) and stability in ambient air. Despite these advantages, all-ceramic devices based on LATP have not been reported, mainly due to interfacial reactivity between components. In this work, bi-layers of (LATP) with different families of electrodes were fabricated using Large-Area Pulsed Laser Deposition (LA-PLD), and characterized by different advanced techniques including operando approaches such as spectroscopic ellipsometry. Spinel $LiMn_2O_4$ and $LiTi_5O_{12}$, and olivine $LiFePO_4$ were chosen, aiming to exploit their outstanding stability, low cost and environmental friendliness. Our results show the interface chemical stability and electrochemical performance of these bi-layers demonstrating their suitability for future thin-film SSLBs.

2:30 PM

(ICACC-S7-003-2024) High entropy alloys as electrocatalysts in zinc-air batteries (Invited)

A. Cabot*[1]

1. Catalonia Institute for Energy Research, Spain

The development of cost-effective catalysts for oxygen evolution and reduction reactions (OER/ORR) is essential for the commercialization of zinc–air batteries (ZABs). High entropy alloys (HEAs) are highly suitable candidates as they offer numerous parameters for optimizing the electronic structure and catalytic sites. Herein, FeCoNiMoW HEA nanoparticles are synthesized using a solution-based low-temperature approach. Such FeCoNiMoW nanoparticles show high entropy properties, subtle lattice distortions, and modulated electronic structure, leading to superior OER performance with an overpotential of 233 mV at 10 mA/cm^2 and 276 mV at 100 mA/cm^2. Aqueous ZABs based on this HEA demonstrate a high open circuit potential of 1.59 V, a peak power density of 116.9 mW/cm^2, a specific capacity of 857 mAh/g$_{Zn}$, and excellent stability for over 660 h of continuous charge-discharge cycles. Flexible and solid ZABs are also assembled and tested, displaying excellent charge-discharge performance at different bending angles. This work shows the significance of 4d/5d metal-modulated electronic structure and optimized adsorption ability to improve the performance of OER/ORR, ZABs, and beyond.

3:20 PM

(ICACC-S7-004-2024) TiO_2 Brookite Nanorods-Based Nanocomposites for Energy Storage and Smart Windows (Invited)

C. Xing*[1]

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

Electrochromic smart windows (ESWs) offer an appealing solution for controlling indoor lighting conditions. Existing electrochromic electrodes grapple with performance degradation, protracted response times, and low coloration efficiency. This endeavor is dedicated to the production of defect-engineered brookite titanium dioxide (TiO2) nanorods (NRs) of varying lengths, with a focus on exploring their electrochromic potential as energy storage materials. The controlled synthesis of TiO2 NRs featuring inherent defects, lower impedance, and heightened carrier concentrations profoundly enhances their electrochromic capabilities. This encompasses superior resistance to degradation, swifter response times, and an intensified coloration efficiency. Notably, TiO2 NRs, particularly those of extended length, exhibit impressive electrochromic attributes, including rapid switching speeds (20 seconds for coloration and 12 seconds for bleaching), high coloration efficiency (84.96 cm2 C-1 at a wavelength of 600 nm), and robust stability. These findings underscore the remarkable potential of TiO2 NRs, especially in their lengthier variants, for advanced applications in electrochromic smart windows, founded on Li-ion intercalation.

3:50 PM

(ICACC-S7-005-2024) Excellent performance of energy storage in $Ag(Nb_{0.8}Ta_{0.2})O_3$ ceramic via tailored relaxor-antiferroelectric state by Gd-doped

S. Tangsuwanjinda*[1]

1. Ming Chi University of Technology, International Ph.D. Program in Innovative Technology of Biomedical Engineering and medical Devices, Taiwan

Superb performance of antiferroelectric state in $AgNb_{0.8}Ta_{0.2}O_3$ ceramics can be modified through the introduction of Gd dopants, resulting in the formation of relaxor-antiferroelectric state ceramics, specifically denoted as $(Ag_{1-3x}Gd_x)(Nb_{0.8}Ta_{0.2})O_3$, where x represents the doping concentration ranging from 0 to 0.04. These modified ceramics are of interest for their potential applications in energy storage. At x = 0.01 can performed the excellent of the recoverable energy storage density (W_{rec}) of ~9.47 J cm^{-3} and the storing efficiency (η) of ~68% was exhibited at x = 0.02. Phase development controlled with ideal percentages of ferroelectric $Pmc2_1$, antiferroelectric Pnma, and nonpolar cubic Pm-3m symmetries have been performance for the superb W_{rec}. The mechanism behind the achievement of a high recoverable energy density, increased bulk density, decreased grain size, and enhanced breakdown electric field (E_b) up to 375 kV cm^{-3} was investigated using various techniques, including, polarization-electric field (P-E) hysteresis loop, dielectric permittivity, atomic vibration, and electron diffraction pattern.

S8 18th Intl Symp on APMT for Structural & Multifunctional Materials & Systems

SYMPOSIUM 8: Novel forming/sintering technologies, near-net shaping I

Room: Coquina F
Session Chair: Anne Leriche, Université Polytechnique Hauts-de-France

1:30 PM

(ICACC-S8-019-2024) Pressureless sintering of SiC ceramics containing non-shrinking TRISO particles (Invited)

Y. Kim*[1]

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

For the successful sintering of fully ceramic microencapsulated fuels (FCM) without an applied pressure, two critical issues should be overcome: (1) The sintering temperature of FCM should be lower than that of uranium-containing kernel in TRISO particles for preventing overfiring of the kernel and (2) a strategy to attack the cracking nature in SiC matrix should be developed. The addition of large non-shrinking TRISO particles (~860 μm) into sinterable shrinking SiC matrix always leads to cracking in the matrix because of the tangential tensile stress build-up around large non-shrinking particles. In the present study, a new additive system and a shrinkage compensating coating layer have been developed to overcome the first and second issues, respectively. Using the new additive composition and the shrinkage compensating coating layer, FCM pellets containing 40 vol% TRISO particles have been successfully sintered without an applied pressure. The details of the processing methods and properties of SiC matrix will be presented.

2:00 PM

(ICACC-S8-020-2024) Fabrication of high strength alumina with compressive residual stress on the surface by crystallographic orientation (Invited)

T. S. Suzuki*[1]; A. Nagase[2]; T. Uchikoshi[1]; H. Kiyono[2]

1. National Institute for Materials Science (NIMS), Japan
2. Shibaura Institute of Technology, Japan

Chemically strengthening is the method to improve the strength by introducing the compressive residual stress to the surface and is known to be very effective in increasing the strength of glass as a brittle material. On the other hand, we already reported that the multilayered structure with different orientation alternatively layer by layer can be fabricated in alumina by the combination of EPD and a magnetic field. The compressive residual stress is expected to be possible to introduce in alumina surface by this method. The CTE of the c-axis is larger than that of the a-axis in alumina. Therefore, the compression residual stress is generated in the layer where the c-axis is aligned perpendicular to the layer. The composites were produced by alternately changing the angle between an electric field and a magnetic field layer by layer during electrophoretic deposition (EPD) in a magnetic field. After EPD in the strong magnetic field, the green bodies were sintered by spark plasma sintering at 1150 °C without the magnetic field. After SPS, textured alumina was transparency. The bending strengths was more than 1GPa of the specimen with the compressive residual stress in the surface. In the contrary, it was 670MPa of the specimen with the tensile residual stress.

2:30 PM

(ICACC-S8-021-2024) Synthesis of α-MoO$_3$ dendrites for producing ^{99m}Tc by hot atom mechanism

Y. Yang*[1]; T. Suzuki[1]; N. Chu[3]; T. Do[2]; T. Nakayama[1]; K. Niihara[1]; H. Suematsu[1]

1. Nagaoka University of Technology, Japan
2. Nagaoka University of Technology, Nuclear System Safety Engineering, Japan
3. National Institute of Advanced Industrial Science and Technology (AIST), Japan

New neutron irradiation targets made of α-MoO$_3$ are thought to be candidate materials to recover high-purity ^{99}Mo and ^{99m}Tc by merely injecting water$^{1)}$. In this work, α-MoO$_3$ dendrites were synthesized to produce ^{99m}Tc by hot atom mechanism considering their unique morphologies. α-MoO$_3$ dendrites were synthesized by a thermal evaporation method using a tube furnace. Dendritic crystals of α-MoO$_3$ were irradiated for 20 minutes in KUR under a neutron flux of 3×10^{13}. Irradiated solid sample was partially dispersed in water with a ratio of 1:50. Then, irradiated solid and solution samples were taken to evaluate radioactivity and isotope ratios using the Ge detector and inductively coupled plasma mass spectrometry (ICP-MS). The mass ratio of ^{99}Mo from Ge-detector is much larger than ^{98}Mo from ICP-MS in the solution and solid samples. Hot atom mechanism was confirmed by the high solubility in water of ^{99}Mo.

S11 Advanced Materials and Innovative Processing Ideas for Production Root Technologies

SYMPOSIUM 11: Starting materials: Mining, particles, bulk, and functional materials and precursors

Room: Ponce de Leon
Session Chair: Sungwook Mhin, Kyonggi University

1:50 PM

(ICACC-S11-007-2024) Spray freeze granulation drying of nonaqueous slurry to fabricate Si$_3$N$_4$ ceramics with highly homogenous internal structure (Invited)

J. Tatami*[1]; R. Yamazaki[1]; M. Iijima[1]; S. Kawaguchi[2]; N. Kondo[3]

1. Yokohama National University, Japan
2. PRECI Co., Ltd., Japan
3. National Institute of Advanced Industrial Science and Technology (AIST), Japan

It has been reported that the defects resulting from granules prepared by spray drying degrade the strength of ceramics. Spray freeze granulation drying is one of the techniques to prepare granules from the ceramic slurry. In general, the slurry is prepared using water as a dispersion media, which is not suitable for nonoxide ceramics, such as Si$_3$N$_4$ and AlN. In this study, spray freeze granulation drying was carried out using slurries of silicon nitride and sintering aid powders prepared in a mixture of tertiary butyl alcohol and cyclohexane. The eutectic composition of the mixture resulted in the granules having a homogeneous internal structure, though the granules prepared from an excess amount of tertiary butyl alcohol and cyclohexane included larger pores. Si3N4 ceramics were fabricated using the freeze-dried granules prepared from the eutectic composition of tertiary butyl alcohol and cyclohexane. As a result, it was found that highly dense and homogeneous β-Si$_3$N$_4$ ceramics were obtained using granules prepared by spray freeze granulation drying.

2:20 PM

(ICACC-S11-008-2024) Effect of Structural Differences in Polymeric Precursors on the Properties of B$_4$C Powders

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

The requirements for more advanced technologies drive researchers to seek lighter, stronger and more durable materials. Boron carbide (B$_4$C) is one of the hardest yet lowest-density materials, which also has a high melting point, high elastic modulus, chemical inertness, high neutron absorption cross-section and excellent thermoelectric properties. This has given it a wide range of applications in the aerospace and defence industry fields. Carbothermal reduction synthesis (CTR) is the most commonly used procedure for the industrial production of B$_4$C powder. Whilst precursor materials are cheap and abundant, the final product requires grinding, which increases production time, energy usage and costs, whilst reducing powder quality. This presentation will focus on the use of sol-gel processing as a potential route to the industrial production of high-quality, energy-efficient B$_4$C powder.

2:40 PM

(ICACC-S11-009-2024) Leveraging diatomaceous earth material for ceramic reef structure via slip casting method

L. I. Cabalo*[1]; R. Agbay[1]; F. Dagaas[1]

1. Mindanao State University-Iligan Institute of Technology, Department of Materials and Resources Engineering & Technology (DMRET), Philippines

Coral reef's being a diverse entity in marine ecosystems faces a lot of problems such as degradation. One innovation to mitigate and recover damaged reefs is the use of artificial reefs. This study explored the development of a ceramic reef structure(CRS) which utilizes locally source materials such as red clay, diatomaceous earth material(DE) and treated alkali rich materials by slip casting method. Various mixtures were prepared and evaluated in terms of rheological, physical and morphological features. Formulation with 15% DE material showed 1.78 g/cm3 bulk density, and 3.33 MPa flexural strength. For formulation with highest amount of DE, exhibited the highest apparent porosity and water absorption. The ceramic reef samples demonstrated erosion resistance in seawater conditions but may require stabilization for improved durability. Microstructures on the ceramic surfaces revealed pore sizes ranging from 0.50 to 7.0 µm, with variations in pore shape. Furthermore, the study examined the biocompatibility of the ceramic reef structures by observing the attachment and growth of marine organisms. Twenty five (25) different species, totaling 1176 individual organisms, thrived in the CRS over 32 days. This study contributes to sustainable ceramic reef structures favoring promising biocompatibility, highlighting their potential as eco-friendly alternatives to natural coral reefs.

S16 Geopolymers Inorganic Polymers and Sustainable Construction Materials

SYMPOSIUM 16: Extrusion and 3D printing

Room: Coquina C
Session Chair: Ana Carolina Trindade, University of Illinois at Urbana-Champaign

2:00 PM

(ICACC-S16-009-2024) Extrudability geopolymer pastes from different metakaolin mixtures

W. Ncho*[1]

1. UNIVERSITE, IRCER, France

Additive manufacturing of geopolymers is a technology for the innovations in research and development. This work highlights the impact of the reactivity of different metakaolin mixtures on additive manufacturing by extrusion geopolymers. Extrudability tests were carried out on geopolymer pastes from different metakaolin ternaries diagram (MIM1M5 and M1M2M5) using two alkaline silicates (K and KNa) solutions. Extrudable areas were delimited. To understand the differences, the reactivity of the different metakaolin mixtures was evaluated. The results showed that for the extrudable areas, the zeta potential values of the metakaolin mixtures were comprise between -50 and -60 mV with K solution and around -60 mV with the change of solution (KNa), the energies of oligomer formation were comprise between 1.67 < extrudable area < 1.8 $KJ.mol^{-1}$ with K and 1.61 < extrudable area < 1.76 $KJ.mol^{-1}$ with the use of the KNa solution. Mechanical strengths increase around 10% from solution K to KNa. All these results are confirmed by other aluminosilicate sources, such as Argilite (clay calcined at 650°C) and mixture of several ($A650_{30}$-$M1_{70}$ and $A650_{50}$-$M1_{50}$).

2:20 PM

(ICACC-S16-010-2024) Extruded metakaolin-based geopolymers for 3D printing application

G. Masi*[1]; A. Saccani[1]; M. C. Bignozzi[1]

1. University of Bologna, Department of Civil, Chemical, Environmental and Materials Engineering, Italy

This study aims at developing metakaolin-based geopolymers by extrusion in view of 3D printing application. Metakaolin was mixed with sodium hydroxide and sodium silicate solutions to obtain optimized theorical molar ratios. Extrusion was carried out manually using a conical nozzle and by applying a commercial 3D printer. Different parameters were considered to optimize the mix design for extrusion process: - micronized fillers sourced from ceramic industry byproducts (porcelain stoneware rectifying and glaze polishing powders) added in different amount to improve the mixture rheology; - different concentrations of polyethylene glycol 600 (PEG 600) added to improve the extrusion of the mixes. Geopolymer curing was carried out at room temperature. Samples characterization firstly involved fresh properties and viscosity determination. Then, the microstructure of the hardened gel was characterized by microscopy and spectroscopic techniques. It was found that the shape, the particle size, and the amount of fillers strongly influence the viscosity of the mix at the fresh state and its extrusion. The addition of PEG 600 also shows a similar effect; however, its presence and its concentration tailoring is fundamental to avoid a too viscous mixture. Finally, the scale up from a preliminary manual approach to commercial 3D printing tests is a fundamental step to face the technical issues related to this forming technique.

2:40 PM

(ICACC-S16-011-2024) Direct ink writing of geopolymer-zeolite (in-situ) composite for CO_2 Capture

S. S. Hossain*[1]; F. Akhtar[2]

1. Luleå University of Technology, Materials Engineering, Sweden
2. Lulea University of Technology, Division of Materials Science, Sweden

Now, climate change is one of the most concerning issues in society, thus, reducing CO_2 emissions has become one of the most urgent research topics in recent decades. Consequently, the scientific community has proposed numerous strategies for CO_2 reduction, and post-combustion capture of CO_2 is one of the effective methods. This study used kaolin-based geopolymer foams fabricated by direct ink writing (DIW) for CO_2 capture. Geopolymer pastes were first designed using metakaolin powder with NaOH and sodium silicate solution, and rheological properties were optimized by adding polyvinyl alcohol (3 wt.%) aqueous solution (plasticizer). The pore size, shape, and volume of geopolymer specimens were adjusted by changing the DIW parameters and using hydrogen peroxide solution in the paste composition. Second, the DIW shapes were cured at 60 °C, and the in-situ zeolite phase in the geopolymer matrix was precipitated through hydrothermal curing at 180 °C in an autoclave reactor. The geopolymer-zeolite composite exhibited a slightly different microstructure and improved CO_2 adsorption (2.4 mmol/g at 20 °C) than the geopolymer (0.8 mmol/g). 3D printing techniques combined with geopolymer technology could be a sustainable approach for development of sorbents for CO_2 capture.

3:20 PM

(ICACC-S16-012-2024) Rheological Insights and 3D-Printability Assessment of an Extruded Ceramic-like Geopolymer

A. Gasmi*[1]; C. Pelegris[1]; R. Davidovits[1]; M. Guessasma[1]; H. Tortajada[1]; F. Jean[2]

1. UPJV, MIM- Laboratoire des Technologies Innovantes, France
2. Université polytechnique Hauts de France, CERAMATHS, France

The advancement of 3D printing has accelerated the production of intricate prototypes with cutting edge ceramic-like geopolymers which can be cured at low temperature. However, robocasting of geopolymers is a challenging task for the industrialization of the process, because of it's limited working-time window for the printing and it's correlation to rheology. This study aims into understanding the impact of incorporating "Xanthan Gum," into a metakaolin-based geopolymer binder enhanced with a tailored particle size distribution of fillers. Our investigation explores the influence of the extrusion process on the rheological properties of the printed paste. Utilizing both oscillation and shear protocols, we assess rheological characteristics to fine-tune the fabrication of printed structures and optimize the extrusion process. Experimental results spotlight an important finding: the introduction of this gelling agent significantly enhances the thixotropy and viscoelastic properties of the 3D printed geopolymer. A comprehensive printability evaluation encompasses structural stability, shape consistency and interlayer bonding. This evaluation, supported by digital image analysis, examines the geometrical attributes of the printed structures. This study builds up the insights for optimizing ceramic-like geopolymers fabrication of complex shape prototypes.

3:40 PM

(ICACC-S16-013-2024) Changes in Adhesion Strength Based on Processing and Reinforcement Variations in Geopolymer Composites

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

Metakaolin based geopolymer composites have shown significant potential for applications in 3D printing and the scaling of 3D printing for large scale structures. To evaluate the structural stability of 3D printed geopolymers, it is important to understand how interlayer adhesion changes with both processing and reinforcement-based variables. Variables such as time between deposition of layer, particle size, inclusion of fiber and solid to binder ratio were investigated. Based on tensile splitting adhesion testing, increased time between layer deposition has a negative effect on interlayer adhesion due to surface drying and incohesive bonding across the adhesion surface. In addition, the inclusion of fiber reinforcements results in a more graceful and less violent failure, but the adhesion strength values tend to vary wildly from the average of 2.05 MPa due to inconsistent failure. Non-fiber reinforced geopolymers fail more consistently with a lower average of 1.61 MPa. Lastly, geopolymers have the most optimal adhesion with a solid to binder ratio of 1.28 with an average strength value of 2.38 MPa.

4:00 PM

(ICACC-S16-014-2024) Additive manufacturing with geopolymers: Opportunities for recycling, sustainability and environmental protection (Invited)

G. Franchin*[1]; M. D'Agostini[1]; F. Gobbin[1]; H. Elsayed[1]; P. Colombo[1]

1. University of Padova, Industrial Engineering, Italy

Additive manufacturing (AM) can be a powerful shaping tool for ceramics, with the potential to unlock customized and intricate designs that are not possible with traditional manufacturing method. However, the printed products are generally still in their green state and have to undergo debinding and sintering treatments which are both time- and energy-consuming. Substituting conventional organic binders with inorganic ones allows to produce ready-to-use components with no thermal treatment required, yet still able to withstand high temperatures and moderate stresses. In particular, the use of geopolymeric binders has shown potential use in construction and environmental applications. The geopolymerization approach can be adapted to both indirect AM technologies, such as binder jetting (BJ), and direct ones, such as direct ink writing (DIW). Geopolymers can be employed not only as binders, but also as active materials with promising performances in the area of carbon capture and storage, water treatment and catalysis. This talk aims at presenting strategies to adapt BJ and DIW technologies to the use of geopolymers binders. Examples of large scale production with recycled waste material will be presented, as well as case studies for the use of smaller scale monolithic components for various environmental applications.

S15 8th International Symposium on Additive Manufacturing and 3-D Printing Technologies

SYMPOSIUM 15: Binder Jetting and Powder Bed Fusion Processes II

Room: Coquina H
Session Chair: Michael Halbig, NASA Glenn Research Center

3:20 PM

(ICACC-S15-025-2024) Additive manufacturing of carbon TPMS structures by binder jetting and powder bed fusion followed by polymer infiltration and pyrolysis

M. Pelanconi*[1]; P. Blyweert[2]; G. Bianchi[1]; V. Nicolas[2]; V. Fierro[2]; A. Celzard[2]; A. Ortona[1]

1. SUPSI, Department of Innovative Technologies, Switzerland
2. Université de Lorraine, France

This study focuses on the design, additive manufacturing, and characterization of porous carbon-based structures in the form of a triply periodic minimal surface (TPMS) with outstanding mechanical properties and oxidation resistance, combined with good electrical and thermal conductivity. Binder jetting (BJ) of graphite-carbon black powders and powder bed fusion of PA11 was used to 3D print computational models of three TPMS with different topologies and geometric porosities. Infiltration and pyrolysis (PIP) with furan resin was then performed to densify the parts. Composite materials, comprising a highly disordered carbon matrix binding well-crystallized graphite grains, were obtained. The printed and pyrolyzed samples are highly porous TPMS cylinders with diameter, height and a surface thickness of ~19 mm, ~33 mm and 0.76 mm, respectively. The samples have better mechanical properties than compressed carbon-graphite composites, as well as 3D-printed carbon produced by direct ink writing and stereolithography. High-temperature tests showed that, although the amorphous carbon matrix is more prone to oxidation than the graphite grains, the overall oxidation resistance remains exceptionally high. These properties allow for applications as Joule resistors and in seasonal thermal storage.

3:40 PM

(ICACC-S15-026-2024) Binder jetting of porous alumina preforms for pressureless melt infiltration of metals

M. Mariani*[1]; E. Mercadelli[2]; C. Galassi[1]; N. Lecis[1]

1. Politecnico di Milano, Mechanical Engineering, Italy
2. CNR-ISSMC, Istituto di Scienza, Tecnologia e Sostenibilità per lo Sviluppo dei Materiali Ceramici, Italy

Binder jetting is an additive manufacturing technique that can be used to produce complex ceramic structures, exploitable as porous preforms for composite production by pressureless melt infiltration of metals. This process is dependent on a number of factors, including the wetting between the two materials, the preform pores size distribution, the temperature gradients, and the gas evolution. This study investigated the influence on different particles size distributions and phase compositions of alumina on the printing and sintering conditions to determine possible routes to control the preform properties. Feedstocks were characterized by granulometry, scanning electron microscopy, x-ray diffraction and rheometry to evaluate the flowability and determine the optimal powder design for particles packing. The printed parts density was evaluated through subsequent thermal treatments (curing, presintering and sintering at 1700/1750 °C). The voids distribution was determined by computed tomography combined with micrographies: particles size and layerwise deposition has a significant impact on the final pores dimension and orientation. Finally, multiple infiltration strategies with steel, copper and zinc were studied to design guidelines for composite materials manufacturing, demonstrating the potential of binder jetting for the production of high-performance ceramic composites.

4:00 PM

(ICACC-S15-027-2024) Binding Mechanism of SiC During Direct Selective Laser Sintering

B. W. Lamm*[1]; O. Karakoc[1]; K. Mao[1]; T. Koyanagi[2]; J. Liu[3]; Y. Katoh[2]

1. Oak Ridge National Laboratory, Materials Science & Technology Division, USA
2. Oak Ridge National Laboratory, USA
3. PolarOnyx Inc., USA

Selective laser sintering (SLS) of SiC is an attractive option for the fabrication of objects with complex geometries. However, the binder-free or direct SLS of SiC has been limited by low as-printed densities and limited understanding of the particle binding mechanism. This study is aimed at understanding laser-SiC interactions during the fabrication of SiC tubes by SLS of pure SiC powders without sintering additives. Analysis by Raman spectroscopy, electron microscopy, and x-ray diffraction indicated that binding of SiC particles was achieved by incongruent melting of SiC. Phase evolution under laser irradiation of SiC was explained by calculated phase diagrams. This study uncovered the SLS SiC microstructure at different length scales and the relationship between processing parameters and microstructure. This research was sponsored by US DOE, ARPA-E and Office of Fusion Energy Sciences under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

4:20 PM

(ICACC-S15-028-2024) Additive Manufacturing of High-Temperature Ceramic Components via Reaction Bonding

Z. Ahmad*[1]; T. Seager[2]; D. Cheikh[2]; B. Li[2]; B. McEnerney[2]; J. P. Borgonia[2]; S. Firdosy[2]; K. Faber[3]

1. California Institute of Technology, Applied Physics and Materials Science, USA
2. NASA Jet Propulsion Laboratory, USA
3. California Institute of Technology, USA

The fabrication of high-temperature, dense materials via Laser-Powder Bed Fusion (LPBF) often encounters limitations such as thermal shock, inadequate densification, and limited light absorbance. To circumvent these challenges, this study presents a novel additive manufacturing approach that integrates LPBF with reaction bonding. The method employs a specialized powder bed composition involving aluminum oxide, an aluminum alloy, and zirconium dioxide. During LPBF, the aluminum alloy, which melts at lower temperature (~660°C) than its oxide counterpart, facilitates composite powder fusion while mitigating thermal shock. Post-LPBF, reaction bonding converts the aluminum to aluminum oxide. Additional studies focus on the kinetics of the reaction bonding process and the mechanical, chemical, and thermal properties of the manufactured components. The method shows promise for near-net-shape structures and sets the foundation for exploring broader applications for ceramic composites for improved thermo-mechanical integration in spacecraft components.

4:40 PM

(ICACC-S15-029-2024) Aqueous Slurry Development and Characterization for Multiple-Oxide Direct Ink Writing

P. Snarr*[1]; C. L. Cramer[2]; A. T. Nelson[2]

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

Direct Ink Writing (DIW) is an extrusion based additive manufacturing process capable of printing multi-oxide ceramic components. Multi-oxide DIW can potentially eliminate the need for assembly of multiple manufactured components and post processing steps such as application of coatings. However, mixing materials with different thermal properties may cause issues such as cracking upon sintering, which can adversely affect material properties. This study developed novel ZrO_2 and Gd_2O_3 bearing aqueous slurries suitable for multi-oxide DIW. Slurry constituents such as solids content, dispersants, and viscosity modifiers were investigated to determine the impact on slurry rheology. After determining the optimal modifiers, each slurry had similar rheology, allowing for uniform printing. Shrinkage behavior of components produced with varying geometries were characterized in conjunction with the resulting microstructures. Ultimately, each DIW processing step was characterized to gain understanding of how a multi-oxide DIW system may behave.

S6 Advanced Materials and Technologies for Rechargeable Energy Storage

SYMPOSIUM 6: All-solid-state batteries IV

Room: Ballroom 5
Session Chairs: Yuki Orikasa, Ritsumeikan University; Hsisheng Teng, National Cheng Kung University

3:20 PM

(ICACC-S6-020-2024) (Ca, Sr)FeO_2 Cathodes with High Capacity using Anionic Redox for All-solid-state Fluoride-Ion Batteries (Invited)

K. Yamamoto*[1]

1. Nara Women's University, Japan

All-solid-state fluoride-ion batteries are attracting attention as the next generation of storage batteries because of their theoretical high energy density. However, conventional metal/metal-fluoride cathodes have disadvantages of the power density and cyclability due to the rapid decrease in electronic conductivity and large volume change during fluorination and defluorination. To solve these problems, cathode materials that use topotactic fluoride-ion intercalation reactions, similar to electrode materials applied in lithium-ion secondary batteries, are being developed. However, these materials have the disadvantage of relatively small capacity, compared to the metal/metal-fluorides. In this study, $SrFeO_2$ and Ca-doped $SrFeO_2$ cathodes with infinite layer structure were developed as new high-capacity intercalation cathodes. The $SrFeO_2$ and optimized Ca-doped $SrFeO_2$ cathodes showed high reversible capacities of 350 and 580 mAh g^{-1}, respectively. X-ray absorption spectroscopy and resonant inelastic X-ray scattering measurements revealed that these high capacities were achieved by charge compensation including cationic and anionic redoxes. The intercalation-type cathode materials that significantly exceed the capacity of conventional lithium-ion battery cathodes were developed for all-solid-state fluoride-ion batteries using inexpensive Ca and Fe.

3:50 PM

(ICACC-S6-021-2024) In silico Design and Optimization of Abundant Energy Materials (Invited)

P. Canepa*[1]

1. University of Houston, Electrical and Computer Engineering, USA

Powering hundreds of millions of laptops, cameras, and phones worldwide, rechargeable Lithium(Li)-ion batteries have performed admirably as the linchpin technology enabling today's mobile electronics industry. Finding alternatives to the Li-ion battery appears a crucial priority in the diversification and modernization of energy storage technologies. When the life-cycle analysis is examined in the design of batteries, sodium (Na) appears attractive because it can be "harvested" directly from seawater, making Na ~50 times lower in cost than Li. An important class of phosphate electrodes and electrolytes discovered by Hong and Goodenough is the Natrium Super Ionic CONductors (NaSICONs) and chemical formula $Na_xMM'(XO_4)_3$, where M and M' are transition metals and X = Si, P and/

or S. I will show that computational materials science, in particular first-principles methodologies, can guide the design of better NaSICON electrode and electrolyte materials, with superior energy densities and improved ion transport. For example, our predictions indicate that suitably doped NASICON compositions, especially with high silicon content, can achieve high Na^+ mobilities. These findings are relevant for the optimization of mixed polyanion solid electrolytes and electrodes, including sulfide-based polyanion frameworks, which are known for their superior ionic conductivities.

4:20 PM

(ICACC-S6-022-2024) Thermodynamic and Elastic Properties of Glass-Ceramic Solid Electrolytes (Invited)

M. Rohde*[1]; C. Hausner[1]; H. J. Seifert[1]

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

The specific heat capacity is a thermodynamic key parameter which determines the thermal storage behavior and stability of materials. It can be used for the calculation of thermodynamic parameters like entropy and enthalpy which are important for the thermodynamic modeling. However, it is also related to vibrational modes which are important for thermally activated or phonon assisted conduction in solid electrolytes. In this work we have measured the specific heat capacity of NASICON structured Li and Na conducting solid electrolytes as a function of the temperature. Their elastic properties and hardness were determined using a nano-indentation technique. The heat capacity data could be modelled by applying a Debye model in order to extract the Debye frequency and temperature, respectively. We could show that the ionic conductivity can be related to the Debye frequency and also to Youngs modulus and hardness, i.e. the conductivity increases with increasing Li or Na content in the NASICON system while the Debye frequency, elastic modulus and hardness tend to decrease. This is an important result since it is often discussed and shown in the recent literature that lattice dynamics and a softer, more polarizable anion sublattice can affect the ionic transport.

4:40 PM

(ICACC-S6-023-2024) Exploiting glass formability for low-temperature assembly of all-solid-state batteries

M. Bertrand*[1]; S. Rousselot[1]; M. Rioux[1]; D. Aymé-Perrot[2]; M. Dollé[1]

1. Université de Montréal, Chemistry, Canada
2. Total Énergies, France

In a lithium-ion battery, a dense and continuous environment is crucial for the effective circulation of ions. This is especially true for all-ceramic-solid-state batteries (ACSSBs) composed of oxide-based materials that require the densification of various layered composites, primarily made of active materials (AM) and ceramic electrolytes (CE). Sintering is the main process used to transform a powder into a dense environment. However, the composite nature of the electrodes complicates this process. To overcome the limitations and prevent performance degradation, this study proposes an innovative approach to lower the sintering temperature. Our proposal involves a ceramic electrolyte that can be synthesized into a glassy state. A glass behaves as a viscous phase when heated beyond its glass transition temperature, enabling simultaneous densification and crystallization. When the glass transition temperature and crystallization temperature are lower than the sintering temperature, the process results in a denser and more conductive CE compared to an already crystallized CE that undergoes the same thermal treatment. Analysis of the crystallization kinetics of glassy LAGP revealed unique mechanisms, leading to early percolation of the lithium-ion pathway. This demonstrates potential for a denser and more conducive environment for lithium-ion movement at a significantly lower temperature.

S8 18th Intl Symp on APMT for Structural & Multifunctional Materials & Systems

SYMPOSIUM 8: Novel forming/sintering technologies, near-net shaping II

Room: Coquina F
Session Chair: Tohru Suzuki, National Institute for Materials Science

3:10 PM

(ICACC-S8-022-2024) Cold Sintering Process: A new method to develop dense and nanometric hydroxyapatite ceramic and biocomposites (Invited)

M. Kumar[1]; M. Ben Achour[1]; M. Lasgorceix[1]; R. Mincheva[2]; J. Raquez[2]; A. L. Leriche*[1]

1. Polytechnical University Hauts de France, CERAMATHS DMP, France
2. University of Mons, Lab Polymeric and composite materials, Belgium

Cold sintering process (CSP) is a non-conventional, low-energy sintering technique that promotes the densification of ceramics in the presence of transient liquids under low temperatures (≤300°C) and pressures (≤500 MPa). This new process is particularly interesting for the achievement of dense hydroxyapatite at lower temperature as usually by conventional sintering to improve the mechanical and biological properties. Additionally, it provides a new strategy for the co-sintering of ceramic and polymers. Exploiting the advantages of cold sintering, this research has aimed to densify the hydroxyapatite (HA) at nano scale as well as the co-sintering of HA/polylactic acid (PLA) based composite for bone regeneration applications. The effect of key processing parameters like the presence or not of the liquid phase and its chemistry (water, acetic acid, and phosphoric acid) in cold sintering of commercially synthesized HA powders were assessed. The influence of the process parameters on the density and microstructure of hydroxyapatite ceramics has been particularly demonstrated. In the case of composites, the influence of different compatibilizers on the homogeneity of the HA/PLA composites was examined.

3:40 PM

(ICACC-S8-023-2024) Densification, microstructure and properties of advanced ceramics sintered under ultra-high pressure (Invited)

W. Ji*[1]; Z. Fu[2]

1. Wuhan University of Technology, China
2. Wuhan University of Technology, State Key Lab of Advanced Technology for Materials Synthesis and Processing, China

High/Ultrahigh pressure sintering technology is one of the most important research scopes for advanced new ceramic materials. It can not only resolve the contradiction between high density and fine grain structure during ceramic sintering, but also lead to unique microstructure and fantastic properties. In traditional sintering theory, atomic diffusion is considered as the dominate densification mechanism in pressure sintering. But in our study, it has been found that the plastic deformation and creep etc. caused by high pressure could dramatically improve the densification. The related unique microstructure could contribute to the high properties such as hardness and strength. Based on the new phenomenon, we investigated the densification behavior, microstructure evolution and properties of typical ceramics under different pressure scales. Combined with the modeling results, the dominate densification mechanism of ultrahigh pressure and high temperature sintering technology, and the relationship between dominate sintering mechanism and properties were studied.

4:10 PM

(ICACC-S8-024-2024) Potential applications of selective laser sintering SLS for the preparation of oxide ceramics (Invited)

T. Graule*[1]; C. Aneziris[2]; S. Pfeiffer[1]

1. Empa, Laboratory for High Performance Ceramics, Switzerland
2. Technical University Freiberg, Germany

Additive manufacturing experienced a huge upsurge in industry and research in the last decades, especially for metal and polymer applications. However, the implementation of AM for ceramics is more challenging, since most of the shaping methods require an additional thermal treatment. Laser powder bed fusion could hereby play an outstanding role, since it is in theory capable to shape ceramics in a direct manner without post-processing. However, low thermal shock resistance, weak densification and low light absorptance at room temperature and strongly temperature-dependent absorptance in the visible or near-infrared range limits laser processing of oxide ceramics. Powder properties can have a strong impact on the quality and performance of the laser manufactured aluminum oxide ceramic parts. The challenge of part densification, powder flowability, laser light absorption and crack formation in laser powder bed fusion of aluminum oxide have been addressed by spray drying of customized granules. To improve the laser absorption by over eighty percent, the alumina granules were doped with colored oxide nanoparticles. Bimodal or trimodal distributions of aluminum oxide were chosen to test different particle packing theories. Dibasic ammonium citrate was found to be a suitable dispersant for all oxides.

4:40 PM

(ICACC-S8-025-2024) A new perspective on the production of traditional ceramics: Touch-free flash sintering

Z. Çetinkaya*[2]; R. Raj[1]

1. University of Colorado, USA
2. Konya Technical University, Metallurgical and Materials Engineering, Turkey

Flash sintering is an alternative to conventional sintering for sintering ceramic bodies at lower temperatures and times with less energy consumption. As an alternative to this system, touch-free flash sintering has been developed by Raj and Jalali in the last year. The touch-free flash sintering system is installed in a cylindrical reactor to examine the suspended whiteware ceramic green sample with a camera, pyrometer, and optical spectrometer. It was demonstrated that a whiteware sample suspended above a dog-bone-shaped sample (yttria-stabilized zirconia, workpiece) held at stage III of the flash under current control in touch-free flash sintering glows, luminesces, and sintered under a magnetic field. The results are presented in a map with the reactor and induction currents as the control variables. Three regimes are identified: good sintering free of defects, poor sintering, and the formation of defects. The reactor current emerges as the important variable: sintering is insufficient if it is too low, and defects form if it is too high, with good sintering achieved in the intermediate values. We are working on new developments such as touch-free flash sintering, a new method in flash sintering of traditional ceramic products, and its use in continuous production lines with machine learning.

13th Global Young Investigator Forum

13th Global Young Investigator Forum: Design and processing

Room: Coquina D
Session Chairs: Chunmei Ban, University of Colorado, Boulder; Bawane Kaustubh, College of Engineering

3:20 PM

(ICACC-GYIF-021-2024) Tailored sintering route, engineered microstructure-performance relationship and AI based property determinations in silicon nitride ceramics (Invited)

Y. Nakashima*[1]

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

This presentation reviews an advanced methodology to produce silicon nitride (Si_3N_4) ceramics with tailored microstructures and distinctive engineering characteristics. Si_3N_4 ceramics are generally composed of really complicated microstructures including elongated grains dispersed into fine grains, and grain boundary and porosities with various sizes, leading to excellent mechanical, thermal and electrical properties. Due to these unique morphologies, Si_3N_4 with enhanced thermal conductivity and mechanical strength has been expected for a practical industrial use in insulated heat-dissipating substrates for power modules. In this presentation, first, the properties of the Si_3N_4 substrates required for this application will be comprehensively summarized, in terms of four important engineering aspects: thermal conductivity, mechanical strength, fracture toughness, and dielectric breakdown strength. Second, the sintered reaction-bonded Si_3N_4 method strategically selected and resultant performances will be mentioned, in comparison with those produced by the other conventional method. Third, the detail of dielectric breakdown for Si_3N_4 will be introduced, although that has been hardly reported, in which large elongated grains work as severe defects.

3:50 PM

(ICACC-GYIF-023-2024) Ultra-rapid debinding and sintering of additively manufactured ceramics by ultrafast high-temperature sintering

S. Bhandari*[2]; C. Manière[3]; F. Sedona[1]; E. Bona[1]; V. M. Sglavo[1]; P. Colombo[2]; L. Fambri[1]; M. Biesuz[1]; G. Franchin[2]

1. Department of Industrial Engineering, University of Trento, Via Sommarive 9, Italy
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3. ENSICAEN, UNICAEN, CNRS, CRISMAT, Normandie Univ, France

In the recent years, field-assisted sintering techniques have gained popularity as a way to reduce the processing time and energy. However, most of the research study has focused on the field-assisted sintering of uniaxially or isostatically pressed powder compacts with simple shapes. Additive manufacturing (AM) allows fabricating complex geometries with fewer constraints compared to the conventional fabrication techniques. Most techniques involve the use of ceramic powders embedded in an organic binder which is typically removed through a slow thermal debinding process, representing the bottleneck of the process. Herein, we prove for the first time that ultra-rapid debinding and sintering are possible for complex 3YSZ components produced using fused filament fabrication (FFF). The printed components were first chemically debinded in acetone for 1 hour, thus removing about one-half of the binder, and then thermally debinded and sintered by ultrafast high-temperature sintering (UHS) in a single-step process (30 to 120 s). With optimized parameters, fully dense and crack-free components were obtained with tailored microstructure and nanometric grain size. This

work provides a first proof of concept for ultra-rapid processing (debinding and sintering) of additively manufactured ceramics, thereby reducing the overall processing time by 99%.

4:10 PM

(ICACC-GYIF-024-2024) Deposition of antimicrobial and antiviral composite coatings by means co-sputtering technique

A. Luceri*[2]; S. Perero[2]; R. Francese[1]; A. Civra[1]; M. Donalisio[1]; D. Lembo[1]; M. Ferraris[2]; C. Balagna[2]

1. Laboratory of Molecular Virology and Antiviral Research, University of Turin, Department of Clinical and Biological Sciences, Italy
2. Politecnico di Torino, Department of Applied Science and Technology, Italy

The need for devices with antimicrobial and antiviral properties is of paramount importance and involves numerous fields of application, ranging from healthcare to automotive, textiles, and food. The antimicrobial effect can be imparted to materials through coatings with specific properties, containing biocidal agents capable of being released and interacting with microorganisms such as bacteria or viruses, slowing down or blocking their proliferation. Among the various systems studied, nanomaterials such as silver, zinc oxide, or titanium dioxide have been extensively investigated. In this work, innovative coatings made up of silver nanoclusters embedded in ceramic or glass matrix, were developed and deposited using the PVD co-sputtering technique. This technique enables the production of thin coatings (less than 200 nm), flexible, well-adhering, that exhibit excellent antiviral and antibacterial properties towards a wide spectrum of bacteria and viruses. The presence of the glass/ceramic matrix allows for a gradual release of metallic ions in a controlled manner, preventing their dispersion into the environment. The obtained coating can be deposited on various materials and has been demonstrated to be resistant to high temperatures, allowing it to undergo sterilization processes without compromising its antimicrobial properties.

4:30 PM

(ICACC-GYIF-025-2024) Oxidation protective ceramic coatings processed by electrophoretic deposition method

E. Zanchi*[1]; G. Cempura[4]; S. Molin[2]; A. R. Boccaccini[3]; F. Smeacetto[5]

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. AGH University of Science and Technology, Poland
5. Politecnico di Torino, Applied Science and Technology, Italy

Electrophoretic deposition (EPD) offers the possibility to deposit homogeneous coatings in a few seconds and at room conditions. Thanks to the flexibility in terms of both coating and substrate materials, EPD can be used in different fields of ceramic processing. The quality of the obtained coatings can be controlled through the optimization of both the starting EPD suspensions, the deposition parameters and the coating sintering procedure. In this presentation, the flexibility and versatility of the EPD technique are discussed, focusing on our recent outcomes in obtaining spinel-based coatings against high-temperature corrosion of stainless-steel substrates. Moreover, we exploit the electrophoretic co-deposition approach to synthesize in-situ (i.e. during the deposition) doped spinels. The optimization of the post-deposition coating consolidation process ensures the incorporation of the additional elements into the spinel phase, as assessed by the XRD analysis and the in-depth TEM characterization. The obtained doped-spinel coatings present tuned functional properties, such as the coefficient of thermal expansion. Overall, implementing the electrophoretic method, with its simple, adaptable and scalable apparatus, could reduce the processing time and cost of protective ceramic coatings.

S11 Advanced Materials and Innovative Processing Ideas for Production Root Technologies

SYMPOSIUM 11: New concepts and emerging technologies for enhanced product performance I

Room: Ponce de Leon
Session Chair: Chisung Ahn, Korea Institute of Industrial Technology

3:20 PM

(ICACC-S11-010-2024) Accelerating materials research 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

Recent advances in computer technology and the development of efficient computational methods have brought innovation to materials research by utilizing computer-based methods to understand and predict material behaviors. These approaches typically use existing theoretical models to predict changes in material behaviors caused by various variables. However, these methods cannot take into account all the variables that occur during material processing. Thus, various attempts are being made to overcome these limitations by utilizing artificial intelligence (AI) to find the multidimensional nonlinear relationships hidden within the data, enabling the discovery of correlations between various process variables and material performance. By combining AI with computational materials science, it is possible to analyze and understand in-depth effects of materials composition and processing variables on material properties and performance, thus accelerating the materials research. In this talk, I will introduce the researches that my group is currently conducting to accelerate materials research by combining AI with computational materials science.

3:50 PM

(ICACC-S11-011-2024) Photocatalytic Overall Water Splitting by Heterogeneous Y^{3+}-doped CeO_2 with Characteristic Doping Structure

H. Furuno*[1]; T. Nakayama[1]

1. Nagaoka University of Technology, Japan

The practical realization of photocatalytic overall water splitting requires the development of efficient photocatalyst with visible light responsive and high activity. In previous study, we reported about heterogeneous M-doped CeO_2 (M = Sm^{3+} or Y^{3+}) that has the heterogeneous doping structure with the interface between pure CeO_2 phase and M-doped CeO_2 phase on particle surface. Neither pure CeO_2 nor pure M-doped CeO_2 had an activity for overall water splitting. On the other hand, as-reported heterogeneous M-doped CeO_2 exhibited sufficient photocatalytic activity for overall water splitting. It this study, Heterogeneous Y^{3+}-doped CeO_2 was synthesized by calcination of Y_2O_3-CeO_2 mixture via the flux method and the colloidal method. Y_2O_3 nano particle was highly dispersed on high crystallinity CeO_2 particle. Synthesized Y^{3+}-doped CeO_2 had characteristic doping structure on particle surface. This photocatalyst showed higher activity than previous Y^{3+}-doped CeO_2. It was reported, the flux method improved photocatalytic activity due to improvement of crystallinity. In addition, charge separation site (reaction site) was investigated by photodeposition. It is considered that photocatalytic activity was improved due to efficient charge separation of photoexcited charges by the characteristic doping structure and high crystallinity.

4:10 PM

(ICACC-S11-012-2024) Surface-Modified Carbon Nanotubes via pulsed laser ablation with Ultrathin Co_3O_4 Layer for Enhanced Oxygen Evolution Reaction

H. Jeon*[1]; J. Kim[2]; S. Mhin[1]

1. Kyonggi Univercity, Advanced materials engineering, Republic of Korea
2. Daegu Mechatronics & Materials Institute, Republic of Korea

Alkaline water electrolysis is essential for sustainable and efficient hydrogen production. However, the sluggish kinetics in oxygen evolution reaction (OER) requires overpotential. Recently, transition metal-based electrocatalysts has been attracted great attention for improving OER performance due to the abundant active sites and efficient charge transfer. Here, we investigate the effect of surface-modified carbon (SMC) via pulsed laser ablation in liquid media (PLAL) on electrocatalytic performance for the OER. We focus on Co_3O_4@SMC heterostructures, where an ultrathin layer of Co_3O_4 is deposited onto SMCs using a combination of PLAL and atomic layer deposition. Comparative analysis with aggregated Co_3O_4 and Co_3O_4@pristine CNTs reveals the superior OER performance of Co_3O_4@SMC. The optimized Co_3O_4@SMC exhibits the lower Overpotential, Tafel slope, and a significantly higher turnover frequency (TOF) in alkaline water splitting. The experimental results, combined with density functional theory (DFT) calculations, indicate that these improvements can be attributed to the high electrocatalytic activity of the Co_3O_4 as active sites achieved through the homogeneous distribution on SMCs. This study contributes to the understanding of SMC-based heterostructures and their potential for enhancing electrocatalytic performance in alkaline water electrolysis.

4:30 PM

(ICACC-S11-013-2024) Fabrication of Self-assembled Dendritic Silicon Carbide Arrays by Nanosecond Pulsed Electric Field

Z. Shen*[1]; W. Mita[1]; H. Saito[1]; T. Fujihara[2]; H. Cho[3]; H. Furuno[1]; Y. Takimoto[1]; T. Nakayama[1]

1. Nagaoka University of Technology, Extreme Energy-Density Research Institute, Japan
2. National Institute of Technology, Anan College, Japan
3. Hanyang University, Department of Materials Science and Chemical Engineering, Republic of Korea

The self-assembled dendritic microstructure capable of bridging the 2D/3D processing scales have a significant role in catalysis, information storage and microelectronic field. A rigorous and complex preparation process is usually required to control the crystallization of ceramics to form dendritic structure. Here, we report a facile and template-free method for the self-assembly of silicon carbide (SiC) particles into 3D dendritic structure arrays. After applying electric field to uniformly dispersed SiC particles/polymer suspension, the SiC particles were rapidly polarized and self-assembled along the direction of electric field. By controlling the contact charging between particles and electrode, a tree-like structure with a trunk along the direction of electric field and branches near the electrode was formed. The process of particle self-assembly was observed in real time by an in situ observation system, and the morphological features of the dendritic structure were investigated using multifractal analysis. The effects of different electric field, voltage and filler concentration parameters on the dendritic structure were analyzed, and the use of high-voltage nanosecond pulsed electric field was found to be beneficial in promoting the formation of dendritic structures by tight inter-particle bonding without dielectric breakdown.

S13 Development & Applications of Adv Ceramics & Composites for Nuclear Fission/ Fusion Energy Sys

SYMPOSIUM 13: Emerging and novel material technologies for nuclear systems

Room: Ballroom 4
Session Chair: Eddie Lopez Honorato, Oak Ridge National Lab

3:20 PM

(ICACC-S13-021-2024) Fabrication and Testing of Hybrid CMC-Metal Cladding Structures Made by Embedded Wire Chemical Vapor Deposition

S. Harrison*[1]; J. Pegna[1]; J. Vervlied[1]

1. Free Form Fibers, USA

Embedded wire chemical vapor deposition (EWCVD) is a technology developed at Free Form Fibers that is capable of fabricating thin shell hybrid structures which blend ceramic matrix composite and metal components. A silicon carbide-based CMC is formed by chemical vapor infiltration of thin non woven layers, utilizing SiC fibers produced by laser-driven chemical vapor deposition (LCVD), wrapped around a metal substrate. The CVI process is driven by the delivery of thermal energy through a resistively heated wire that is wound around the non woven fiber layers. This processing approach to achieve quickly formed, high purity CMC structures that are well-adhered to the underlying substrate was demonstrated for a nuclear fuel rod cladding application, utilizing a SiC fiber reinforced - SiC matrix CMC on a zirconium tube. Results from mechanical behavior evaluations will be presented, including expanding plug testing performed at elevated temperatures.

3:40 PM

(ICACC-S13-022-2024) Response of ODS-FeCrAl alloys with different Cr contents studied using transmission electron microscopy with in-situ single/dual Fe and He ion beams

J. A. Hinks*[1]; H. Le[1]; Y. de Carlan[2]; D. Hoelzer[3]; K. Sakamoto[4]; P. Persson[5]; K. Lambrinou[6]

1. University of Huddersfield, School of Computing and Engineering, United Kingdom
2. CEA Saclay, DES/ISAS/DMN/SRMA/LA2M, France
3. Oak Ridge National Laboratory, USA
4. Nippon Nuclear Fuel Development, Co., Ltd., Fuel Materials Group, Research Department, Japan
5. Linköping University, Thin Film Physics, Department of Physics, Chemistry and Biology (IFM), Sweden
6. University of Huddersfield, School of Computing and Engineering, United Kingdom

Oxide dispersion strengthened FeCrAl (ODS-FeCrAl) alloys combine resistance to both oxidation and radiation-induced swelling. They are promising fuel cladding materials for fission reactors and first wall materials for fusion reactors. However, a major challenge associated with the use of ODS-FeCrAl alloys in nuclear applications is their embrittlement under irradiation due to the formation of the Cr-rich α' phase at relatively low temperatures (<=475°C). This work reports on in-situ ion irradiation of three ODS-FeCrAl alloys with different Cr contents (10, 12, and 20 wt.%), microstructures (grain size, texture) and thermomechanical histories (plastic deformation). Each of these ODS-FeCrAl alloys has been observed using TEM whilst being irradiated at 20°C and 300°C under single (Fe or He) and dual ion beams (Fe and He) to explore the radiation response as a function of damage dose, temperature and damage dose rate. Post-irradiation examination has been conducted using high-resolution scanning TEM (STEM), EDS and EELS. The results reveal that: (a) Fe irradiation leads to α' phase precipitation at lower temperatures and higher dose rates; (b) He implantation forms larger bubbles in the oxide dispersoids and promotes intergranular precipitation of the Cr-rich α' phase; and (c) dual beam irradiation damage is Cr-content dependent.

4:00 PM

(ICACC-S13-023-2024) Post-irradiation examination of substoichiometric yttrium dihydrides for solid neutron moderator applications

M. N. Cinbiz*[1]; C. N. Taylor[1]; T. Johnson[1]; J. Charboneau[1]; I. Hobbs[1]; J. Burns[1]; G. C. Papaioannou[1]; E. P. Luther[2]; A. P. Shivprasad[2]

1. Idaho National Laboratory, USA
2. Los Alamos National Lab, USA

Solid neutron moderator technology is a key enabler for small nuclear reactors that are transportable via current transportation infrastructure. While there are multiple moderator candidate materials based on carbon or beryllium, hydrogen bearing metal hydrides, particularly zirconium or yttrium hydrides, enable a significantly more compact reactor design. With the presence of hydrogen, the main challenge is its retention in the reactor during elevated operation temperatures and neutron irradiation. This study investigated the impact of the high-temperature neutron irradiation on yttrium hydride specimens, as prepared at Los Alamos National Laboratory and irradiated at the Advanced Test Reactor at Idaho National Laboratory. The post-irradiation examination activities focused on identifying metrics to assess hydrogen loss or content, as well as thermal property measurements. Optical and electron microscopy characterizations were also performed to investigate the hydrogen-content-related structural changes in metal hydrides. Thus, potential qualification metrics for neutron-irradiated yttrium hydrides were investigated along with the comparative analysis of PIE activities.

4:20 PM

(ICACC-S13-024-2024) Thermophysical and microstructural property changes in neutron-irradiated metal hydrides

D. Sprouster*[1]; M. Ouyang[1]; Y. Huang[2]; P. Neggi[1]; A. Sharma[1]; D. Bhardwaj[1]; N. Cetiner[2]; L. Snead[1]

1. Stony Brook University, USA
2. Massachusetts Institute of Technology, USA

Metal hydrides, including Yttrium hydride and Zirconium hydride, are of particular interest for advanced thermal fission reactors as they have attractive thermal conductivities and high neutron moderator ratios. They have direct applications as core components including as matrix materials for fissile material, and as neutron moderator or shield. While the neutronic properties intrinsic to these hydrides offer promising performance, thermal and irradiation-induced property changes question their viability. Specifically, evolving metal-hydrogen ratios are of critical concern. In this presentation we discuss the post-irradiation examination of neutron irradiated YHx and ZrHx specimens. We employ multiple characterization techniques including crystallographic, morphological, and thermophysical to determine the irradiation-induced macro- and microstructural evolution as a function of neutron fluence. We quantify degradations in the thermal diffusivity and changes in lattice parameters, indicative of minor hydrogen release in both hydride materials. Additional irradiation-induced defects, including two-dimensional defects were also apparent from the detailed x-ray diffraction quantification. These results serve to improve both the fundamental understanding of neutron-solid interactions, and the development of applied structure-property evaluation of an important class of core materials.

4:40 PM

(ICACC-S13-025-2024) On the Hydrogen Stability in Ceramic Matrix Entrained Hydride Composite Shield

D. Sprouster[1]; N. Rani[1]; D. Bhardwaj*[1]; L. Snead[1]

1. Stony Brook University, USA

High Temperature Superconducting (HTS) fusion systems are being widely pursued to access higher magnetic field, allowing similar fusion power in a more compact design. These compact HTS fusion designs offer the potential for both lower capital cost and nearer-term realization, but due to the enhanced neutron wall loading and significantly reduced combined structure at the toroidal inboard region the radiation threat the HTS magnet is significant. For this reason, a new class of shielding composites have been developed under the ARPA-E GAMOW project, a fully dense composite consisting of an irradiation stable MgO-matrix with an entrained neutron-absorbing metal hydride (HfH_2) with high HfH_2 loadings (25-55 v/o). Once formed, a key issue to be discussed here is the final hydrogen loading, a key parameter in shield neutron moderation, as well as long-term thermal stability of the hydride under assumed normal and "accident" conditions. As the primary motivation for this composite is as solution to the rapid release and potential for hydrogen explosion associated with monolithic hydrides, understanding the kinetics of release under elevated temperature excursions is necessary. Thermal aging was performed at nominal operating temperatures (400°C, 600 °C), and representative accident temperatures (1000 °C) with quantitative x-ray analysis and x-tomography the primary data tools.

S19 Molecular-level Processing and Chemical Engineering of Functional Materials

SYMPOSIUM 19: Additive Manufacturing

Room: Ballroom 3
Session Chair: Aitana Tamayo, Institute of Ceramics and Glass, CSIC

3:20 PM

(ICACC-S19-018-2024) 3D-printed porous SiOC monoliths functionalized with Pd by Atomic Layer Deposition for promising heterogeneous catalysis (Invited)

C. Salameh*[1]

1. European Institute of Membranes, France

The manufacture of porous monoliths plays an essential role in the field of heterogeneous catalysis. These complex structures feature a multitude of interconnected pores, offering high surface area and exceptional mass transport properties. This enables efficient use of catalytic sites and promotes rapid diffusion of reactants, both of which are essential for catalytic processes. In particular, 3D-printing of ceramic monoliths as potential catalyst supports represent a remarkable innovation in this field providing a customizable platform for tailoring catalyst properties to specific applications. The incorporation of catalytic particles by atomic layer deposition (ALD) further enhances the potential of such materials, making them a promising choice for catalytic transformations. The combination of 3D printing and ALD not only highlights a cutting-edge technology, but also underscores the vital role of stable ceramic monoliths in the advancement of heterogeneous catalysis, paving the way for sustainable and efficient chemical processes. Here, by coupling stereolithography 3D printing of polymer-derived SiOC and ALD, we succeeded in fabricating porous 3D ceramic monoliths decorated with highly dispersed Pd nanoparticles with a low particle size distribution. We studied the effect of Pd loading on catalyst activity with a simple Suzuki-Miyaura coupling in water.

3:50 PM

(ICACC-S19-019-2024) In-situ development of nanostructured macroporous Silicon oxycarbide ceramics (Invited)

A. Choudhary*[1]; S. k. Behera[2]

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Nanostructured Silicon oxycarbide (SiOC) ceramics has gained a wide interest owing to its various application including filtration, purification and catalysis. The ease with which the surface area and porosity of SiOC can be tuned attracts researchers in the field of porous polymer derived ceramics. In this work, basically two different types of porous (SiOC) ceramics were developed, one is macroporous SiOC without any nanostructures and the other is nanostructured SiOC. Both macroporous and nanostructured porous SiOC had open porosity greater than 90 %. The former is developed using a very simple positive sponge replication technique while the latter is fabricated by a combination of sponge replication and catalyst assisted pyrolysis. To develop in-situ grown nanostructures, we incorporate various transition metal cations in the structure. It was shown that the morphology of these nanostructures could be varied by choice of different metal catalyst. Growth mechanisms were proposed based on the micro-structural evidences along with EDX and XRD analysis. Growth of nanostructures on the surface and pores of macroporous SiOC ceramics could provide basically two advantages. Firstly the surface area could be enhanced compared to neat macroporous SiOC ceramics and secondly, the nanostructures grown on macroporous SiOC ceramics could be functionalized for adsorption applications.

4:20 PM

(ICACC-S19-020-2024) Next Generation Preceramic Polymer-grafted Nanoparticles for Fabrication of Ceramic Matrix Composites

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Ceramic matrix composites (CMCs) are advantageous for aerospace-grade structural materials due to their high strength to weight ratio and performance at high temperatures (e.g., hot section turbine engine parts). One method by which CMCs are fabricated is through polymer infiltration and pyrolysis (PIP) of woven fibers using preceramic polymers slurried with ceramic powders. Because of preceramic polymer's propensity for shrinkage and a slurry's tendency to settle, our group has previously developed polymer-grafted nanoparticles using polycarbosilanes (corona) and silica nanoparticles (core). The grafted nature of the nanoparticles mitigates settling and maintains the fluidity of the material, while the nanoparticle filler helps to increase char yield and potentially limit shrinkage. Ongoing efforts to optimize these materials for higher temperature applications will be presented. Our approach falls into two main thrusts 1) substituting core materials for silica and 2) modifying polycarbosilanes to promote crosslinking in the corona of these materials.

4:40 PM

(ICACC-S19-021-2024) Kinetic Analysis of Pyrolysis of a Polysiloxane

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We perform quantitative analysis of thermogravimetric (TG) data of the pyrolysis of preceramic polymers. Our model system comprises polymethylhydrosiloxane (PMHS) cross-linked with divinylbenzene (DVB). Attention is placed on the molar ratio of Si-H on PMHS to vinyl groups on DVB. We synthesize the preceramic polymer with different amounts of solvent to create chemically similar materials with different surface areas. The pyrolysis of the siloxane is well described by a series of distinct processes. We perform a variety of traditional kinetics analyses that yield kinetic parameters of activation energies and pre-factors. We also show a preliminary analysis based on a recent development combining parallel and sequential reactions. Notably, some – but not all – decomposition processes strongly depend on surface area. Our model analysis indicates that pyrolysis in this system is a complex process involving a variety of fundamental reaction mechanisms.

S16 Geopolymers Inorganic Polymers and Sustainable Construction Materials

SYMPOSIUM 16: Use of waste materials

Room: Coquina C
Session Chair: Henry Colorado L., Universidad de Antioquia

4:30 PM

(ICACC-S16-015-2024) Prediction of chemical stability of volcanic ash-based AAMs using Artificial Neural Network (Invited)

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Volcanic ashes are known as suitable precursors for alkali-activated binder production thanks to their high amorphous aluminosilicate contents. The investigation on the reticulation degree of volcanic alkali-activated materials, AAMs, is particularly complex being the solidified product a composite material where unreacted volcanic particles as embedded in a non-crystalline matrix. Such reticulation degree has been experimentally determined in terms of chemico-physical properties: weight loss after leaching test in water, leachate characterization, and compressive strength. Additionally, Artificial Neural Network (ANN) was trained on selected experimental data and successfully applied to predict the chemical reactivity of a different class of volcanic alkali-activated materials. Excellent correlations between experimental and calculated data of the same precursor type were found. The reactivity of the starting volcanic materials in the alkaline media appeared more evident in the chemical stability tests rather than in the mechanical resistance ones.

Poster Session I- Group A presenting

Room: Ocean Center

5:30 PM

(ICACC-P001-2024) Praseodymium(Pr) Doping Effect on Ni-substituted $BaZrO_3$ Catalyst for Hydrogen Production by Partial Oxidation of Methane

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The demand for hydrogen(H_2) is increasing worldwide as hydrogen energy is being considered to solve global warming caused by fossil fuels. Partial oxidation of methane(POM) reaction that can produce hydrogen by methane(CH_4) combustion, is an exothermic reaction (ΔH = -36 kJ/mol). Nickel(Ni) is widely used as a catalyst for POM due to its economic advantages over noble metals (Pt, Pd and Ru etc.). However, catalyst degradation due to Ni aggregation and carbon coking occurs at high temperatures for POM. Therefore, it is necessary to develop a catalyst with high activity and stability at high temperatures for POM. Recently, it has been reported that praseodymium(Pr), one of the rare earth elements of the lanthanides, enhanced the catalytic activity for hydrogen production of perovskite-based oxide catalysts($LaNiO_{3-\delta}$) and prevented carbon deposition on Ni catalyst, resulting in excellent durability of the POM catalyst. Likewise, this study investigates the influence of Pr doping in Ni-substituted $BaZrO_{3-\delta}$ catalyst for hydrogen production through POM.

(ICACC-P002-2024) Investigation of Direct Recycling of Cathode Active Materials for Lithium Ion Batteries by Simple Grinding and Classification Processes

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As the demand of LiB increases with the spread of EVs, the amount of waste is also expected to be increased, and an optimal recycling process must be established. Some of the cathode materials for LiB contain rare metals, and it is inevitable to recover by environmental-friendly. In existing recycling processes, each valuable metal is recovered separately. Therefore, the process has many steps, and the cost of chemicals is high. In this study, we investigated a direct recycling process to separate and recover cathode active materials by environmental-friendly process such as grinding and classification, and made a new battery using the recovered materials. First, a sample of the cathode compound(cathode active material, binder, and conductive agent) was prepared. The samples were primary ground by a jaw crusher and were secondly ground by an attritor. Then, the sample were sieved at 32 μm. The sieved material under 32 μm was classified into coarse, medium, and fine fractions by an elbow-jet classifier. Analysis of the classified material confirmed that cathode active material was preferentially concentrated in the coarse and medium fractions. Battery cells were prepared using the coarse and medium fractions. Charge-discharge experiments and cycle tests were conducted on its to confirm that the performance was equivalent to that of the product.

(ICACC-P003-2024) 3D printed PLA-TCP-SiO_2 and PLA-TCP-MgO composite scaffolds for tissue engineering

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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) with bioactive fillers such as β-tricalcium phosphate (TCP), SiO_2 and MgO aimed to produce 3D scaffold for hard tissue repair. Filaments composed of PLA-TCP matrix (90 wt%-10 wt%) containing 1, 5, and 10 wt% of SiO_2 or MgO were produced by melted state extrusion method. Next, 3D polymer composite scaffold was printed using the fused filament fabrication (FFF) technique. The incorporation of SiO_2 did not affect the thermal properties of the PLA-TCP matrix, however the addition of MgO lead to a decreased polymer crystallinity, and molecular weight. The fast degradation induced by higher concentrations of MgO (5 and 10%) lead to an acid environment that imparted the composite biocompatibility. Biological assays using mesenchymal stem cells confirmed that the presence of SiO_2 and low concentration of MgO promoted cell proliferation. Furthermore, the bioactive fillers stimulated cell differentiation towards osteoblast, enhancing extracellular matrix mineralization, alkaline phosphatase (ALP) activity, and bone-related genes expression. Overall, the PLA-10%TCP-10% SiO_2 composite presented the most promising results, especially for bone tissue regeneration, due to its intense osteogenic behavior.

(ICACC-P006-2024) ASTM International Standards for Properties & Performance of Advanced Ceramics – ASTM Has Been Helping Our World Work Better for 125 years

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Properties and performance of brittle bodies (e.g., advanced ceramics and glasses) are challenging to measure accurately and precisely unless the proper techniques are used. After nearly five decades 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 constituents 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-P007-2024) Influence of sintering parameters on the structure of alumina tubular membranes obtained by freeze-casting

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This study represents the inaugural investigation into the comparative effects of distinct sintering methodologies on the structural characteristics of alumina specimens prepared through the freeze-casting technique. Two-stage and fast sintering procedures were juxtaposed against the conventional approach, unveiling the pivotal role of the sintering regimen in the modulation of alumina ceramic microstructures. Notably, the two-step sintering approach exhibited enhanced efficacy in constraining alumina particle growth (0.8 μm) in contrast to the conventional (1.2 μm) and fast (0.9 μm) sintering techniques. A noteworthy phenomenon emerged during the two-step sintering process, whereby a distinct "kinetic window" materialized during the second stage of thermal treatment, imparting a repressive influence on particle growth. Furthermore, with regard to mechanical properties, a discernible augmentation in flexural strength was discerned upon the utilization of a two-stage sintering protocol (6.15 MPa), ascribed to the refinement of the microstructure. These results collectively elucidate the tailoring of alumina sample microstructures through strategic alterations in the sintering route employed within the context of freeze-casting.

(ICACC-P008-2024) Life Cicle Assesment in the PIP process for manufacturing C/SiC and the consideration of Pareto efficient manufacturing methods

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Climate and environmental protection are playing an increasingly important role in our everyday lives. Companies are also increasingly being made responsible for minimizing the ecological impact of their products. Sustainability aspects as well as life cycle assessment must also be considered in the production and use of innovative materials such as the CMC. In this poster, the Pareto-efficient production of C/ SiC via PIP-process is investigated at the TomAc laboratory furnace at the University of Augsburg. For this purpose, C/ SiC test specimens were produced and characterized by varying relevant parameters with the help of a statistical experimental design. Correlations and causalities between furnace parameters, starting materials, cycle times and the mechanical performance of the material are shown. This work resulted finally in a Pareto-efficient LCA evaluation of the PIP manufacturing route.

(ICACC-P009-2024) Fracture Toughness Measurement of Ceramics Having Fuel Pellet Geometry

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Cracking of nuclear fuel pellets, driven by extreme temperature gradients, is a critical factor impacting reactor performance. However, limited fracture toughness data exists for specimens of representative geometry and fabricated using relevant processing methods. Toward addressing that limitation, monolithic ceramic specimens, having conventional fuel-pellet geometry (1 cm diameter and length "short rod"), were chevron-notched and subjected to an opening load to measure fracture toughness (K_{Ic}). Continuous Differential Interference Contrast (DIC) imaging of the crack mouth opening was combined with the measured loading response and finite element analysis (FEA) to track and interpret (per Barker's analysis) the advancing stable crack propagation to ultimately estimate K_{Ic}. The fuel-pellet-size specimens were harvested from the same billets of three monolithic ceramics (Ce-TZP, SiC, and Si_3N_4) whose K_{Ic}'s were measured using ASTM C1421. The herein chevron notch test method and analyses, the determined K_{Ic} values and their validity and repeatability of measurement, and the prospects of K_{Ic} test standardization with this fuel-pellet geometry are described. This manuscript has been authored by UT-Battelle, LLC, under Contract No. DE-AC05-00OR22725 with the U.S. Department of Energy.

(ICACC-P010-2024) Methodology for PE tube connection with heat shrinkage casing in the alignment of district heat pipe

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Heat shrinkage casing is a method used to connect district heat (DH) pipes. This process begins with the welding of the inner pipes within the DH pipes, followed by the joining of heat-shrinkable polymer tubes such as HDPE and PEX to the outer HDPE pipes using heat, compression and adhesive. Additionally, an insulating material called PUF is injected between the inner and outer pipes. The tube material undergoes a significant transformation, contracting when heated to high temperatures, which is facilitated by changes in its crystal structure. This transformation is ensuring the effectiveness of the adhesive joining. However, the working issue due to the confined workspace, leads to ununiform joining quality as workers rely on their experience and expertise. To ensure uniform joining quality, we propose the development of equipment that imitate the joining process and lead to the contraction behavior of heat-shrinkable polymers under various heating and compression conditions.

(ICACC-P011-2024) Advanced ceramics reinforced with carbon-based materials: A comparative study

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Ceramic composites reinforced with graphene or carbon nanotubes have shown improvements in properties such as: hardness, fracture toughness, and rupture strength in numerous studies. However, such gains are not uniform in all properties and can vary multifactorially, influenced by factors such as grain size and matrix composition while the reinforcing materials can contribute positively by their outstanding mechanical properties but also negatively due to agglomeration, geometry, and positioning. Additionally, density, porosity, phase transition, and sintering temperature of the composites are important parameters that must be controlled. Thus, a series of specimens was produced to encompass these variations and investigate how they can alter the hardness, rupture strength, and fracture toughness of such composites. To achieve this, the results obtained with different ceramic materials such as pure alumina, and zirconia doped alumina were reinforced with graphene with varying numbers of layers, graphene oxide, and carbon nanotubes. Carbon diffusion in the matrix, crack deflection effects, densification, and agglomeration of the reinforcing material were observed to improve the understanding of the observed mechanical properties.

(ICACC-P012-2024) Understanding the Role of Gas Phase Reactions during Surface Siliconization of Carbon Composites

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Carbon Fiber Reinforced Carbon Matrix composites with densified Silicon Carbide surface (C/C-SiC) are advanced materials known for their exceptional resistance to high temperatures and oxidation. One of the manufacturing processes to impart oxidation resistance to these composites involves converting the surface of carbon matrix into silicon carbide using Reactive Melt Infiltration (RMI). A successful silicon infiltration produces a dense silver coloured SiC surface, while a failed process results in a porous green SiC surface layer with reduced thermal resistance, prone to surface damage at high temperatures. Understanding the reasons behind failed surface siliconization and its influencing factors is vital, as it can significantly impact the composite's ability to withstand high temperatures. This study demonstrates that gas phase reactions occurring in siliconization process before the melting point of silicon are the primary cause for the greenish SiC layer. Through siliconization experiments and simultaneous thermal analysis (STA), the extent of these reactions is found to depend on the specific surface characteristics of both silicon and carbon. Microstructural differences between SiC formed from vapor and liquid reactions are compared. Based on the findings, a four-step reaction path is proposed to explain the formation of green, grey and silver surface layers during RMI.

(ICACC-P013-2024) Novel colloidal production route for all-oxide CMC by combining 3D braiding and pressure slip casting

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The development of new high-temperature turbine materials is necessary to enable the combustion of H2 in modern gas turbines and also to increase plant efficiency and reduce exhaust gas emissions. Fibre-reinforced (oxide) ceramics (ceramic matrix composites - CMC) offer great potential for turbine applications. Thanks to their high oxidation resistance in combination with excellent structural-mechanical properties at high temperatures, oxide CMCs can increase the performance of thermal processes. This enables the development of new turbine generations so that turbines fired with synthesis gases (e.g. renewable hydrogen and biogas) can be used for energy conversion with increased efficiency and low maintenance. Within the 3DOxOxTurbine project (FKZ 03EE5074C) a novel colloidal production route for near-net-shape CMC elements is investigated. While conventional fibre-reinforced ceramics have so far been manufactured to the size of a single piece in factory-like processes, the research project is combining pressure slip casting with 3D braiding. To demonstrate the overall process performance, a demonstrator of an all-oxide CMC combustion chamber cladding and a stationary turbine blade will be developed.

(ICACC-P014-2024) Electrical discharge machinable SiC composites for high temperature sliding wear applications

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While SiC ceramics are preferred for high temperature wear resistance components, challenges in processing to high density and machining to complex shapes limit applicability. In the present work, SiC- (0 to 20 vol%) TiB_2 composites with ten vol% Y_2O_3-AlN additives were developed by spark plasma sintering at a considerably lower sintering temperature of 1800 °C. Restricted $\beta \rightarrow \alpha$ SiC phase transition is observed in sintered composites which can be attributed to lower sintering temperature, nitride additives and nitrogen environment. With increase in TiB_2 content, hardness increased from ~21 to ~24 GPa. The fracture toughness increased from 3.6 to 5.5 MPa $m^{1/2}$ with increased crack deflection and crack bridging. The electrical conductivity of SiC-TiB_2 composites improved because of the conductive TiB_2, nitride-containing additives and sintering atmosphere. The composites were successfully machined to complex shapes by wire-EDM with higher material removal rate and low surface roughness. Furthermore, the presence of TiB_2 increases friction and reduces wear of SiC composites in reciprocated sliding contacts against SiC. Results reveal one order of lower wear at 600 ºC due to tribo-oxidation. Overall, SiC composites developed by strategic liquid phase spark plasma sintering in the present investigation demonstrate potential for expanding the application regime of SiC ceramics.

(ICACC-P015-2024) Comparison of the high-velocity impact performance of boron carbide ceramics

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Three different boron carbide materials, with distinct microstructures, were impacted over two high velocity ranges to study their fracture mechanisms as well as crater formation and penetration performance. The first velocity range (1-3 km/s) was used to compare the materials without penetration, and the second velocity range (3-5 km/s) served to compare the penetration performance of these materials. Initial work by Malhotra and colleagues (ICACC 2022) had shown significant differences in the dominant failure mechanisms between the materials, as well as indications of improvement in penetration performance for one microstructure. This work seeks to improve and expand upon that work through more complete statistical investigations, as well as expanding the in-situ diagnostics during the tests to include flash x-ray. Post-mortem analysis of cratering and fracture surface through micro-CT and TEM are used to identify differences in failure mechanisms.

(ICACC-P016-2024) Fundamentals of Reaction-Bonded Ceramic Matrix Composites by Liquid Silicon Infiltration

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Liquid silicon infiltration (LSI) is a low-cost manufacturing method used to fabricate ceramic matrix composites (CMCs) for structural, thermal, and wear-resistant applications. In this process, a carbonaceous ceramic preform is infiltrated by a Si-melt via capillary action. The result is a dense SiC-ceramic body with some residual silicon. However, complete infiltration is not guaranteed as several phenomena must be understood and controlled, including preform development, wicking agent, and crucible and furnace environment. In this work, preforms consisting of various powder size distributions of boron carbide with a fixed amount of pyrolytic carbon are developed to optimize their green density. In this way, a clear investigation can be made to study the effect of preform density on the microstructural evolution of these materials and the final phase fraction content. Findings will be reported and discussed in the context of aforementioned phenomena.

(ICACC-P018-2024) End-to-end multidisciplinary optimal design for improved personalized bioactive glass/ceramic bone substitute implants (ReBone): A new MSC PhD Network

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8. Paris Lodron University of Salzburg, MorphoPhysics Group, Department of the Chemistry and Physics of Materials, Austria
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The musculoskeletal system is particularly susceptible to aging and traumatic occurrences and, thus, new solutions are required for patients who require bone-substitute implants to treat critical-size bone defects timely. The four-year ReBone Doctoral Network (2024-2027), funded by the Europe Horizon Marie Skodowska programme, aims to train a new generation of researchers working in this multidisciplinary field; specifically, ten doctoral candidates will jointly develop an innovative and integrated methodology to design and develop novel personalized ceramic-based bone substitute implants. In order to achieve this ambitious purpose, a European network of partners and associated partners has been established encompassing diverse disciplines including materials engineering, biomechanics, clinics, mechano-biology, additive manufacturing technologies and mixed reality models for surgical planning simulations. Clinical data will be used to create personalized multi-scale models of the implant at the organ level; concurrently, the design of device architecture, materials and parameters for the manufacturing technology will be optimized to achieve improved implant outcome in terms of optimal biomechanical performance related to the defect shape and anatomical location.

(ICACC-P019-2024) Bioactive glass-glycerol pastes for potential use in bone tissue repair

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An ideal biomaterial for bone repair should match the anatomy of the bone defect to be treated; therefore, injectable substances carry an important added value. In this study, injectable pastes based on a clinically-tested bioactive glass (BG1d composition: $46.1SiO_2$-$28.7CaO$-$8.8MgO$-$6.2P_2O_5$-$5.7CaF_2$-$4.5Na_2O$ wt%) and glycerol, used as organic carrier, were produced and characterized for possible use in regenerative medicine. Glass frit was produced by pouring the melts in cold water, followed by crushing and sieving to a particle size within 10-15 μm. BG1d-glycerol pastes were prepared by mixing glass powder and glycerol (88 wt% aqueous solution) with glass-to-carrier ratio of 73/27 by weight. The pastes exhibited an improved bioactivity due to chemical interaction of glycerol with the surface of the glass. Furthermore, being easily injectable through a syringe, the prepared glass paste shows promise in fulfilling two key requirements, i.e. (i) the ease of use by the surgeon and (ii) the ability to conform to the geometry of osseous defects. Preliminary in vivo animal tests demonstrated the bone regeneration ability of the paste.

(ICACC-P020-2024) Formation of Fluorapatite-Hydrogel Composites in a Diffusion-Controlled System

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Teeth and bones are composite materials consisting of organic and inorganic components, possessing unique structures and superior properties. Particularly, mesocrystals are frequently observed in biominerals formed in mild environments as a result of interactions between organic polymers and inorganic substances within living organisms. Among the various biominerals, hydroxyapatite, including fluorapatite, which constitutes human bones and teeth, has been intensively studied for applications for bone and dental therapy. In previous researches, the primary method employed to mimic bones and teeth involved introducing soluble polymers and insoluble matrices into the crystallization process. However, slow and controlled ion transport similar to that in living organisms has not been widely accomplished. In this study, we synthesized fluorapatite by diffusing calcium ions into the hydrogel containing phosphate and fluoride ions, resulting in a composite material composed of hydrogel and fluorapatite. Depending on the flux of ions, various forms of fluorapatite were generated in the hydrogel. Intriguingly, a converged rod-like fluorapatite mesocrystal, resembling the structure of enamel, was exclusively formed under specific conditions. This research contributes to our understanding of crystal synthesis within bio-ceramic composites, with potential applications in the field of dentistry.

(ICACC-P021-2024) Improved energy-storage performance in AgNbO3-based relaxor antiferroelectric ceramics by co-doping with Gd3+ and Ta5+

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With the rapid development of power electronics, dielectric materials with high energy-storage density, low loss, and good temperature stability are eagerly desired for the potential applications in high power pulsed capacitors. Lead-free antiferroelectric ceramics with high energy storage performance show great potential in pulsed power capacitors. However, poor breakdown strength and antiferroelectric stability are the two main drawbacks that limit the energy storage performance of antiferroelectric ceramics. Herein, high-quality $(Ag_{1-3x}Gd_x)(Nb_{0.8}Ta_{0.2})O_3$ ceramics (x = 0-0.04) were prepared by the solid-state reaction method. The breakdown strength was greatly improved as a result of the high density and fine grains, while the antiferroelectric stability was enhanced owning to the M_2 phase. Benefiting from the synergistic improvement in breakdown strength and antiferroelectric stability, $(Ag_{0.97}Gd_{0.01})(Nb_{0.8}Ta_{0.2})O_3$ ceramic reveals a benign energy storage performance of W_{rec}=9.47 J/cm^3 and η=65.93% with good temperature stability, frequency stability and cycle reliability. It is also found that the high applied electric field can promote the M_2-M_3 phase transition, which may provide ideas to improve the thermal stability of the energy storage performance in $AgNbO_3$-based ceramics.

(ICACC-P022-2024) Engineering Relaxor Ferroelectric Ceramics by Configurational Entropy for Electric Energy Storage

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Dielectric capacitors have gained attentions in high power-density energy storage because of fast charging and discharging processes. Perovskite-type relaxor ferroelectric ceramics are the potential materials for static electric energy storage due to excellent energy storing performance. In this study, we have demonstrated an efficient design of lead-free $BiFeO_3$-$BaTiO_3$-based relaxor ferroelectric ceramics through tailoring configuration entropies (S_{config}) to reduce remanent polarization (P_r) and increase electric (E) breakdown strength (E_b). The S_{config} is linked to the disorder and random E field. E_b is an important factor that impacts on the energy storage because the maximum polarization (P_{max}) is proportional to E field. The $(Bi_{0.7}Ba_{0.3})_{1-x}Na_x(Fe_{0.7}Ti_{0.3})_{1-x}Ta_xO_3$ ceramics were synthesized via the solid-state-reaction at x = 0 - 0.3 along with S_{config} from 1.21R to 2.07R. The increased S_{config} can disrupt the long-range order to form the nanoclusters and core-shell structures, accompanied by the reduced P_r and the improved E_b. E_b was significantly increased up to 550 kV/cm at x = 0.20 accompanied by a W_{rec} ~ 8.7 J/cm^3 and a η ~ 66%. The grain boundary density and nanoscale structures play critical roles as electric barriers to increase breakdown strength. These results deliver an approach to exploit the high-entropy $BiFeO_3$-based ceramics for dielectric energy storage.

(ICACC-P025-2024) Sn-Substituted $Li_{6+x}P_{1-x}Sn_xS_5Cl_{0.5}Br_{0.5}$ Sulfide Solid Electrolyte for All Solid State Battery

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All solid state battery has shown attractive attention for their better safety and higher energy density. Among the solid electrolytes, sulfide solid electrolytes have advantage of relatively high ionic conductivity and good ductility. However, due to high reactivity of S species and weak P-S bond, argyrodite sulfide solid electrolytes suffer from unstable interface with Li-metal and poor air stability. One of the methods to improve these problems is the elemental substitution with Si, Cu, or Sn. In this study, Sn-substituted mixed-halide $Li_6PS_5Cl_{0.5}Br_{0.5}$ was synthesized. The crystal structure and ionic conductivity by substitution were evaluated using X-ray diffraction (XRD) and electrochemical impedance spectroscopy (EIS) analyses respectively. The interfacial stability with Li-metal anode and electrochemical performances were evaluated by symmetric cell configurations and galvanostatic charge/discharge test. The improved air stability was also investigated by measuring the H_2S gas generation.

(ICACC-P026-2024) NMR versus conductivity spectroscopy: A complementary approach to characterize ion dynamics in solid electrolytes

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Conductivity (or impedance) measurements are widely used to characterize ion transport in solids. While suffering from post preparation methods to guarantee optimal test specimens in the form of pressed pellets with applied (metallic) electrodes, nuclear spin relaxation (NSR), which is based on magnetic resonance, serves as a contactless and non-destructive method suitable to analyze crystalline as well as amorphous (powder) samples. Here, we compare results from NSR such as activation energies and jump rates with those from alternating current (AC) conductivity spectroscopy to identify the similarities and differences of the two approches. In many cases, rather consistent pictures of the various dynamic situations are found if we take into account that the physical quantities depend on the time window to which the methods applied are sensitive. Besides other examples, we will discuss recent results from conductivity measurements and ^{23}Na, ^{7}Li NSR experiments on Nb-bearing $Na_3Zr_2(SiO_4)_2(PO_4)$ and on hectorite-type $Li_{0.5}[Mg_{2.5}Li_{0.5}]Si_4O_{10}F_2$ (1,2). (1) F. Stainer, B. Gadermaier, A. Kügerl, L. Ladenstein, K. Hogrefe, and H.M.R. Wilkening, Solid State Ionics 395, 2023, 116209. (2) C. Hiebl, P. Loch, M. Brinek, M. Gombotz, B. Gadermaier, P. Heitjans, J. Breu, and H.M.R Wilkening, Chem. Mater. 32, 2020, 7445.

(ICACC-P027-2024) Minimum Thermal Conductivity in Ceramic Solid Electrolytes

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Thermal conductivity data are important engineering parameters for every material or system which is exposed to thermal loading or internal heat generation. The knowledge of this thermophysical property is of particular importance for the thermal management of batteries on the macroscopic scale. However, on the microscopic scale the study of the thermal conductivity of ion conductors can give further insight into transport mechanisms due to the fact that the phonon assisted ion migration is of importance for ionic and thermal transport in solid electrolytes. In this work we have studied the thermal conductivity of NASICON structured Li and Na conducting solid electrolytes as well as Li conducting garnet systems as a function of the temperature. Different phonon transport models ranging from phonon gas models to concepts of minimum thermal conductivity were applied in order to describe the experimental data with respect to phonon-phonon as well as phonon-vacancy interaction processes, respectively. It could be shown that with increasing ionic conductivity associated with high carrier and vacancy concentration the thermal conductivity approaches the minimum thermal conductivity within the given ion conducting solid electrolyte system.

(ICACC-P028-2024) Preceramic polymer derived dual core shell porous Si/C composite as an anode material of Li-Ion battery

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Silicon is one of the most promising anode materials for Li-ion batteries due to its high gravimetric and volumetric capacities. However, Si undergoes volume change (~400%) during the Li alloying process, leading to severe mechanical strain on the anode particles, resulting in capacity decay. To mitigate such problems, we demonstrate the synthesis of dual core-shell porous Si/C composite by co-assembling two different preceramic polymer precursors and Si nanoparticles. One type of preceramic polymer creates an open space around the Si nanoparticles to absorb the mechanical strain, while the other encapsulates the structure with an electrically conductive carbon shell. Complimentary XRD, Raman spectroscopy, BET, XPS, FE-SEM, and TEM are used to understand the composite's phase, structure, and microstructure. TEM microstructure reveals the encapsulation of Si nanoparticles in the mesoporous carbon matrix originating from the preceramic precursors. The porous Si/C composite delivered a stable discharge capacity of 1310 mAh g^{-1} over 400 cycles at 100 mAg^{-1} with a minimal capacity decay. Lithiation kinetics were studied using cyclic voltammetry. High current rate capability showed good consistency. This work elaborates on the processing of microstructural evaluation and underpins the relationship between structure and electrochemical performance.

(ICACC-P029-2024) Large-scale phase-field simulation for prediction of realistic sintered microstructures

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The phase-field method is the most powerful numerical model for investigating material microstructure evolutions during sintering. However, the phase-field simulations of sintering have been limited to a small system due to those high computational cost. In small-scale simulations with a few hundred powder particles, the predicted microstructure deviates significantly from the actual bulk behavior, because the surface ratio in the sintered body is dominant. Therefore, it is essential to enable large-scale phase-field simulations for accurately predicting the microstructure evolutions during sintering. In this study, to accelerate large-scale phase-field simulations of sintering, we developed a high-performance parallel computing scheme with multiple graphics processing units. This scheme enabled the phase-field sintering simulation with approximately 40,000 powder particles on the 1,280 × 1,280 × 1,280 computational grids. In such a large computational domain, bulk densification behavior was observed in the center domain removing the surface area. This presentation is based on results obtained from a project, JPNP22005, commissioned by the New Energy and Industrial Technology Development Organization (NEDO).

(ICACC-P030-2024) Finite element analysis of thermal cycling test for metallized ceramic substrate

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Metallized ceramic substrates are used in a variety of products, such as electronic devices, and are required to have higher reliability. Experiments to measure the mechanism of failure under cyclic thermal loading conditions are important for this purpose, and can be used in complement with numerical analysis to enable detailed investigations. In this study, we proposed a finite element analysis (FEA) method for thermal cycling tests of metallized ceramics substrate composed by copper and ceramics. Here, initial residual stresses were taken into account by modeling the bonding process and etching treatment of the metallized ceramics to faithfully reproduce the experiments. The validity of the FEA model was confirmed by comparison with experimental results. Furthermore, the present FEA revealed that work hardening of copper during the thermal cycle loading process affects the stress concentration in the joint part. This presentation is based on results obtained from a project, JPNP22005, commissioned by the New Energy and Industrial Technology Development Organization (NEDO).

(ICACC-P031-2024) Enhanced Oxygen Evolution Reaction of Co_3O_4 anchored on Carbon Matrix for Alkaline Water Splitting

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Alkaline water electrolysis plays an important role in sustainable hydrogen production, yet faces challenges due to the sluggish kinetics of the 4-electron pathway in the oxygen evolution reaction (OER), requiring overpotential. Carbon nanostructures, notably carbon nanotubes (CNTs), present promising alternatives due to their abundant active sites and efficient charge transfer properties. This study explores the impact of surface-modified carbon (SMC) via pulsed laser ablation in liquid on the electrocatalytic performance of the OER. Specifically, our focus lies on Co_3O_4@SMC heterostructures, where a thin layer of Co_3O_4 is deposited onto SMCs using a combination of PLAL and atomic layer deposition. Comparative analysis with aggregated Co_3O_4 and Co_3O_4@SMC demonstrates the superior OER performance of Co_3O_4@SMC. The optimized Co_3O_4@SMC exhibits lower overpotential, a reduced Tafel slope, and a significantly higher turnover frequency (TOF) in alkaline water splitting. Experimental results, coupled with density functional theory calculations, attribute these improvements to the high electrocatalytic activity of Co_3O_4 active sites achieved through their homogeneous distribution on SMCs. This study enhances our understanding of SMC-based heterostructures and their potential to enhance electrocatalytic performance in alkaline water electrolysis.

(ICACC-P032-2024) The Effect of Al_2O_3 Coating on Electrochemical Performance of Aqueous Zinc Ion Battery

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As the demand for energy storage systems (ESS) increases, the pursuit of safe battery technologies takes on ever-greater significance. Rechargeable aqueous zinc-ion batteries (AZIBs) have emerged as a compelling alternative to lithium-ion batteries, owing to their advantages, such as aqueous electrolytes, high energy density per unit volume, and cost-effectiveness. However, various factors, encompassing hydrogen evolution reactions, corrosion, dendrite growth, structural deterioration, and the formation of undesirable compounds, contribute to the degradation of AZIBs' electrochemical performance. In this study, we investigated the effect of Al2O3 coating on the electrochemical performance of Aqueous Al_2O_3@Zn//MnO_2 Battery. Experimental results exhibit that the cycling performance of a full cell comprising an Al_2O_3-coated Zn anode and MnO_2 cathode closely matches that observed in the case of the bare Zn//MnO2 battery. This observation emphasizes that the inferior cycling characteristics of AZIBs stem from phase transformations and structural breakdown. Consequently, this study underscores the vital importance of exploring optimized materials for both anodes and cathodes in AZIBs to enhance cycle performance.

(ICACC-P033-2024) New route to synthesis nanomaterial of lead sulfide (galena) films from bis(alkylxanthato) lead(II) precursors

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Lead sulfide films were produced from a series of bis(alkylxanthato) lead(II) complexes by a spray-coat-pyrolysis method. We confirmed that the resulting nanomaterial was influenced by the preparation technique and type of ligand. The crystallite domain size of PbS from bis(heterocyclic-dithiocarbamato) lead(II) greater than bis(alkylxanthato) lead(II) precursors. The sizes were decreased in bis(alkylxanthato) lead(II) precursors when the alkyl chain increased. All films were characterized by powder X-ray diffraction (p-XRD), Raman spectroscopy, secondary electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDX).

(ICACC-P034-2024) Investigation of the damage recovery in ion-irradiated $Nd_2Zr_2O_7$ pyrochlore using in-situ synchrotron X-ray diffraction

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Recently, significant efforts have been made to understand the radiation effects in nuclear materials in order to improve their performance under extreme conditions of the nuclear reactor system, including waste management. In the extreme temperature of the reactor, these materials undergo a continuous cycle of amorphization and thermally induced recrystallization. The ability to retain the crystalline phases in irradiated materials demonstrates how the complex microstructure of these ceramics influences the kinetics of temperature-induced structural transition. This study reports the thermal annealing-induced recovery of crystalline phases in ion-irradiated $Nd_2Zr_2O_7$ pyrochlores. XRD results show that $Nd_2Zr_2O_7$ is fully amorphized at a fluence of 5×10^{13} ions/cm^2 after irradiation with 100 MeV I^{7+} ions. In-situ thermal annealing investigation was performed in the temperature range of 300 to 1000 C on pristine and irradiated $Nd_2Zr_2O_7$ pyrochlore using synchrotron x-ray diffraction. The behavior of isochronal annealing is described in terms of improved defects recovery, which demonstrates that pyrochlore superstructure does not appear even at a high temperature of 1000 C. The findings of this study reveal that local electronic structure influences the structural stability, thus providing a critical factor in regulating the functional properties of these materials.

(ICACC-P035-2024) Thermal Characterisation of Ceramic Coated Nuclear Fuel Particles

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TRistructural ISOtropic nuclear fuel (TRISO) is a type of ceramic coated nuclear fuel that is under consideration for the next generation of nuclear reactors. TRISO particles consist of a central fuel kernel, a porous carbon ‘buffer’ coating and a SiC coating that is sandwiched between two pyrolytic carbon (PyC) coatings. To accurately model heat transfer across the coatings, two key material properties must be known, the thermal conductivity and the porosity distribution of the coating. The porosity distribution can often be expressed as a function of density; however, the thermal conductivity of the coatings must be directly measured. Previous methods, such as laser flash, could only give an effective thermal conductivity of the entire particle, since they could not resolve individual coatings. A dual laser technique called time-domain thermoreflectance has since been implemented, and due to its small spot size, can individually resolve TRISO coatings. For this work, the thermal conductivity of TRISO coatings will be measured with time-domain thermoreflectance. By pairing the thermal conductivity results with local thermal characterisation of the coatings, using COMSOL modelling and previous tomography results, a more representative heat transfer model can be produced of the coatings. This will highlight any potential hotspots within coatings and the temperature gradient across the radius of a particle.

(ICACC-P036-2024) Pseudo Plasticity Model for Silicon Carbide Cladding

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Silicon carbide (SiC) fiber-reinforced SiC matrix composite cladding undergoes microcracking when subjected to tensile stress beyond a certain limit. The generated microcracks change the stress-strain response of the composite from linear elastic to pseudo-plastic. The pseudo-plastic response of the SiC composite was modeled using the Hill’s yield criterion implemented in the MOOSE framework. The presentation will highlight the details of the model and sensitivity of the model parameters to the predicted anisotropic elastic-plastic behavior of the SiC composite cladding.

(ICACC-P037-2024) Effect on the Compressive Strength of Lightweight Concrete Using a Blended Cementitious Matrix

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Concrete production has a significant environmental impact due to raw materials mining extraction and Ordinary Portland Cement (OPC) production. Alternative materials like blended cement and recycled polystyrene (EPS) as an aggregate can reduce this damaging impact. This poster will presented the effect on the compressive strength of lightweight concrete produced with a blended cementitious matrix composed of OPC and Alkali-Activated Cement (AAC) based on fly Ash (FA) and an additional calcium source (Lime). A compressive strength test was carried out on the 7th and 28th days of curing. X-Ray Diffraction (XRD) and Fourier Transform Infrared Spectroscopy (FTIR) techniques were used to identify the presence of gels forms due to alkaline activation. The lightweight concrete obtained in this research project could be used to design a concrete structure according to Colombian technical regulations for structural design. In that case, reducing the cross-section of the elements that make up the structure would be possible. Thus, decreasing the dead loads directly related to its own weight. The results also indicated that replacing up to 70% of the OPC with alternative cementitious materials is possible. This could finally be translated into savings in the total cost of the construction work and, with the use of FA and lime, reducing CO_2 emissions.

(ICACC-P038-2024) Hydration Properties of Cement Composites using Ground Granulated Blast Furnace Slag and Electrolysis Alkaline Aqueous

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Cement is an essential construction material, but it is a material that emits large amounts of CO2. Therefore, much research is being conducted around the world on geopolymer cement materials using ground granulated blast furnace slag and alkali activators to replace cement. However, alkali activator such as NaOH, KOH are still difficult to put into practical use because they are dangerous and expensive to use as construction materials. In this experimental study, we performed some experiments to analysis properties of cement composite containing ground granulated blast furnace slag while replacing the use of general alkali activators. To do so, an alkaline aqueous solution obtained by electrolysis of aqueous potassium carbonate(K2CO3) was used as mixing water. Also, the mixing water for making the cement composite was general water and electrolysis alkaline aqueous. Tests such as compressive strength, SEM and MIP tests were conducted to verify the hydration reaction characteristics of cement composite specimens substituted with ground granulated blast furnace slag. In this experimental study, we confirmed that cement composite formed using the electrolysis alkaline aqueous with blast furnace slag exhibited a faster hydration reaction than an equivalent cement matrix mixed using general water.

(ICACC-P039-2024) The substitutional effect of hemp ash on the processing and physical properties of geopolymers

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Hemp ash is the residue of the hemp plant after incineration. The produced ash contains silicates and a low amount of Ca-related phases with significant amorphous content. The substitution of hemp ash for metakaolin as well as a filler material to exhibit good physical properties, thermal resistance as well as low density to be evaluated for sustainable constructional materials were investigated. Hemp ash was processed, dried and sieved to different particle sizes to understand the water adsorption properties including during geopolymerization and it was examined with different analytical techniques such as SEM, XRD, and Archimedes' density. BET analysis of hemp ash and particle size analyses were also included for interpreting the material properties while being processed in geopolymer paste. The geopolymer mixtures were substituted for metakaolin, beginning from 20 wt% to 100 wt% hemp ash to be used in geopolymerization, and their curing period was recorded and analyzed by the Vicat needle test. The resultant cured geopolymers were studied with mechanical tests (3 point flexural and compression), thermal analyses by TGA-DSC to evaluate the formation of different structures, and by XRD and SEM. The hemp ash can be a candidate to substitute for metakaolin to be more cost effective and sustainable to reduce the ash deposition as well as be mechanically suitable for future applications.

(ICACC-P040-2024) Construction and Demolition Waste for Valuable Eco-sustainable Geopolymer Materials

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Geopolymerization and alkali-activation processes have the potential to foster the sustainability and green development of the building industry by reusing and valorizing construction and demolition waste (CDW). CDW can properly act as a solid precursors and the presence of additional phases such as alkaline-earth oxides, pozzolanic and hydraulic phases allow the formation of hydrated and mixed phases of the C(N)ASH type by simple room-temperature reactions. The research focused on the development of geopolymer/alkali-activated formulations using unsorted CDW from residential building as solid precursor is here reported. Secondary reactive precursors, such as metakaolin, were also tested in partial substitution of CDW (10% to 50%). Further, the addition of hydrogen peroxide to the formulated mixtures was also evaluated, in order to reduce the density of the material and induce a second-order functional porosity, This study was carried out within the MICS (Made in Italy – Circular and Sustainable) Extended Partnership and received funding from the European Union Next-Generation EU (PIANO NAZIONALE DI RIPRESA E RESILIENZA (PNRR) – MISSIONE 4 COMPONENTE 2, INVESTIMENTO 1.3 – D.D. 1551.11-10-2022, PE00000004). This manuscript reflects only the authors' views and opinions, neither the European Union nor the European Commission can be considered responsible for them.

(ICACC-P041-2024) Production of Asphalt block (Nonfiring Fencing Slab} using Polyethylene Terephthalate (PET) as an Aggregate for Structural Porous Materials

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The increasing concern over plastic waste has prompted innovative approaches to recycling and repurposing materials for sustainable construction practices. This study explores the feasibility of using Polyethylene Terephthalate (PET) waste, that is found in the community dumpsite of Aloran, Misamis Occidental, derived from discarded beverage bottles, as an alternative coarse aggregate in the fabrication of asphalt blocks-fencing slabs. The research investigates the mechanical properties of PET-enhanced asphalt blocks such as compressive and flexural strength. The fabrication process involves shredding PET waste and mixing it with bitumen and sand aggregate. The resulting mixture is molded into cylindrical blocks, cured, and tested for compressive and flexural strength. The results revealed 26.0783 MPa (compressive strength) and 1.8025 MPa (flexural strength). This finding demonstrated that the blocks produced can be used as an alternative fencing slab (standard concrete slab: compressive strength– 17-24 MPa).

(ICACC-P042-2024) Investigation of mechanical and dielectric propertieso f kaolin and halloysite-based textured ceramics

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This work will focus the characterization of structural and mechanical properties of kaolin-halloysite samples before and after sintering at specific temperatures (sintering at 1300 and 1400°C). Two commercial kaolins noted as KCS and KRG were used together with a halloysite (H) provided by Imerys Company. Moreover, addition of zirconia, alumina or zinc oxide were performed up to 50 mass% the as-obtained products were sintered up to 1400°C and the relative properties changes were investigated and correlated to the sintering behavior. Zirconia and alumina, tended to improve the occurrence of zircon and mullite respectively. Nevertheless, the densification state need to be improved by sintering at temperature higher than 1400°C, due to the refractoriness of zirconia and alumina compared to kaolins. Besides, the improvement of dielectric properties of phyllosilicates ceramics sintered at different temperatures has been investigated. Dielectric constant, dielectric loss tangent and loss factor (were very sensitive to the glassy phase content and the occurrence of multiple crystalline phases upon sintering. The best conditions for dielectric properties were obtained with kaolin-alumina samples, while the best mechanical properties were reached while mixing KRG with H.

(ICACC-P043-2024) Effect of formation rate on the chemical precipitation synthesis of calcium phosphate and magnesium phosphate apatites from natural sources

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To add value to a national product using a sustainable method, two types of apatites were synthesized from a bioleachate of a phosphate rock (RF) of low phosphorus content with sulfuric acid obtained from a strain of A. thiooxidans ATCC 13977, which solubilizes sulfur in the 9K medium in a 5-liter reactor. The phosphate rock contained 5.5 %w/w phosphorus, determined by X-Ray Fluorescence (XRF) and atomic absorption (AA) techniques. 100000 mg/L sulfate content was obtained in the acid, determined by UV-Vis method a wavelength of 420 nm. The leaching liquor was characterized, according to the method described in Standard Methods for the Examination of Water and Wastewater 4500-P C, with a wavelength of 470 nm, achieving 74% of solubilized phosphorus. The two materials were synthesized by the chemical precipitation method with the following initial conditions: leaching liquor as a source of phosphorus, magnesium and calcium, pH of 5.32 and temperature of 25C. To favor the formation of orthophosphates, NaOH was added to the leachate until a pH of 10 was obtained, varying the agitation speed at 300 and 400 rpm. The resulting materials were dried at 80C and characterized by the following techniques: X-Ray Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR), and X ray Flourescense (XRF).

(ICACC-P045-2024) Comprehensive analysis of dielectric properties and AC conductivity in Sr_2TiO_4 Ruddlesdon popper oxide

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The Ruddlesdon popper oxide, Sr_2TiO_4, was synthesized using the solid-state route, resulting in a singular Sr_2TiO_4 phase achieved through calcination at 1200°C for 10 hours. This study offers a comprehensive study of the structural, optical, and electrical characteristics of the synthesized sample. The analysis of X-ray diffraction data, employing Rietveld refinement, confirmed the tetragonal structure of Sr_2TiO_4, attributed to the I4/mmm space group. Further scrutiny of the powder's purity was conducted using Raman and infrared spectroscopy techniques. Tauc's plot was employed to determine the optical band gap, resulting in a value of 3.494 eV. The material's microstructure was assessed via SEM, revealing an average grain size of 0.75 μm. Dielectric and electrical measurements were carried out over a frequency range spanning from 20 Hz to 2 MHz, covering temperatures between 300°C and 600°C. The higher dielectric constant observed at lower frequencies suggests interfacial polarization in the sample. The AC conductivity of the sample follows universal Jonscher power law. The activation energy for DC conductivity was determined to be 1.08 eV, indicating the possibility of conduction through the migration of doubly ionized oxygen vacancies. This ceramic material exhibits potential as a promising candidate for next-generation medium-permittivity radio wave dielectric ceramics.

(ICACC-P046-2024) Chemical Vapor Deposition of Phase-Pure Thorium Dioxide Thin Films from Thorium(IV) Molecular Precursors

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Thoria (ThO_2) has been identified as an excellent candidate for the use as a heterogeneous catalyst in the ammonia (NH_3) synthesis from nitrogen (N_2) and hydrogen (H_2), as indicated by theoretical calculations.[1] In pursuit of an efficient method for generating ThO_2 materials, heteroleptic thorium(IV) alkoxo-enaminonate complexes have been synthesized and employed as single-source molecular precursors for the metal organic chemical vapor deposition (MO-CVD) of thoria thin films. The high volatility and thermal decomposability of these precursors facilitate the production of homogenous thin films, which subsequently exhibit a porous morphology after undergoing thermal treatment. [1] G. Wang G, E.R. Batista, P. Yang, Front. Chem. 2023, 10, 1051496.

(ICACC-P047-2024) Thermally and mechanically stable superhydrophobic glass coatings containing hexagonal boron nitride particles

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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, hexagonal Boron nitride (hBN) particles can also be used for coating. In a first step hexagonal Boron nitride (hBN) particles were hydroxylated with further functionalization with 1H,1H,2H,2H-perfluorooctyl-triethoxysilane (PES). Glass coatings containing functionalized hexagonal boron nitride (hBN) particles 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.

(ICACC-P048-2024) HfC/HfO_2 nanocomposites with controlled properties from a commercial single source precursor

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Hafnium carbide (HfC) is a promising material for high-temperature applications due to its high melting point, excellent thermal conductivity, and good mechanical strength. In this study, a commercial HfC precursor was used and characterized at different crosslinking temperatures ranging from 100 to 400 °C and pyrolysis temperature from 1200 to 1600 °C. The present study focuses on their microstructure evolution at various temperatures in argon atmosphere. FTIR spectroscopy provided information on the chemical composition and functional groups present in the precursor molecules at various stages of crosslinking process. ^{1}H and ^{13}C NMR spectrum provided information on the chemical shifts of the nuclei, which was used to identify the types of atoms and the nature of the chemical bonds within the HfC precursor. In addition, XPS was used to gather information on the chemical composition and electronic structure of the precursor and ceramics. The resulting XPS spectrum provided information on the chemical states of the elements, as well as their oxidation states and chemical environments. It is essential to develop techniques for synthesis of ceramic powder with reasonable low production cost. Therefore, studies on low-temperature synthesis and low-cost precursors are among the main emphases of ongoing research effort.

(ICACC-P050-2024) Tailoring the Structure and Composition of SiOC Electrodes for High Li Storage

S. Mujib*[1]; A. Roy[1]; P. Owiredu[1]; G. Singh[1]

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

Silicon-based anodes have the potential to significantly increase the energy density of lithium-ion batteries, but their use is limited by their tendency to expand in volume which leads to poor performance. Silicon oxycarbides (SiOC) offer a solution to this problem, as they experience less volume change with high theoretical capacity. However, the electronic conductivity of most pure SiOC anodes is poor and their volume expansion is still a concern. To address these issues, a high-performance hybrid SiOC anode was derived from the mixture of two different polyorganosiloxanes: 1,3,5-Trivinyl-1,1,3,5,5-PentamethylTriSiloxane and 1,3,5,7-tetravinyl-1,3,5,7-tetramethylcyclotetra-siloxane. The resulting SiOC is composed of free carbon phase, SiO_4, SiO_3C, and SiO_2C_2 units. This macroporous SiOC anode has a specific capacity of 750 mAh g^{-1} at 50 mA g^{-1} demonstrating high Li^+ ion reversibility compared to the SiOC derived from individual precursors. The combination of robust multidimensional conductive architecture and chemical composition lead to superior specific capacity and cycling performance.

(ICACC-P052-2024) Evaluating Na+ ion storage in TMD nanotube embedded SiOC fibers

S. Dey*[1]; P. Owiredu[1]; A. Roy[1]; S. Mujib[2]; G. Singh[2]

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

Layered transition metal dichalcogenides (TMDs) such as tungsten disulfide (WS2) are promising materials for a wide range of applications, including charge storage in batteries and supercapacitors. Nevertheless, TMD-based electrodes suffer from bottlenecks such as capacity fading at high current densities, voltage hysteresis during the conversion reaction, and polysulfide dissolution. To tame such adverse phenomena, we fabricate composites with WS2 nanotubes. Herein, we report on the superior electrochemical performance of ceramic composite fibers comprising WS2 nanotubes (WS2NTs) embedded in a chemically robust molecular polymer-derived ceramic matrix of silicon-oxycarbide (SiOC). Such a heterogeneous fiber structure was obtained via electrospinning of WS2NT/preceramic polymer solution followed by pyrolysis at elevated temperatures. The electrode capacity fading in WS2NTs was curbed by the synergistic effect between WS2NT and SiOC. As a result, the composite electrode exhibits high initial capacity of 454 mAh g-1 and the capacity retention approximately 2-3 times higher than that of the neat WS2NT electrode.

(ICACC-P053-2024) TMD nano sheet decorated SiOC fibers for beyond Li+ ion storage applications

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Transition metal dichalcogenides (TMDs) such as the WS2 have been widely studied as potential electrode materials for lithium-ion batteries (LIB) owing to TMDs' layered morphology and reversible conversion reaction with the alkali metals between 0 to 2 V (v/s Li/Li+) potentials. However, works involving TMD materials as electrodes for sodium- (NIBs) and potassium-ion batteries (KIBs) are relatively few, mainly due to poor electrode performance arising from significant volume changes and pulverization by the larger size alkali-metal ions. Here, we show that Na+ and K+ cyclability in WS2 TMD is improved by introducing WS2 nanosheets in a chemically and mechanically robust matrix comprising precursor-derived ceramic (PDC) silicon oxycarbide (SiOC) material. The WS2/SiOC composite in fibermat morphology was achieved via electrospinning followed by thermolysis of a polymer solution consisting of a polysiloxane (precursor to SiOC) dispersed with exfoliated WS2 nanosheets. The composite electrode was successfully tested in Na-ion and K-ion half-cells as a working electrode, which rendered the first cycle charge capacity of 474.88 mAh g−1 and 218.91 mAh g−1, respectively. The synergistic effect of the composite electrode leads to higher capacity and improved coulombic efficiency compared to the neat WS2 and neat SiOC materials in these cells.

(ICACC-P054-2024) Differentiating carbon allotropes via cost-effective Electrochemical Impedance Spectroscopy

S. Dey*[1]; P. Owiredu[1]; A. Roy[1]; S. Mujib[2]; G. Singh[2]

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Optimizing the next-generation Sodium-ion batteries (NIBs) requires a comprehensive analysis of cycling behavior and kinetic information. While continued research on developing negative electrodes for NIBs occur, most require cumbersome fabrication procedures, increasing the operation time and cost. While Graphite still dominates as a robust negative electrode material in Lithium-ion Batteries (LIBs), tantamount properties still do not exist in modern negative electrodes for NIBs. Although copious demanding techniques exist for electrode fabrication, surface functionalization, and nanostructure formation are ineffable and widely applicable. In this study, we study surface functionalization and nanostructuring techniques, use modified traditional Graphite, and exhaustively investigate the Na+ ion storage and kinetic behavior. Graphene nanoplatelets (GNP) and Graphene Oxide (GO) show high Na+ ion storage (50 mAh g-1 and 157 mAh g-1 after 60 cycles, respectively), while Graphite failed so (27 mAh g-1) at constant cycling conditions. Over and above, we also take advantage of ex-situ electrochemical impedance spectroscopy (EIS) at progressive cycles and correlate capacity degradation with impedance arising from the electrolyte, SEI formation, and charge transfer.

(ICACC-P055-2024) Study of adsorption of ceria according to Ce^{3+}/Ce^{4+} concentration in Chemical Mechanical Planarization (CMP)

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2. SungKyunKwan University, Department of Polymer Science and Engineering, Republic of Korea

As semiconductor devices shrink, improvements in the CMP (Chemical Mechanical Planarization) process are also needed. Since the node of semiconductor is small and micro defects caused by CMP are critical to yield and performance of semiconductor, not only polishing ability but also post CMP cleaning ability is important. Ceria (CeO_2) nanoparticles are a main material of CMP slurry, and it is widely used for STI (Shallow Isolation Trench) and ILD (Interlayer Dielectric) process which are separating nodes making strong chemical bonding with SiO_2. This interaction can be further increased by changing oxidation state of Ceria. In other words, as the concentration of Ce^{3+}/Ce^{4+} increases, the interaction between with SiO_2 becomes better and the removal rate of CMP can be increased. However, adsorption of ceria nanoparticles on SiO_2 surface is difficult to remove and eventually may remain attached. As a result, ceria nanoparticles remaining on SiO_2 surface are un-etched in subsequent process and do not form patterns properly. This paper deals with the comparison of CMP performance and adsorption level according to Ce^{3+}/Ce^{4+} concentration. The characteristics of ceria nanoparticles were analyzed by HRTEM and XPS, and the adsorption of ceria on SiO_2 surface was investigated using FESEM.

Wednesday, January 31, 2024

S2 Advanced Ceramic Coatings for Structural/Environmental & Functional Applications

SYMPOSIUM 2: Environmental Barrier Coatings III

Room: Flagler C
Session Chair: Elizabeth Opila, University of Virginia

8:30 AM

(ICACC-S2-036-2024) Investigation of single-phase high-entropy zirconates and their thermophysical properties as future thermal barrier coatings (Invited)

P. Hutterer[2]; J. J. Pflug[1]; M. Schenker[1]; M. Lepple*[1]

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

High entropy oxides have attracted great interest in recent years because of their unique and tunable properties. Like high entropy alloys, they consist of five or more cations in approximately equal amounts distributed on one or more sublattice sites. In terms of application as TBC materials, HEOs are promising because the high configurational entropy results in improved phase stability at high temperatures. Due to the different cations, phonons are additionally scattered, which leads to a reduction of thermal conductivity. However, since the material class is still relatively young, there is a lack of systematic studies regarding phase formation, stability, and properties. In this work, HEO zirconates with the general formular $A_2Zr_2O_7$ and up to nine different cations in equimolar amount on the A site were investigated. The composition has been varied systematically to evaluate its influence on crystal structure and material properties. The samples were successfully synthesized using reverse co-precipitation, crystal structure and chemical composition were characterized using X-ray diffraction (XRD) and scanning electron microscopy. The high temperature stability, thermophysical properties, such as thermal conductivity and coefficient of thermal expansion and the stability against CMAS corrosion were investigated.

9:00 AM

(ICACC-S2-037-2024) Calorimetric Measurements of the Thermodynamic Properties of Environmental Barrier Coatings

G. Costa*[1]; N. P. Bansal[1]; R. I. Webster[1]; B. Kowalski[1]; J. L. Stokes[2]

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

Thermodynamic properties of rare earth (RE) silicate materials are crucial for thermochemical modeling of phase equilibria to improve the durability of the hot-section components of gas-turbine engines. Some of the most important properties, which are used as input parameters for CALPHAD methods, are enthalpies of formation, fusion and heat capacities. Furthermore, the correlation between these properties, structural parameters, bonding and phase chemistry provides important insights on the energetic stability of compounds related to their constituents (oxides and elements). In this work, high-temperature oxide melt drop solution calorimetry was used to measure the enthalpies of formation of RE silicates ($RE_2Si_2O_7$, RE_2SiO_5 where RE = Yb, Er, Y, Dy, Nd, Lu and Gd) environmental barrier coatings (EBCs). In addition, heat capacities and enthalpies of fusion of the RE silicate coating materials were measured by differential scanning calorimetry and differential thermal analysis, respectively. Trends in the enthalpy of formation and fusion as well as heat capacities of the RE silicate coating materials are discussed in terms of their structural parameters.

9:20 AM

(ICACC-S2-038-2024) Mullite processing in high temperature EBCs and performance impacts

K. Lee[2]; R. I. Webster[2]; A. Setlur*[1]; J. Wan[1]; R. Sarrafi-Nour[1]

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

Environmental barrier coatings (EBCs) including mullite layers were investigated and used in earlier EBCs for Si-based ceramics. Owing to its higher temperature capability and a low coefficient of thermal expansion, mullite continues to be investigated as the inner layer of EBC architectures with high temperature capabilities. Typical mullite ceramics with low amorphous phase content are densified at temperatures greater than the mullite-SiO_2 eutectic temperature, approximately 1587°C. These high densification temperatures are generally not compatible with SiC-SiC ceramic matrix composites (CMCs), making mullite EBC densification on these CMCs more challenging. In this presentation, we first discuss potential sintering routes using liquid phase sintering aids to lower mullite sintering temperatures while preserving high-temperature capabilities for inclusion in EBC architectures. Sintering evaluations using different oxide additives are initially made through simple cold-pressed pellets tested under different sintering conditions with effective densification for rare-earth based sintering aids. We further discuss the effect of different rare earth sintering aids on mullite EBC processing and performance, including oxidation stability and microstructure evolution over extended times. This work has been supported through NASA HyTEC funding (contract# 80GRC021CA005).

9:40 AM

(ICACC-S2-039-2024) Recent Advancements in High Temperature Slurry Environmental Barrier Coatings for SiC/SiC Ceramic Matrix Composites

K. Lee*[1]; R. I. Webster[1]; B. Puleo[1]; M. J. Presby[1]; B. J. Harder[1]; J. A. Setlock[2]; L. C. Hoffman[3]

1. NASA Glenn Research Center, USA
2. University of Toledo, USA
3. HX5, LLC, 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 upper use temperature of current CMCs is ~1316 °C (2400 °F). The next generation CMCs are targeting for 1482 °C (2700 °F) temperature capability. The Si bond coat employed in current state-of-the-art EBCs is not a viable bond coat for next generation CMCs due to its low melting point (1414 °C). The viability of next generation CMCs, therefore, is contingent upon the successful development of a higher temperature bond coat to replace the Si bond coat. Oxide-based bond coats are a logical choice to replace the Si bond coat. Previously NASA demonstrated the feasibility of 1482 °C oxide-based bond coats using slurry process. NASA has been advancing the high temperature EBC technology by scaling up the process and conducting rig tests simulating engine environments on large CMC test pieces and CMC airfoils. This paper will discuss the current status of high temperature slurry EBC technology at NASA.

S13 Development & Applications of Adv Ceramics & Composites for Nuclear Fission/Fusion Energy Sys

SYMPOSIUM 13: Chemical compatibility and corrosion

Room: Ballroom 4
Session Chair: Shuigen Huang, KU Leuven

8:30 AM

(ICACC-S13-026-2024) A Comprehensive Study on SiC/SiC Fiber Composites and Corrosion Kinetics in Light-Water Reactors (Invited)

K. Shirvan*[1]

1. Massachusetts Institute of Technology, USA

Recognizing the limitations of existing hydrothermal corrosion models, which are primarily predicated on temperature and oxygen concentrations, our multidisciplinary team conducted a comprehensive experimental campaign to go beyond existing hydrothermal corrosion models, which mainly consider temperature and oxygen levels. We examined individual elements like CVD layers and fibers and expanded corrosion models to include factors like irradiated microstructure and flow rate. Of particular interest is our investigation into the role of CRUD (particle adhesion, revealing its significant impact on mitigating corrosion rates. These updated models were incorporated into the RADICAL code, improving its ability to simulate coolant loop behaviors in LWRs. Our simulations indicate that while CVD SiC is promising for Pressurized Water Reactors (PWRs), challenges remain for Boiling Water Reactors (BWRs). We propose a mitigation strategy involving a double-layer metal coating—specifically, a FeCrAl overcoat with a Cr bond coating, which proved stable and adherent under extreme conditions. We also investigated the performance of additively manufactured SiC fibers under corrosion and irradiation, offering a multifaceted perspective on SiC/SiC challenges and solutions.

9:00 AM

(ICACC-S13-027-2024) Development of CVI/CVD-SiC/SiC Composite for Accident-Tolerant Fuels of LWR Plants

S. Suyama*[1]; M. Ukai[1]; T. Nishimura[1]; S. Kuboya[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 its inherent low activation, low neutron absorption cross-section, and stability under irradiation due to its high purity and crystalline SiC matrix. After the nuclear power plant accident in Fukushima in March 2011, it started to be developed in many research programs as a component of accident-tolerant fuels (ATFs). In Japan, as of 2023, more than 10 years after the Fukushima nuclear accident, only 10 of the 33 nuclear reactors have been restarted. Therefore, there are growing demands for the development of accident-resistant core materials to ensure improved safety in Japan. Toshiba Energy Systems Corporation has been developing process technologies for thin-walled and long tubes and boxes made of CVI/CVD-SiC/SiC since 2012. This CVI-SiC/SiC is expected to be resistant to water vapor environments above 1200°C during accidents. In addition, resistance to hydrothermal corrosion for the operating duration under normal LWR conditions is required. This study gives an account of progress in the development of process technology for thin-walled and elongated CVI/CVD-SiC/SiC-based tubes and boxes. In particular, it explores the use of coatings of CVD-SiCs on the surface layer of CVI-SiC/SiC to improve the hydrothermal corrosion resistance under LWR conditions.

9:20 AM

(ICACC-S13-028-2024) High-temperature Oxidation, Corrosion and Wear Resistance of Cr/Cr_2AlC Coatings on Zircaloy-4 Alloys for Accident Tolerant Fuel

Y. Lei*[1]; J. Zhang[1]; J. M. Schneider[3]; J. Wang[2]

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2. Shenyang National Laboratory for Materials Science, Institute of Metal Research, Chinese Academy of Sciences, High-performance Ceramics Division, China
3. RWTH Aachen University, Materials Chemistry, Germany

The development of accident tolerant fuel (ATF) has been launched worldwide to enhance accident tolerance in light water reactors. Protective coatings are fabricated on zirconium-based alloys, as one of ATF strategies, to improve the margin of safety under extreme conditions and maintain good performance under normal operating conditions for light water reactors (LWRs). Cr_2AlC coating, attractive coating candidate for ATFs, is consumed by reaction with Zircaloy substrate when the temperature is up to 1000 °C, leading to severe performance degradation. In this wok, suitable buffer layer was selected among Cr, Mo and Nb candidates, aiming to restrict the inward diffusion of Al during high temperature oxidation. Thereafter, Cr/Cr_2AlC bilayer coatings were synthesized by magnetron sputtering, and performance under normal operations (corrosion resistance and fretting wear resistance) and accident scenarios (high temperature oxidation resistance) are investigated. For the application of protective coatings on Zr-based cladding, Cr/Cr_2AlC coatings are expected to improve the safety of nuclear fuel for the current LWRs in both normal operating condition and LOCA scenario.

9:40 AM

(ICACC-S13-029-2024) Be-bearing molten fluoride salt corrosion of SiC/SiC composites

B. W. Lamm*[1]; T. Koyanagi[2]; J. Keiser[1]; J. Lee[1]; Y. Katoh[2]

1. Oak Ridge National Laboratory, Materials Science & Technology Division, USA
2. Oak Ridge National Laboratory, USA

The development of SiC-based components for molten salt reactors requires an understanding of the chemical compatibility to the proposed molten salts. For the material screening purposes, chemical vapor infiltrated SiC_f/SiC composites were exposed to a static molten salt, 2LiF-BeF_2, for 500 and 1000 h at 750 °C. Composites comparing two kinds of SiC fiber (Tyranno SA3 and Hi-Nicalon type S) both experienced slight weight loss and nominal surface etching. Preferential corrosion of SA3 fibers was observed. For both matrix and fibers, localized pitting and nonuniform Si removal from surface regions, associated with trace metal impurities and/or oxygen were observed. The minimum weight loss warrants further investigations of the materials under relevant reactor coolant environments. This research was sponsored by US DOE, Office of Nuclear Energy and Fusion Energy Sciences under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

S15 8th International Symposium on Additive Manufacturing and 3-D Printing Technologies

SYMPOSIUM 15: Fused Filament Fabrication and Direct Ink Writing I

Room: Coquina H
Session Chair: Martin Schwentenwein, Lithoz GmbH

8:30 AM

(ICACC-S15-030-2024) Water soluble feedstocks for additive manufacturing of complex shapes and flexible interconnected ceramic parts (Invited)

D. Penner*[1]; R. Wick-Joliat[1]

1. ZHAW Zurich University of Applied Sciences, Switzerland

Material extrusion MEX of complex thermoplastic structures often depends on the reliable printing of a water-soluble support structure. The material of choice is typically polyvinyl alcohol (PVA), which is difficult to use in ceramic MEX printing due to a limited printing compatibility with most ceramic feedstocks (poor layer adhesion). A new thermoplastic feedstock concept was developed as temporary water-soluble support material on the basis of NaCl mixed with a commercial injection molding binder system. Additive manufacturing enables the introduction of new molding concepts not only through the direct printing of ceramic green bodies with or without support material, but also generally through the production of water-soluble mold materials. The particular advantage of additive manufacturing's virtually unlimited geometry complexity then additionally allows the production of undercut models, threads, channels, and other otherwise complicated mold elements. The use of soluble molds instead of direct 3D printing of ceramic green bodies thus offers the possibility to partially circumvent existing limitations of currently available technologies and materials.

9:00 AM

(ICACC-S15-031-2024) Developing and comparing binder formulations for fused filament fabrication of 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

Additive manufacturing (AM), also known as 3D printing (3DP), has been used as a production and shaping method for polymers, ceramics and metals. Interest in this method increases due to reduced costs and providing design freedom without expensive tools. The development in AM of ceramic materials is relatively slow compared to polymers and metals. To improve 3DP of ceramics, various AM methods are being investigated. Fused Filament Fabrication (FFF) is one of them; a ceramic powder is mixed with a polymer binder system and used to produce a filament that becomes the feedstock that can be printed. $ZrSiO_4$ is the ceramic powder used in this work to produce a ceramic matrix composite. Filaments with three different feedstocks have been produced and characterized to observe their potential for FFF. Characterization has involved determining the rheological behaviour, surface properties, bending strength and dimensional tolerance of the filaments and the subsequent composites. The results of this work provide a good understanding of developing a binder formulation for FFF.

9:20 AM

(ICACC-S15-032-2024) Fused filament fabrication of lead-free piezoceramics: From filament production to sintered components

S. Bhandari*[1]; P. Veteška[2]; G. Vajpayee[3]; L. Bača[2]; Z. Hajdúchová[2]; Z. Špitálsky[4]; M. Hinterstein[3]; G. Franchin[1]; M. Janek[2]

1. Department of Industrial Engineering, University of Padova, Via Marzolo 9, Italy
2. Department of Inorganic Materials, Faculty of Chemical and Food Technology, Slovak University of Technology in Bratislava, Radlinského 9, Slovakia
3. Fraunhofer IWM, Germany
4. Polymer Institute, Slovak Academy of Sciences, Dúbravská cesta 9, Slovakia

Fused filament fabrication (FFF) of ceramics can potentially enable a broader diffusion of AM ceramic components; in fact, this prototyping method utilizes ceramic loaded filaments as feedstock for relatively cheap equipment meant for polymeric materials, with little adaptation of the process parameters. The characteristics of the filament play a key role in the fabrication of high-quality components. In this work, composite filaments with various weight fractions (60 – 80 wt.% of BaTiO 3) were fabricated using a twin-screw extruder, which were further characterized by SEM, compressive mechanical test, rheometer and TGA to ensure a smooth and reliable printing process. After optimizing the printing parameters, the printed (porous and dense pellets) samples were carefully debinded and sintered to obtain a dense (~90%) and defect-free ceramic body. The sintered samples were characterized for the phase development, the microstructure and the pore size distribution. The measured dielectric and ferroelectric properties of the fabricated samples were in good agreement with previously reported literature. This straightforward and low-cost material extrusion process can be further exploited to print functional structures, in contrast to the costly equipment or the precise control of the rheology for the feedstock required for other AM techniques.

9:40 AM

(ICACC-S15-033-2024) Silicon carbide additive manufacturing for space mirrors

M. Gauthe*[1]; X. Tonnellier[1]; L. Chaffron[2]; J. Rodolfo[1]; Y. Sortais[3]; C. Lorrette[2]

1. Safran REOSC, France
2. Université Paris Saclay, CEA, France
3. Université Paris-Saclay, Institut d'Optique Graduate School, France

Silicon carbide (SiC) is a commonly used technical ceramic in various fields such as space and energy applications. This material is chosen due to its low density, high stiffness, good mechanical and thermal properties and excellent oxidation stability. The fabrication of traditional sintered SiC (SSiC) parts includes the green-machining, sintering, grinding and polishing. Additive manufacturing (AM) technologies have provided new fabrication opportunities for near-net-shape ceramic components. Some of these technologies are difficult to implement because of SiC properties (UV light absorption, sintering difficulties) and are either set up to fabricate reaction-bonded SiC or using infiltration methods to consolidate parts. Only extrusion methods such as Fused Deposition Modeling (FDM) offer for now the possibility to manufacture pressureless sintered SiC. This work presents measurements of microstructural and mechanical properties (density, hardness, Young modulus, bending strength) of SSiC parts made using FDM. Promising results are obtained with grain size around a few micrometers, density above 94% and very high hardness. However, macro-size porosities inherent to the 3D printing process were observed. A design of experiments using Taguchi's method was set up to understand the influence of the 3D printing parameters on porosities and geometries. Samples were examined using nondestructive X-ray tomography.

S16 Geopolymers Inorganic Polymers and Sustainable Construction Materials

SYMPOSIUM 16: Use of waste materials

Room: Coquina C
Session Chair: Henry Colorado L., Universidad de Antioquia

8:30 AM

(ICACC-S16-016-2024) Geopolymers and alkali activated materials: Mix design and characterization in view of their safe use in different applications (Invited)

M. C. Bignozzi*[1]; G. Masi[1]; A. Zappi[2]; L. Tositti[2]

1. University of Bologna, Department of Civil, Chemical, Environmental and Materials Engineering, Italy
2. University of Bologna, Department of Chemistry "G. Ciamician", Italy

Geopolymers and alkali activated materials are a new class of materials largely investigated in the last years, despite their characteristics and performances largely depend on several parameters such as selected precursors and activating agents, mix design, curing conditions, etc.. Currently, metakaolin and carbon fly ash/ground granulated blast furnace slag are the most popular precursors for geopolymers and alkali activated materials, respectively. However, several types of industrial waste have been largely investigated to highlight their potentialities as secondary raw materials. Although chemical, physical, mechanical characterizations are fundamental to address these materials to their final application, it is very important to be sure that safety characteristics such as heavy metals content and/or their release and radiological safety comply the regulations in force. In this study, a comparison of different typologies of geopolymers and alkali activated materials is carried out to obtain awareness about their safety when used in different sectors.

S17 Advanced Ceramic Materials and Processing for Photonics and Energy

SYMPOSIUM 17: Advanced and nanostructural materials for photo-voltaics and solar fuels

Room: Coquina G
Session Chairs: Mohamed Siaj, University of Quebec, Montreal; Marina Leite, UC Davis

8:30 AM

(ICACC-S17-024-2024) Rapid laser-induced low temperature crystallization of thermochromic VO_2 sol-gel thin films (Invited)

M. Basso[1]; E. Colusso[1]; E. Napolitani[2]; A. Martucci*[1]

1. University of Padova, Industrial Engineering, Italy
2. University of Padova, Physics and Astronomy, Italy

The thermochromic properties of vanadium dioxide (VO_2) offer great advantages for energy-saving smart windows, memory devices, and transistors. However, the crystallization of solution-based thin films at temperatures lower than 400°C remains a challenge. Photonic annealing has recently been exploited to crystallize metal oxides, with minimal thermal damage to the substrate and reduced manufacturing time. Here, VO_2 thin films, obtained via a green sol-gel process, were crystallized by pulsed excimer laser annealing. The influence of increasing laser fluence and pulse number on the film properties was systematically studied through optical, structural, morphological, and chemical characterizations. From temperature profile simulations, the temperature rise was confirmed to be confined within the film during the laser pulses, with negligible substrate heating. Threshold laser parameters to induce VO_2 crystallization without surface melting were found. With respect to furnace annealing, both the crystallization temperature and the annealing time were substantially reduced, with

VO_2 crystallization being achieved within only 60 s of laser exposure. The laser processing was performed at room temperature in air, without the need of a controlled atmosphere. The thermochromic properties of the lasered thin films were comparable with the reference furnace-treated samples.

9:00 AM

(ICACC-S17-025-2024) Extraordinarily transparent compact metallic metamaterials (Invited)

V. Giannini*[1]

1. TII, Advanced Materials, United Arab Emirates

Metals are highly opaque, yet we show numerically and experimentally that densely packed arrays of metallic nanoparticles can be more transparent to infrared radiation than dielectrics such as germanium, even for arrays that are over 75% metal by volume. Despite strong interactions between the metallic particles, these arrays form effective dielectrics that are virtually dispersion-free, making possible the design of optical components that are achromatic over ultra-broadband ranges of wavelengths from a few microns up to millimeters or more. Furthermore, the local refractive indices may be tuned by altering the size, shape, and spacing of the nanoparticles, allowing the design of gradient-index lenses that guide and focus light on the microscale. The electric field is also strongly concentrated in the gaps between the metallic nanoparticles, and the simultaneous focusing and squeezing of the electric field produces strong 'doubly-enhanced' hotspots, which could boost measurements made using infrared spectroscopy and other non-linear processes over a broad range of frequencies, with minimal heat production.

9:30 AM

(ICACC-S17-026-2024) Unlocking Water Splitting Potential: 2D Materials and Beyond (Invited)

T. A. Shifa*[1]

1. Ca' Foscari University of Venice, Department of Molecular Sciences and Nanosystems, Italy

Water splitting is considered one of the most promising approaches to power the globe without the risk of environmental pollution. However, it is thermodynamically an uphill process. Researches are taking place to design an excellent catalyst that would effectively facilitate hydrogen evolution reaction (HER) and oxygen evolution reaction (OER). Here, I introduce methodologies aimed at boosting catalytic performance with a special focus on transition metal chalcogenides, transition metal phosphorus trichalcogenides, as well as non-layered materials. I discuss the advantages of these materials for catalysis and the different routes available to tune their electronic states and active sites. Another compelling issue in this research area is about solving the sluggish kinetics of the OER. 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 (M= Mn, Ni, Fe, Cu/In) nanosheets as promising materials in sacrificial agent-free photocatalytic water splitting under simulated Sun light (AM 1.5G) illumination.

10:20 AM

(ICACC-S17-027-2024) Europium-doped ZnO – How are the dopants distributed? (Invited)

G. Westin*[1]

1. Uppsala University, Sweden

There is a strong demand for tailored oxide semiconductor materials driven by applications in e.g. sensors, catalysts, solar-cells and energy storage. Here ZnO is one of the most important and studied oxides. The ZnO is often doped by d- or f-elements for enhanced or added functionality. These dopant ions are often alio-valent and much larger than the Zn^{2+}-ions of the host crystal which indicate that it should not be possible for ZnO to include more than very minor amounts of them. However, there are reports of doping with up to 20% Eu^{3+} in ZnO and doping with up to 5%Eu has been proven by TG/DSC, XRD, IR spectroscopy, SEM, TEM, XPS, and EXAFS. DFT generated Eu_4- and Eu_8-oxide clusters within ZnO crystals showed the lowest energy and excellent fit to experimental data. Here the study is extended to higher ZnO:Eu concentrations of 5 to 33%Eu using a salt-based route to ZnO:Eu nano-sponges built of ca 10 nm sized ZnO:Eu crystals. XRD and IR spectroscopy were used to obtain the structure and contents of the ZnO:Eu sponges. While the 10%Eu sample was similar to the 5%Eu sample, the 20 and 33%Eu samples indicated at least a partial change in doping mode which may be due to formation of a second Eu-richer phase. The 20%Eu sample was therefore studied in detail using STEM/ED/HAADF/EDX mapping and electron PDF to describe the Eu surrounding. A structure with ZnO:Eu crystals and an Eu-richer inter-granular phase was revealed.

10:50 AM

(ICACC-S17-028-2024) Organic magnetoresistance in conjugated polymers (Invited)

E. Orgiu*[1]

1. Institut National de la Recherche Scientifique (INRS), EMT Centre, Canada

Organic conjugated polymers are especially suited for applications in (opto)electronics and thermoelectrics and represent a fantastic playground for fundamental studies of charge, heat and spin transport. While the physical phenomena associated with the application of a magnetic field are well-known for vertical devices using a non-magnetic material sandwiched between two ferromagnetic electrodes, large magnetic-field effects in devices in which the organic semiconducting layer is sandwiched between non-ferromagnetic electrodes came as a great surprise. While the precise origin of these magnetic-field effects in organics is still a matter of debate and involves interactions between charged and uncharged species of different natures, here we present a comprehensive study wherein the organic magnetoresistance (OMAR) of different types of polymers are presented. The polymers are characterized structurally and their paracrystallinity is measured and correlated to the OMAR observed. Differently than most previous reports on OMAR, the latter is measured in three-terminal devices and not in vertical diodes. Low and high magnetic fields characteristics are measured that strongly depend on the chemical structure of the polymer. We characterized n-type, p-type and ambipolar polymers and draw general conclusions on the fundamental mechanisms underlying the observed OMAR effects in conjugated polymers.

11:20 AM

(ICACC-S17-029-2024) Nanoceramics-AI-based smart enernergy saving control for Agriculture 4.0 systems (Invited)

V. M. Castano*[1]

1. Universidad Nacional Autonoma de Mexico, Mexico

A novel Agro-industrial IoT technology and architecture for intelligent frost forecasting in greenhouses for Agriculture 4.0 via hybrid Artificial Intelligence (AI) and nanoceramics, is reported. The Internet of Things (IoT) allows the nanoobjects interconnection on the physical world using sensors and actuators via the Internet. The smart system was designed and implemented through a climatological station equipped with Artificial Neural Networks (ANN) and a fuzzy associative memory (FAM) for ecological control of the anti-frost disaster irrigation. The ANN forecasts the inside temperature of the greenhouses and the fuzzy control predicts the cropland temperatures for the activation of five output levels of the water pump. The results were compared to a Fourier-statistical analysis of hourly data, showing that the ANN models provide a temperature prediction with effectiveness higher than 90%, as compared to monthly data model.

11:50 AM

(ICACC-S17-030-2024) Water-based 2D material inks: From printed electronics to biomedical applications (Invited)

C. Casiraghi*[1]

1. University of Manchester, United Kingdom

Solution processing of 2D materials allows simple and low-cost techniques, such as ink-jet printing, to be used for fabrication of heterostructure-based devices of arbitrary complexity. Our group has developed a supramolecular approach able to provide highly concentrated, defect-free, printable and water-based 2D crystal inks, suitable for fabrication of large area arrays of photosensors on plastic [1], programmable logic memory devices [1], capacitors [2], transistors on paper [3-5] and printed memristors on rigid and flexible substrates [6]. Furthermore, our supramolecular approach allows to easily tune the charge of graphene, enabling production of amphoteric, cationic and anionic graphene dispersions [7]. Cytotoxicity tests confirm biocompatibility of the graphene ink, making it very attractive for applications in drug delivery and imaging [8].

S18 Ultra-High Temperature Ceramics

SYMPOSIUM 18: Advanced Characterizations and Simulations

Room: Coquina A
Session Chairs: Scott McCormack, University of California, Davis; Theresa Davey, Bangor University

8:30 AM

(ICACC-S18-024-2024) High-fidelity 3D microstructural characterization of ZrB_2 during hot-pressing (Invited)

R. Swanson[4]; D. A. Kosanovic[3]; M. Chapman[5]; M. D. Uchic[2]; 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 and Technology, Department of Materials Science and Engineering, USA
4. University of California, Davis, Chemical Engineering, USA
5. UES, Inc., 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, cracks, pores, etc. This is problematic when designing reproducible UHTC components. The goal of this project is to understand how these critical flaws evolve during hot pressing of ZrB_2 (a UHTC) by examining them in 3D. This study incorporates 3D imaging such as (i) preliminary in-situ high-temperature pressureless sintering X-ray μ-CT, (ii) ex-situ X-ray μ-CT, and (iii) 3D electron imaging and backscattered diffraction data collected at different stages of densification. 3D microstructure statistics along with unique observations of individual pore and secondary phase evolution will be presented. This data is brought together to give a holistic view of the densification of ZrB_2 during hot pressing at multiple length scales. This data will be incorporated into a process-structure-property (PSP) database for statistical modeling to reduce uncertainty during ZrB_2 processing.

9:00 AM

(ICACC-S18-025-2024) Retained flexural strength of Cf-ZrB2 UHTCMCs after arc-jet tests at 2200°C

D. Sciti*[1]; P. Galizia[1]; S. Mungiguerra[2]; R. Savino[2]; A. Airoldi[3]; A. Caporale[3]; A. Vinci[4]; L. Zoli[1]; M. De Stefano Fumo[5]

1. National Research Council of Italy, ISSMC (former ISTEC), Italy
2. University of Naples Federico II, Department of Industrial Engineering, Italy
3. Politecnico di Milano, Department of Aerospace Science and Technology, Italy
4. CNR - ISSMC, Italy
5. CIRA - Italian Aerospace Research Centre, Italy

Ultra High-Temperature Ceramic Matrix Composites (UHTCMCs) represent an innovative class of materials that potentially combine the high oxidation resistance of UHTCs with the damage tolerance and lightweight properties of CMCs. Few studies have been conducted so far to understand the impact of re-entry conditions on the mechanical properties. In this study, fully dense UHTCMCs based on ZrB2-SiC and Cf, were produced using a slurry impregnation and sintering process to create bars for flexural strength tests. Additionally, sample holders suitable for fitting into an arc jet chamber and holding the bars were fabricated from the same material. Five bars, each with dimensions 60 x 10 x 2.5 mm3 (length-width-thickness), were exposed to a plasma of dissociated air at a temperature of 2200°C for two minutes. Subsequently, they were weighed and subjected to 3-point bending tests to evaluate the impact of oxidation damage on their properties. Unexposed bars were also tested to assess their pristine strength. The oxidized layer was analysed using optical microscopy and SEM-EDS. The materials retained more than 80% of the initial strength despite the high temperature reached. Furthermore, prolonged tests lasting up to 4 minutes at 2200°C were conducted on additional samples, demonstrating the material's durability. The reusability of the sample holder for up to four cycles was also confirmed.

9:20 AM

(ICACC-S18-026-2024) Novel contactless measurement technique to determine the ultra-high temperature (>2000 °C) thermal conductivity and spectral emissivity of UHTCs

H. B. Schonfeld*[1]; M. Milich[1]; D. Robba[2]; L. Vlahovic[2]; K. Boboridis[2]; R. Konings[2]; E. Opila[3]; P. E. Hopkins[1]

1. University of Virginia, Mechanical and Aerospace Engineering, USA
2. European Union Joint Research Centre, Germany
3. University of Virginia, Material Science and Engineering, USA

New ultra-high temperature ceramics (UHTCs) are being developed as candidate hot structures for use as thermal protection systems with space and hypersonic vehicle applications. UHTCs experience extreme heat fluxes in flight, resulting in peak surface temperatures well above 2500 °C. Currently there is limited understanding of the thermal and radiative properties at relevant temperatures (>2000 °C). In this work we present a newly developed contactless measurement technique based on modulated laser heating and hyperspectral radiative pyrometry to measure the thermal conductivity and spectral emissivity of UHTC materials above 2000 °C. We validate this technique on standard metals tungsten and molybdenum by measuring their thermal and radiative properties from 2000 °C through their melting points. We then further evaluate our technique by measuring the thermal conductivities of TaC, HfC, ZrC and TiC from room temperature to 2000 °C, and compare our values with literature. Lastly, we measure the thermal conductivity and spectral emissivity of these materials above 2000 °C for the first time. Thermal conductivity and spectral emissivity measurements at relevant temperatures are crucial for evaluating and influencing the design of the next generation of extreme temperature thermal protection systems.

9:40 AM

(ICACC-S18-027-2024) Thermodynamic analysis of metal segregation in dual phase high entropy ceramics

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

1. Missouri University of Science & Technology, Materials Science and Engineering, USA
2. Duke University, Materials Science, Electrical Engineering and Physics, USA

Dual phase high entropy ceramics contain both a high entropy boride and a high entropy carbide. Previous research has shown that some transition metals in dual phase high entropy ceramics tend to segregate to either the boride or carbide phase. Typically, Ti and Nb segregate to the boride phase while Hf and Ta segregate to the carbide phase. The present work aimed to develop a thermodynamic model to predict the segregation behavior of metals in dual phase ceramics. Sets of binary solution equilibria were analyzed to predict trends in metal segregation. The governing reaction for equilibrium between the boride and carbide phases was defined and the standard Gibbs' free energies of reaction were calculated using FactSage. The relationship between the experimental data and the standard Gibbs' free energy was determined. Energy dispersive spectroscopy was used on dual phase ceramics to measure the metal content of each high entropy phase and the results were compared to the thermodynamic model.

10:20 AM

(ICACC-S18-028-2024) Carbon vacancies in high-entropy UHTC carbides (Invited)

T. Davey*[1]; Y. Chen[2]

1. Bangor University, Nuclear Futures Institute, United Kingdom
2. Tohoku University, School of Engineering, Japan

High-entropy or multi-principal component UHTCs, such as MC_{1-x} carbides (where the cation M=Ti,Zr,Hf,Nb,Ta), may have potential improved or tuneable properties such as melting point, hardness, ductility, and oxidation resistance. The individual carbides, such as ZrC_{1-x}, have a wide range of stoichiometry facilitated by significant numbers of carbon vacancies, but the vacancy behaviour in the mixed-cation carbides is unknown. Using DFT calculations, this work explores the effect of a multi-principal cation lattice on the carbon vacancy ordering, at 0K and considering finite temperature effects. Miscible and immiscible mixtures of both vacancy-disordered and vacancy-ordered carbides are identified with approximate temperatures for miscibility gaps calculated. Approximate order-disorder transition temperatures are also obtained for mixed-cation substoichiometric carbides and compared with the miscibility gaps in the same system. On this basis, the possibility of synthesising various structures of vacancy-ordered multi-principal cation compounds is revealed, alongside the required range of synthesis temperatures.

10:50 AM

(ICACC-S18-029-2024) Accelerated discovery of high entropy ultra-high temperature ceramics by machine learning and high throughput experiments

K. Wang*[1]; S. T. Misture[2]

1. Alfred University, USA
2. Alfred University, MSE, USA

Machine learning (ML) methods have been successfully applied to predict phase formation and properties of novel materials, such as high entropy materials. However, the application of ML approach in high entropy ceramics (HECs) used in extreme environments, such as nuclear reactors and hypersonic vehicles etc., remains limited due to the serious data scarcity as well as data quality issues. Herein, the machine learning (ML) model is trained on data collected via high-throughput experiments (HTEs). Because the experiment was conducted under the same conditions, HTEs enable generating high-quality dataset for ML training. The experimental validation is performed to examine the performance of the ML model. In addition, the ML is also applied to inversely understand the underlying physical mechanisms. For instance, we demonstrate that a mismatch of the bonds between boron and transition metals (δB-TM) dominates the formation of HEBs. We propose an empirical rule that HEBs favor forming a single phase when δB-TM < 3.66; otherwise, multiphase. The rule has a high accuracy of 93.33% for new HEBs predictions. Additionally, we are the first attempt to apply high-throughput experiments (HTEs) assisted data-driven strategy in the prediction of oxidation recession of HEBs-SiC ceramic composites.

11:10 AM

(ICACC-S18-030-2024) Discovery of Novel High-Entropy Transition Metal Borides: Theoretical Insights and Experimental Confirmations

I. Zhukova*[1]; M. Tatarkova[2]; A. Kovalcíkova[3]; Z. Chlup[4]; T. Csanadi[3]; I. Dlouhy[5]; D. Zagorac[6]; B. Matovic[6]; P. Tatarko[2]

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2. Institute of Inorganic Chemistry, Slovak Academy of Sciences, Department of Ceramics, Slovakia
3. Institute of Materials Research, Inorganic Chemistry, Slovakia
4. Institute of Physics of Materials, Inorganic Chemistry, Czechia
5. Institute of Materials Science and Engineering, Mechanics and Design of Materials, Czechia
6. "Vinča" Institute of Nuclear Sciences, Department of Materials Science, Serbia

High-entropy materials, especially transition metal borides, have emerged as a promising frontier in materials science, offering an array of advanced properties. In our study, we identified 126 unique transition metal boride systems, drawing from a palette of nine refractory metals. Through the strategic use of configuration generation, we targeted and evaluated disordered 5-metal borides, which show potential for diverse applications. A key of theory is the discovery of a structure that could be easily formed, has a stable phase and demonstrates a significantly reduced density, positioning it as an ideal candidate. Relying on the calculated formation energies, we have determined potentially easily synthesized compositions and their likelihood of synthesis. Our computational methodologies, anchored in DFT calculations using the Vienna ab initio simulation package (VASP), with experimental synthesis offers a holistic approach to the exploration of high-entropy systems. For empirical validation, selected compositions were synthesized using Spark Plasma Sintering (SPS) and characterized via X-ray diffraction (XRD), providing crucial insights into the tangible realization of our theoretical predictions.

S19 Molecular-level Processing and Chemical Engineering of Functional Materials

SYMPOSIUM 19: Gas-Phase Synthesis Approaches

Room: Ballroom 3
Session Chair: Peter Kroll, University of Texas, Arlington

8:30 AM

(ICACC-S19-022-2024) Choice of molecular precursors in magnetic field assisted chemical vapor deposition (mfCVD) (Invited)

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

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

Magnetic Field Assisted Chemical Vapor Deposition (mfCVD) enables additional control on film morphology, texture and composition besides well-known process parameters like pressure, temperature and time in conventional gas phase deposition methods. External field effects from electrical fields have been investigated and recently also the influence of external magnetic fields became more evident. Starting at relatively low magnetic field strength of 0.25 T significant changes in film texture, morphology and composition can be observed, depending on the precursor chemistry involved. Especially paramagnetic molecular compounds show a pronounced dependence on the external magnetic fields applied during the decomposition and deposition steps. This presentation will provide an overview on recent advances of the mfCVD process and the influence of different diamagnetic and paramagnetic molecular precursors for the formation of textured metal oxide and metal nitride surfaces.

9:00 AM

(ICACC-S19-023-2024) Synthesis of Manganese (II) complexes and Their Decomposition in Magnetic Field Assisted Chemical Vapor Deposition

M. A. Steiner*[1]; D. Patrun[3]; Z. Aytuna[4]; S. Mathur[2]

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2. University of Cologne, Institute of Inorganic Chemistry, Germany
3. University of Cologne, Inorganic/Materials Chemistry, Germany
4. Institute of Inorganic Chemistry, Department of Chemistry, Germany

Chemical vapor deposition is a versatile tool for thin film formation. The choice of process conditions and precursor gives fine control over the chemical composition of the thin film and the materials properties. Also an external stimuli like an applied magnetic field during the deposition introduces an additional parameter, which has been proven to alter morphology, chemical composition, and functional properties of the material. New single-source-precursors for MnF_2 and MnO_2 are synthesized and structurally resolved using single crystal X-Ray diffractometry and deposited on various substrates under the influence of magnetic fields with varying field strength. Thin films are subsequently analyzed using IR- and UV/Vis-spectroscopy as well as SEM, showing morphological changes in the obtained material, and increased grain growth under the influence of the magnetic field and different chemical compositions regarding of process conditions. Thin films obtained this way are possible candidates for water splitting applications. This work exemplifies the potential of magnetic field induced changes during material synthesis and broadens the conceptual toolbox for thin film formation.

S1 Mechanical Behavior and Performance of Ceramics & Composites

SYMPOSIUM 1: Ceramics processing–microstructure–mechanical properties correlation

Room: Coquina E

Session Chairs: Monica Ferraris, Politecnico di Torino; Kaitlin Detwiler, Air Force Research Lab

8:30 AM

(ICACC-S1-028-2024) Andra's R&D program on ceramic materials as alternative and innovative solutions for the geological disposal of high-level radioactive waste (Invited)

A. Debelle*[1]; F. Bumbieler[1]; E. Perret[2]; M. Box[2]; S. Miot[3]; C. Besnard[4]; S. Couillaud[4]; P. Ganster[6]; R. De Cassia Costa Dias[6]; I. Cornu[5]; F. Rossignol[5]

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2. Site Nouvelle Aquitaine, IRT St-Exupery, France
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4. Galtenco, France
5. Institute of Research for Ceramics (IRCER), UMR CNRS 7315, France
6. Université de Lyon, CNRS UMR 5307 LGF, Mines Saint Etienne, France

Andra is in charge of the project called Cigeo, an Industrial Centre for Geological Disposal of radioactive waste, where the high-level waste (HLW) will be disposed of underground, at ~500 m, inside horizontal tunnels (cells) dug in a claystone whose properties allow protecting the environment against the radioactivity for a very long timescale. For a shorter period (a few centuries), an additional (artificial) barrier, namely a container holding the waste primary package, will also be used. Currently, in Cigeo, low-carbon steel has been identified as the reference material for the HLW containers; another low-carbon steel is also selected for the cell liner. These steels will be subjected to severe challenges, the corrosion under anoxic conditions, already considered via strict safety criteria, being one issue to consider owing to the associated $H_{2(g)}$ production. To ensure an even safer disposal, bulk or coating materials not prone to corrosion are also investigated. In this framework, R&D activities are conducted by Andra and his partners to select, size, qualify and fabricate ceramic-based components instead of steel ones. Here, we present the work carried out on alumina-based, very thick monolithic or ceramic-coated containers. Ceramic matrix composites for the cell liner are also shown to be promising candidates.

9:00 AM

(ICACC-S1-029-2024) Processing of C/C-SiC based on thermoplastic polymers – additive manufacturing and hybrid yarns (Invited)

S. Schafföner*[1]; N. Langhof[1]; W. Freudenberg[1]; M. Moos[1]; J. Best[1]

1. University of Bayreuth, Chair of Ceramic Materials Engineering, Germany

C/C-SiC is used for demanding applications such as automotive brake discs and rocket nozzles. Usually thermoset polymers with a high char yield are used as the carbon precursor. Although there is a lot of experience with thermosets for C/C-SiC, these polymers also have limitations. For example, they contain hazardous constituents and there is no option for recycling. Yet, high-performance thermoplastic polymers crosslink without condensation reactions and allow a transfer of a wide range of polymer processing methods to CMC fabrication. However, challenges are often associated with the high viscosity of thermoplastics, which could result in insufficient fiber bundle infiltration, and with the remelting of the thermoplastics, which is associated with a lack of shape stability. Even so, an advantage of thermoplastics is the possibility of AM via fused filament fabrication incorporating carbon fibers. The present contribution therefore investigates this route to produce C/C-SiC using fiber-filled PEEK filaments. To prevent the remelting of the thermoplastic polymer, an oxidative crosslinking step was applied and optimized. Secondly, the development of a carbon fiber/PEEK hybrid yarn is described. For both technologies, physical, chemical and mechanical properties as a function of the microstructure are discussed.

9:30 AM

(ICACC-S1-030-2024) Thermomechanical behaviour of ex-pitch YS-15 carbon fiber after high temperature treatment

J. Braun*[1]; C. Sauder[2]; S. Le Bras[2]

1. CEA, France
2. CEA, DRMP, France

C/C composites and C/SiC composites are considered as basis for the processing of ultra high temperature materials. High rigidity is a major criteria for specific applications but is difficult to obtain because of matrix multicracking, resulting from mismatch in thermomechanical properties of the fibers and the matrix. Employing high modulus anisotropic carbon fibers is a way to solve this issue but textile shaping is highly challenging or nearly impossible for complex structures. One way to avoid this problem is to use a low modulus anisotropic ex-pitch carbon fiber for textile processing step and apply a high temperature thermal treatment on textile to raise the carbon fiber modulus. Granoc YS-15 carbon fiber has been supplied. Thermomechanical characterizations after High Temperature Treatments (HTT) from 1400 to 2400°C are presented. Increase of modulus is observed and especially above 2000°C, up to 750 GPa for 2400°C. Strength is not affected and stable form 1400°C to 2200°C HTT. Evolution of modulus and coefficient of thermal expansion are also presented. To approve such methodology, 2.5D weaving reinforcement were produced from YS15 fiber and then heat treated at 2400°C. Last, CVI-SiC infiltration was processed to evaluate mechanical tensile behaviour of such C/SiC composite compared to SiC/SiC composite.

9:50 AM

(ICACC-S1-031-2024) Microstructure and Properties of Low Si Content Reaction Bonded SiC

J. Wang*[1]; M. Aghajanian[1]

1. Coherent Inc., USA

Silicon carbide (SiC) is a hard chemical compound widely used as an abrasive and it is also widely used in applications requiring high hardness, refractory properties, high endurance and chemical stability, such as car brakes, ceramic plates in bulletproof vests, refractory furnace heating elements, wear-resistant parts for rocket engines and in semiconducting substrates etc. High density SiC is usually sintered or hot pressed at high temperature (>2000°C). Alternatively, reaction bonded SiC is produced by the infiltration of molten Si into preforms of SiC particles with carbon additive. Molten Si reacts with carbon additive, forming additional SiC and leaving some residual Si in the matrix. With a low processing temperature of ~1500°C and nominally zero process shrinkage, the reaction bonding process allows the cost-effective fabrication of large and complex shapes. The issue with reaction bonding for some applications is the presence of a residual Si phase. In this study, we optimized the microstructure of the reaction bonded SiC by controlling the carbon additive chemical structure, mixing ratio and the infiltration process, to minimize residual Si content in the matrix to less than 5 vol% (> 95 vol% SiC). This work represents a study that relates the residual Si content to the microstructure, mechanical and thermal properties (CTE and thermal conductivity) of the reaction bonded SiC.

10:30 AM

(ICACC-S1-032-2024) Development of oxide-based ceramic matrix composite with high thermal stability fabricated by novel oxide fibers

Y. Nawata[1]; I. Ohta*[1]; Y. Hirataka[1]; I. Yamashita[1]

1. Tosoh Corporation, Japan

Oxide-based CMCs (Ox/Ox CMCs) have lower cost and better stability against corrosive and oxidative environments than SiC-based CMCs. Therefore, Ox/Ox CMCs are promising candidates for thermally resistant materials as alternative to conventional nickel super alloys. However, the primary limitation of Ox/Ox CMCs in practical applications is their thermal stability. Previous commercially available Ox/Ox CMCs degrade after thermal exposure at up to 1200 °C. The degradation of Ox/Ox CMCs after thermal exposure is mainly due to thermal degradation of the reinforcing fibers. Fiber degradation due to grain-growth is the most essential problem for oxide fibers. We have developed uniform doping method (UDM) which is a novel method of doping oxide fibers with grain-growth inhibitors to improve the thermal stability of oxide fibers. Developed two Ox/Ox CMCs which is fabricated by UDM treated alumina fiber (TCA-01) and mullite fiber (TCM-01) exhibited no reduction in tensile strength after thermal exposure at 1200 °C for 1000 h. TCA-01 and TCM-01 had better thermal stability than the conventional Ox/Ox CMCs. Additionally, both the tensile strength and the creep lifetime of TCM-01 at 1200 °C were better than those of either a single-crystal nickel-based super alloy or a conventional Ox/Ox CMCs. Details of high temperature mechanical properties of TCM-01 are discussed.

10:50 AM

(ICACC-S1-033-2024) Aligned Nanofiber Reinforced Ceramic Matrix Nanocomposites with Ultrahigh Nanofiber Packing Density

J. Dai*[1]; S. Jagani[1]; L. Acauan[1]; P. B. Patel[1]; C. Hilman[1]; B. L. Wardle[1]

1. Massachusetts Institute of Technology, Department of Aeronautics and Astronautics, USA

Nanofibers like carbon nanotubes (CNTs) and boron nitride nanotubes (BNNTs) possess a unique combination of mechanical, thermal, and electrical properties that make them ideal reinforcements for ceramic matrix composites (CMCs). However, the current methods to fabricate nanofiber-reinforced CMCs often involve direct mixing, leading to damaged, randomly aligned, and agglomerated nanofibers of low volume fraction, contributing to limited property enhancement. To overcome these limitations, we developed the bulk nanocomposite laminating process for ceramic matrix composite (BNL4CMC). Instead of mixing, the polymer precursor of the desired ceramic matrix was infused into horizontally aligned nanofibers, followed by stacking, densification, and curing to form a laminate. The cured laminate was then heat treated at high temperatures to convert the polymer precursor to the ceramic matrix. Using this method, horizontally aligned-CNT reinforced silicon carbide (HACNT/SiC) laminates were successfully fabricated using polycarbosilane as the precursor. The fabricated laminates exhibited microstructures free of micro/macro-voids, with aligned and uniformly distributed CNTs of ultrahigh (~40-50 vol%) packing density. Detailed characterizations are underway to investigate the effects of highly packed and aligned nanofibers on the laminates' mechanical and multifunctional properties.

11:10 AM

(ICACC-S1-034-2024) Silicate interphases for SiC/SiC CMCs

O. Gavalda Diaz*[1]

1. Imperial College, Materials, United Kingdom

Interphases in SiC/SiC CMCs are crucial to reach the target toughness required in structural applications. Current interphases (BN and C based) have shown some limitations when used in the extreme environments defined by the aerospace and nuclear industries. However, there is still little information showing how other interphases should be designed to improve the performance of these materials. In this work we investigate how Y- and Yb- based silicate interphases deposited by Chemical Vapor Infiltration (CVI) could play a role in the design of future materials. We study via SEM in-situ micromechanical testing and TEM nanoscale characterisation how the microstructure, chemistry and thickness of interphases dominates the interfacial toughness and friction properties. We use this date to explain the failure of minicomposites.

11:30 AM

(ICACC-S1-035-2024) Manufacture of green ceramic micro-parts using a micro-machining process

A. Aliouat*[1]; V. Pateloup[1]; P. Geffroy[1]

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Ceramics are increasingly used for a broad range of industrial applications due to their unique physical and chemical properties, including strong corrosion resistance, extraordinary high-temperature stability, and favourable mechanical and chemical properties at elevated temperature. Those properties pose difficulty in processing ceramic materials to desired shapes and dimensions, especially in micro-machining because of the low mechanical strength of tiny tools and the extreme hardness of ceramics. The micro-machining of ceramic green body may be a solution to the problem. The aim of this work is to develop a new manufacturing process for microwave components for markets primarily linked to the telecommunications sector, at a controlled cost. Our recent work has enabled us to develop a new grade of material with very low dielectric losses and temperature drifts. The exceptional properties of this grade of material make it ideal for use as a dielectric resonator in the manufacture of filters operating at very high frequencies, typically for 5G applications (millimeter frequency bands). The idea is to make the most of this grade of material by developing filters with a planar (or 2D) geometry, with a view to reducing component manufacturing costs. In fact, we're planning a new manufacturing process based on strip casting coupled with laser cutting of raw ceramic strip.

11:50 AM

(ICACC-S1-036-2024) High-pressure synthesized HfB$_2$-based UHT ceramic for aerospace applications

T. Prikhna*[1]; A. Lokatkina[1]; P. Barvitskyi[1]; B. Büchner[2]; J. Werner[2]; R. Haber[3]; Z. Yasar[3]; S. Ponomryov[4]; M. Karpets[5]; R. Kluge[2]; A. Bondar[6]; V. Moshchil[1]; O. Borymskyi[1]; S. Rychev[1]

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Further development of modern aerospace programs requires new ultrahigh-temperature materials operating in neutral and oxidizing media at temperatures of 1600–2000 °C. The HfB$_2$-based ceramics is promising for this application. Sintering under high quasi-isostatic pressure (2 GPa) or hot pressing (30 MPa) and the availability of large-volume high-pressure tools made it possible to manufacture aircraft engine parts from the developed ceramics. The initial melting temperatures in vacuum were determined by the Pirani-Altertum method, the resistance to oxidation up to 1600 °C was studied by the methods of differential thermal (DTA) and thermogravimetric (TGA) analyzes on TAG16, Setaram instruments in an artificial 20 cm air flow. A study of the mechanical characteristics of materials showed that the addition of 30 wt.% SiC makes it possible to reduce the specific gravity of the material and increase its hardness and fracture toughness by 46% and 21%, respectively, slightly reduce the Young's modulus and increase the damping ability. The onset of melting temperature in vacuum was 2160 °C. A TGA study carried out in air showed that a significant increase in mass during the first heating starts from 1000 °C and then was practically constant ($4,2\times10^{-3}$ mg/K) up to 1600 °C; upon repeated heating, the increase in mass starts from 1400 ºC and increases linearly up to 1600 °C at a rate of $2,5\times10^{-3}$ mg/K.

S3 21th Intl Symp on Solid Oxide Cells Materials Science & Technology

SYMPOSIUM 3: Novel Processing / System Design

Room: Ballroom 1-2
Session Chairs: Scott Barnett, Northwestern Univ; Sebastian Molin, Gdansk University of Technology

8:30 AM

(ICACC-S3-025-2024) Functional Oxide Thin Film Fabrication by Flash Light Irradiation for Solid State Energy Devices (Invited)

Y. Kim*[1]; J. Park[1]; H. Lee[1]; S. Kim[1]

1. Hanyang University, Mechanical Engineering, Republic of Korea

Conventional time-consuming annealing process conducted at high temperature is a fundamental requirement for manufacturing functional oxide thin films used in solid state energy devices such as solid oxide fuel cells (SOFCs) and all-solid-state batteries (ASSBs). However, this procedure presents significant challenges when considering the commercial viability of such devices, as it can potentially compromise material performance due to unanticipated side reactions occurring at high temperatures. Therefore, in this study, an innovative approach utilizing the flash-light sintering method is proposed to create functional oxide thin films. It can dramatically reduce processing time to a few seconds range under standard pressure condition. Furthermore, recent investigations have revealed that the introduction of flash-light sintering into SOFC electrolyte and electrode materials, which are known to form secondary phases under high-temperature conditions, enables efficient sintering without undesirable side reactions, ultimately resulting in enhanced performance. This study encompasses solid oxide fuel cells and all-solid-state batteries, offering a promising pathway for the expansion of these technologies to other solid state energy devices.

9:00 AM

(ICACC-S3-026-2024) 3D Printing of functional ceramics for Solid Oxide Cells at IREC (Invited)

A. Sabato*[1]; S. Marquez[1]; A. Martos[1]; N. Kostretsova[1]; M. Lira[1]; I. Babeli[1]; M. Nuñez Eroles[1]; M. Torrell[1]; A. Tarancón[2]

1. IREC, Nanoionics and Fuel Cells, Spain
2. IREC / ICREA, Spain

Nowadays conventional Solid Oxide Cells (SOC) are based on multilayered functional ceramic materials produced by expensive and long manufacturing processes. Furthermore, conventional manufacturing techniques limit SOC geometries to typical planar or tubular ones. Here we present the recent breakthroughs obtained by innovative additive manufacturing technology of functional ceramic for SOC. The use of stereolitography (SLA) of Yttria Stabilized Zirconia (YSZ) electrolytes was explored at IREC in order to produce complex shaped SOC, impossible to obtain with conventional methods. New designs are considered in order to increase the active area of the devices leading to an increase of the performance per projected area. 3D printed electrolytes geometries are also tailored with special geometrical features (with 25μm of resolution) that allows not only an increase on the volumetric power density but also other key advantages such as better mechanical properties or complex shaped sealing features. The implementation of SLA for YSZ manufacturing at the same time can lead to advantages at stack level: i.e. reduction of the overall volume of the stack, use of flat thin interconnects, high pressure resistance without the use of vessels. In addition, hybridized (SLA + robocasting) multimaterial is presented together with co-sintering, characterization and preliminary electrochemical performance of the full printed device.

9:30 AM

(ICACC-S3-027-2024) How additive manufacturing is becoming a game changer for the production of clean hydrogen

C. Clark*[1]; S. Schweizer[1]

1. SAS 3DCERAM SINTO, France

Additive Manufacturing has been around for decades, but its innovation is accelerating continuously, pushing the boundaries to open up new ideas to explore for industrials as well as researchers. In a European collaborative project starting this year called HYP3D, AM is helping to improve the standards of hydrogen production with pressurized 3D printed SOEC stacks. The introduction of 3D-printed cells with the capacity to withstand elevated pressures during operation represents an unparalleled advancement. Furthermore, existing commercial SOEC technology currently lacks a feasible solution for the expansion of pressurized systems. The challenges that come from this technology lie in 3 main domains: production, storage, and supply chain. Here, the program is focusing on improving the clean production of hydrogen. Currently, the electrode supported cells are flat in shape, produced by traditional shaping methods, & the metallic interconnects are voluminous and complex in shape. 3DCeram's SLA technology is giving the possibility to print thin corrugated ceramic cells in 8YSZ, increasing the surface area by 60% which in turn enhances the performance of the cells. It's also enabling the cells to have more functionality, reducing the size of the interconnects and therefore the stack as a whole.

9:50 AM

(ICACC-S3-028-2024) In-The-Loop-Recycling of Fuel-Electrode Supported Solid Oxide Cells

S. Sarner*[1]; N. H. Menzler[2]; J. Malzbender[3]; B. De Haart[4]; O. Guillon[1]

1. Forschungszentrum Juelich, IEK-1, Germany
2. Forschungszentrum Jülich GmbH, IEK-1, Germany
3. Forschungszentrum Juelich, IEK-2, Germany
4. Forschungszentrum Juelich, IEK-9, Germany

To ensure successful implementation of solid oxide cells (SOCs) in a future hydrogen economy, reducing the system costs is crucial. SOC systems are expected to run at least for > 40,000 hours, with a targeted degradation rate of below 1 % per 1000 h. Currently, there are no recycling processes applied when the system reached the End-of-Use- (EoU) or End-of-Life- (EoL) state. However, integration into a circular economy might have a beneficial effect from both an economic and environmental perspective. While recycling of metals (interconnects, frames, base and top plates) is state-of-the-art, to date very less recycling strategies or pathways for used ceramics exist. On cell level, there are multiple valuable materials which should be subjected to recover, such as rare earth elements (La, Y, Gd, Ce), cobalt, and nickel. This study shows for the first time a scalable possibility to deal with ceramic SOC waste, using the example of the fuel electrode-supported cell design. Within the recycling process, the main weight share of the cell (~85 wt%) is reused in the production of new ceramic fuel electrode supports. These recycled substrates exhibit only slightly different behaviors in their chemical composition, microstructure, and mechanical stability. The cell fraction that is not suitable for substrate remanufacturing (~15wt%) will be recovered as raw material, with an emphasis on lanthanum recovery.

10:30 AM

(ICACC-S3-029-2024) Novel SOC stack design to enhance the uniformity of thermal conditions and thermo-electrochemical reaction zone in a commercial-scale stack (Invited)

W. Lee[1]; J. Lim[1]; M. Lang[2]; R. Costa[2]; I. Lee[3]; Y. Lee[3]; J. Hong*[1]

1. Yonsei University, Mechanical Engineering, Republic of Korea
2. DLR - German Aerospace Center, Germany
3. E&KOA, Republic of Korea

This study presents a novel solid oxide cell stack design that can solve the instability problem caused by the non-uniform distribution of local thermodynamic states inside a commercial-scale stack. Unbalanced distribution of temperature and species is a major limitation of conventional stack designs because certain regions of the stack are inevitably exposed to a much harsher environment than the rest, leading to faster degradation and reduced stability of the stack. To overcome these issues, we developed a novel stack design with enhanced uniformity of thermal conditions and reaction zone. First, the vertical gas manifold holes are rearranged to be placed in all perimeters, symmetrical in both diagonals. This arrangement makes the stack consist of four symmetric parts, and all thermodynamic variables and chemical reactions are distributed symmetrically. Second, the guide rib pattern and flow resistance field in the interconnect are designed to have uniform gas distribution. Finally, a new component, called a 'cover', is added in between the interconnect and cell to control the diffusive mass transport in a vertical direction. Through the control of mass transport in the vertical direction, chemical reactions can occur in much larger regions rather than concentrated in the fuel inlet regions.

11:00 AM

(ICACC-S3-031-2024) Reversible SOEC/SOFC system for zero emission energy network: Presentation and progress within the 24/7_ZEN project

M. Torrell[1]; L. Bernadet[1]; D. Montinaro[5]; D. K. Niakolas[4]; A. Souvalioti[4]; F. Zaravelis[4]; S. Neophytides[4]; F. Smeacetto[3]; E. Zanchi[3]; A. Morata*[1]; A. Tarancón[2]

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2. IREC / ICREA, Spain
3. Politecnico di Torino, Applied Science and Technology, Italy
4. FORTH/ICE-HT, Greece
5. SolydEra, SpA, Italy

The 24/7 ZEN European project is dedicated to conceiving and constructing a remarkably efficient reversible Solid Oxide Cell (rSOC) power system, boasting a capacity of 33/100 kW and seamless integration with both the electricity and gas grids. Leading the way is a pioneering, multidisciplinary consortium renowned for its groundbreaking innovations in energy management. The system crafted by this consortium will demonstrate its prowess in optimizing efficient pathways for Power-to-Gas-to-Power conversion, utilizing either hydrogen or natural gas as fuel sources. This plant's noteworthy feature lies in its ability to transition between modes in less than 30 min, delivering a round-trip efficiency of 45%, all while adhering to established standards and safety protocols. Efforts to boost cost competitiveness will be also set as a priority. Furthermore, this research highlights the foremost progress made in enhancing the performance of state-of-the-art rSOC components. These achievements include reducing degradation rates to a mere 0.4%/kh and attaining current densities of 1.5 A/cm^2 in both operational modes. These advancements primarily hinge on the utilization of novel materials for fuel and oxygen electrodes, such as Fe–Au–Ni/GDC and $La_{0.7}Sr_{0.3}Cu_{0.15}Fe_{0.85}O_{3-\delta}$, as well as innovative interconnect coatings employing electrophoretic deposition and other enhancements.

S6 Advanced Materials and Technologies for Rechargeable Energy Storage

SYMPOSIUM 6: Sodium batteries, potassium batteries, magnesium batteries and calcium batteries

Room: Ballroom 5
Session Chairs: Takashi Nakamura, Tohoku University; Shih-kang Lin, National Cheng Kung University

8:30 AM

(ICACC-S6-024-2024) Atomistic understanding of ion conduction and interfacial processes in emerging sodium batteries (Invited)

B. Narayanan*[1]

1. University of Louisville, Mechanical Engineering, USA

Rechargeable sodium batteries are sustainable alternatives to Li-ion technology owing to abundance of sodium (Na). Especially, all-solid-state sodium batteries (ASSSBs) using sulfide electrolytes (SEs), and aqueous sodium batteries (ASBs) hold tremendous promise due to their safety and low cost. Unfortunately, long-standing issues with (a) electrochemical instability of Na/SE interface in ASSSBs, and (b) lattice collapse of layered metal-oxide cathodes in ASBs during deep charging have precluded commercialization of these batteries. These challenges stem from a dearth of atomic-scale understanding of solvation chemistry, ion-transport, and electrochemistry at electrode/electrolyte interfaces. Here, we show how atomic-scale modeling can advance such understanding, and, in turn, accelerate design of next-generation sodium batteries. Specifically, I will highlight our recent successes in designing (a) fast Na^+ ion conductors via partial substitution of Na in Na_3SbS_4 with multivalent dopants (e.g., doping Na_3SbS_4 with trivalent cation with similar size as Na^+ can yield as high 30 times enhancement in Na^+ conductivity), (b) effective ionic-liquid interlayers that significantly improve electrochemical stability of Na/Na_3SbS_4 interface in ASSSBs, and (c) doping strategies that prevent prevents lattice collapse in layered birnessite cathodes in ASBs by immobilizing structural water.

9:00 AM

(ICACC-S6-025-2024) K_3MnO_4 as electrode material for K-ion Batteries

A. Sagot[1]; L. Stievano[2]; V. Pralong*[1]

1. CNRS ENSICAEN, France
2. Université de Montpellier, Institut Charles Gernardt Montpellier, France

Potassium batteries, with improved lifespan and capacity, will become advantageous compared to lithium due two major advantages: the low cost of potassium, the 7th most widespread element in the earth's crust, and it's low redox potential, similar to that of lithium[1–3]. To successfully develop commercially relevant potassium-ion batteries (KIBs), it is necessary to find new cathode materials with a reasonable potassium diffusion to improve the capacity[4]. For these reasons, we decided to explore the inexpensive and non-toxic K-Mn-O system. Hereby, we report for the first time the electrochemical behaviour of K_3MnO_4 phase[5]. This material, first reported by Hagenmuller[6], could be described as a non-dimensional structure built of isolated $[MnO4]^{2-}$ tetrahedra surrounded by complex potassium ion polyhedra. For the first time, we report the synthesis and electrochemical activity of a new form α-K_3MnO_4. The phase crystallizes in the orthorhombic space group Pnma with the following cell parameters: a = 7.75(4), b = 8.96(1), c = 7.91(8) Å and volume of V = 550.99 $Å^3$. The charge-discharge profiles of α-K_3MnO_4 show on the potential window 3.1 – 1.6 V, a reversible phenomenon is observed with the exchange $0.6K^+$ i.e. a capacity of 70 mAh/g at 2.37 V vs. K^+/K. Once the charge is extended above 3.1V, the process is no longer reversible. In our presentation, we will discuss the structure/properties relationships of this family of materials.

9:20 AM

(ICACC-S6-026-2024) Synthesis of highly dispersed metallic bismuth nanoparticles in CuO-Bi_2O_3-SiO_2 glass-ceramics for sodium ion battery anode

M. Kuroiwa*[1]; T. Honma[1]; Y. Daiko[2]

1. Nagaoka Univeristy of Technology, Department of Materials Science and Bioengineering, Japan
2. Nagoya Institute of Technology, Japan

Developing promising anode materials is essential for practical use in all-solid-state sodium-ion batteries. In this study, we propose a synthesis method of composite ceramics with dispersed bismuth nanoparticles by phase separation and crystallization of glass precursor. The glass system of interest is a CuO-Bi_2O_3-SiO_2 ternary glass, in which copper oxide is added because we expect to improve the electrical conductivity of the glass phase. Glass-ceramics were prepared by reduction heat treatment of the glass synthesized by the melting method, and their properties as anode active materials were clarified. Precursor glass in the composition of $xCuO$-$(85-x)Bi_2O_3$-$15SiO_2$ glass was prepared by melting at 1100°C for 30 minutes and then press quenching. Reduction heat treatment at 400°C and 500°C revealed the precipitation of Bi_2O_3, Bi, and Cu at 400°C and Bi and Cu at 500°C. Rietveld analysis and SEM observation confirmed that the particles were approximately 100 nm to 1 μm in size. The sample was heat-treated by reduction at 500°C, and charge-discharge tests was conducted using it as the active anode material in the CR2032 half-cell, and it operated at 1C for the theoretical capacity of 384.8 mAh/g. The sample is promising as an anode material for all-solid-state batteries by robust interface between anode-solid-electrolyte formed by heat treatment.

9:40 AM

(ICACC-S6-027-2024) Oxygen redox and delocalization of electrons in sodium-based layered cathode materials: A first principles DFT and GW study

K. Koester*[1]; P. Kaghazchi[2]

1. Forschungszentrum Juelich, Institute of Energy and Climate Research - Materials Synthesis and Processing (IEK1), Germany
2. Forschungszentrum Juelich, Germany

Activating oxidation of anionic species to achieve higher capacities and voltages attracted much attention in recent design of high-performance battery cathode-materials. Understanding the redox mechanisms and TM-O interactions of Na_xTMO_2 is necessary to develop even more elaborate design approaches. For sodium-ion batteries, mixing transition metals of different types and concentrations can promote anionic O-redox (in addition to TM-redox) and thereby increasing the battery capacity. In this work, we study the redox mechanism and stability of a novel cathode material ($Na_x[Co_{0.78}Ru_{0.22}]O_2$) by DFT calculations with PBE and hybrid HSE06 functionals. Besides TM-redox, activation of O-redox and increase of the capacity was found. Our calculations predicted a delocalization of electrons between Ru and O which increases the electrochemical stability of the cathode. Study the nature and the strength of these delocalizations call for more accurate descriptions of the electronic structures by hybrid functionals with appropriate mixing of HF-exchange and PBE to reproduce the results of higher levels of theory (GW). Our recent insights on this topic are summarized by presenting our results on model systems as well as novel cathode materials.

S7 18th Intl Symp on Functional Nanomaterials & Thin Films for Sustainable Energy Harvesting

SYMPOSIUM 7: 18th International Symposium on Functional Nanomaterials and Thin Films for Sustainable Energy, Environmental and Health Applications

Room: Coquina B
Session Chair: Muhammet Toprak, KTH Royal Institute of Technology

8:30 AM

(ICACC-S7-006-2024) Valorization of glycerol using binary alloys as electrocatalysts (Invited)

A. Anil[1]; J. Wwhite[2]; E. C. dos Santos[3]; T. Kubart[4]; R. Brucas[1]; L. G. Pettersson[5]; A. Cornell[2]; G. Salazar Alvarez*[1]

1. Uppsala University, Materials Science and Engineering, Sweden
2. KTH Royal Institute of Technology, Division of Applied Electrochemistry, Sweden
3. Tohoku University, Advanced Institute for Materials Research, Japan
4. Uppsala University, Electrical Engineering, Sweden
5. Stockholm University, Physics, Sweden

The production of biodiesel in the EU in 2022 amounted to ca. 15 Mm^3 with glycerol as the main byproduct.1 The generation of valuable glycerol oxidation products and hydrogen production at the anode become interesting from an economic and scientific standpoint.2 Noble metals catalysts suffer from well-known drawbacks, alloying them with other transition elements can potentially solve many of these drawbacks.3 In this presentation wIe will show how thin films based on Pt- and Pd- alloys are prepared using sputtering techniques and their use to electrooxidise glycerol in aqueous media. The oxidation pathway and presence of intermediate products is modelled using DFT calculations. Analysis of the oxidation products is carried out using high-performance liquid chromatography. Structural and chemical changes to the catalysts under operando conditions are followed using synchrotron grazing incidence X-ray diffraction where we can correlate the changes in activity due to the leaching of non-metal cations into the reaction media.

9:00 AM

(ICACC-S7-007-2024) Electrodeposition of binary alloys on gas diffusion electrodes for CO2 electroreduction (Invited)

T. Andreu*[1]; M. Amazian[1]; M. Sarret[1]

1. Universitat de Barcelona, Spain

Carbon dioxide reduction reaction (CO2RR) is a key technology for the chemical industry in a highly electrified energy scenario. Besides the need of the development of efficient and selective catalysts, fabrication routes should be easily escalable. In this context, we present electrodeposition as an effective and versatile tool to obtain thin film layers of electrocatalysts on microporous layers of gas diffusion electrodes. As model catalysts, AuCu alloy, CuIn alloy and pure metals (Au, Cu, In) were electrodeposited on GDE and tested in a flow cell employing different electrolytes. Results show that the presence of halides in the catholyte can avoid the non desirable hydrogen evolution reaction and favors the formation of carbon monoxide or formic acid, effect that is enhanced by the use of binary alloys. On the other hand, the high current densities attained using GDE enables CO2RR at neutral pH on sulphate electrolytes.

9:30 AM

(ICACC-S7-008-2024) Nitrogen Catalysis by an Induced Polarization through a Piezo-assisted Catalysis

B. Witulski*[1]

1. University of Cologne, Institute of inorganic chemistry, Germany

Despite recent advances in sustainable chemical synthesis, finding technically feasible solutions for the fixation of nitrogen (N_2) remains challenging. The N_2 fixation usually leads to ammonia (NH_3) that has high energy content (18.8 MJ kg^{-1}), low storage costs, and has been done for more than a century using the Haber-Bosch method that demands high temperatures (400 – 600 °C) and pressures (20 – 40 MPa). To achieve a less time- and money-intensive process, electrochemical NH_3 production arises as a feasible path to synthesize a green fertilizer with zero carbon footprint. Piezoelectric materials can be activated through mechanical vibrations, thereby decreasing the overpotential required to perform the nitrogen reduction reaction. The piezomaterial ($Na_{0.5}K_{0.5}NbO_3$, KNN) was synthesized via the sol-gel route using alkoxide precursor and deposited on stainless steel and titanium substrates by spray coating. Silver (Ag) was used as the electrocatalyst and deposited over the KNN layer. This work will address the use of the as-synthesized working electrode (KNN + Ag) for the piezo-enhanced nitrogen fixation. It was observed that the created transient dipoles in surface-absorbed non-polar N_2 molecule increases the N_2 coverage on the electrocatalyst surface. Furthermore, the ammonia production could be enhanced by more than four times with piezo-actuation compared to without piezo-actuation.

9:50 AM

(ICACC-S7-009-2024) Nanostructured catalysts for photo-, piezo- and electrochemical processes (Invited)

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

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

Nanostructured metal oxide semiconductors are well known catalysts for small molecule activation in photo-, piezo- and electrochemical processes. The need for more sustainable energy conversion technologies starting from solar water splitting for hydrogen production, over CO_2 reduction for solar fuel applications to nitrogen activation for fertilizer production demand more efficient catalysts and alternate catalytic cycles. New momentum in this field stems from promising synergistic catalytic approaches like photoelectrocatalysis (PEC), piezophotocalaysis as well as piezoelectrocatalysis, which demand tailored semiconductor materials with suitable band-gap, stability, conductivity and activity. This presentation will highlight different approaches for fabrication of nanostructured metal oxide catalysts using gas phase deposition methods and their application in photo-, piezo- and electrochemical processes for small molecule activation.

10:40 AM

(ICACC-S7-010-2024) Advanced magneto-structural characterization of magnetic-based oxide nanocomposites (Invited)

M. Estrader*[1]; A. Roca[2]; A. López-Ortega[3]; R. U. Ichikawa[4]; I. Peral[5]; X. Turrillas[6]; D. del-Pozo-Bueno[7]; M. Varela[8]; S. Estradé[7]; F. Peiró[7]; J. Nogués[2]

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2. Catalan Institute of Nanoscience and Nanotechnology (ICN2), Spain
3. Universidad Pública de Navarra, Spain
4. Nuclear and Energy Research Institute (IPEN/CNEN-SP), Brazil
5. 5Department of Physics and Materials Science, University of Luxembourg, Luxembourg
6. Institut de Ciència de Materials de Barcelona- CSIC, UAB Campus, Spain
7. LENS-MIND, Departament Enginyeries Electrònica i Biomèdica and Institute of Nanoscience and Nanotechnology of the University of Barcelona (IN2UB), Spain
8. Departamento de Física de Materiales e Instituto Pluridisciplinar, Universidad Complutense de Madrid, Spain

Batteries, electro/photochemical catalysts for energy storage and production are constantly renewed with advanced materials exhibiting higher performance. Within these systems, hybrid materials where each counterpart has dissimilar properties are gaining much interest as synergistic properties may arise. Interestingly, in some cases the external activation of one property might induce changes into the other one, often showing increased performances for the battery/catalytic processes. In the last few years, the magnetic effects of either an external magnetic field or even a magnetic material as the hybrid counterpart of the battery/catalyst has emerged as a hot topic owing to the significant improvement of the respective performances. I will first explain some of these exciting examples. Subsequently I will move into the cases when such hybrid composites fall within the nanoscale and the implications this might have. For instance, for nanocomposites, the structure and composition are often not straightforward to disentangle. Having unknown compositions or phases within a magnetic nanomaterial can hamper the interpretation of the magnetic properties, which are, ultimately, responsible for the improvement of the battery/catalytic processes. Thereby, I will explain and show some examples and the related characterization techniques used at the nanoscale for magnetic-based nanocomposites.

11:10 AM

(ICACC-S7-011-2024) Methane Oxidation Reaction Pathway and Activity of CeO_2 Catalyst with a Variation of Surface Fermi Level: A Multi-scale Simulation Study

S. Ji*[1]; H. Ko[3]; H. Choi[1]; S. Cho[2]

1. University of Cologne, Department of Chemistry, Germany
2. Ajou University, Republic of Korea
3. Korea Institute of Ceramic Engineering and Technology (KICET), Republic of Korea

Catalytic oxidation of methane over CeO2 has been investigated to enhance the reactivity. However, there is still a lack of understanding in greatly changeable catalytic activities of CeO2 even small changes by material engineering e.g. doping or cocatalyst decoration and so on. We studied the underlying mechanism of synergistic catalytic methane oxidation activity change of CeO2 by combining density functional theory (DFT) calculations with mathematical models. Our new theoretical model of surface Fermi level dependent reaction activity enables us to precisely predict reaction activities and pathways of wide band gap semiconductors by explaining how bulk impurity doping and cocatalyst deposition on CeO2 can greatly affect the reactivity in methane oxidation.

11:30 AM

(ICACC-S7-012-2024) Selective placement of modifiers on hematite thin films for solar water splitting

F. L. de Souza*[1]

1. Brazilian Center for Research in Energy and Materials, Brazil

The design of nanostructured materials for photoelectrochemical water splitting relies on a detailed understanding of the reactional bottlenecks. Here, we introduce a single polymeric precursor solution that enables the design of hematite (α-Fe_2O_3) with synergistic bulk and interfacial engineering using trivalent (Al^{3+}, Ga^{3+}), tetravalent (Zr^{4+}, Ti^{4+}, and Hf^{4+}) and a cocatalyst ($NiFeO_x$). The solution causes trivalent to dope hematite lattice to reduce polaronic effects, while simultaneously induces tetravalent enrichment at both surface and grain boundaries, improving charge separation and reducing recombination. Zr^{4+} and Hf^{4+} also led to a refined microstructure derived from interface stabilization, which associated with trivalent bulk doping and $NiFeO_x$ electrodeposition resulted in a photoanode with 65 to 85% of overall efficiency. As a consequence, the modified hematite ultrathin film (176nm-thick) delivered a water oxidation photocurrent of 2.30 to 4.5 mA cm^{-2} in contrast to 0.37 mA cm^{-2} for the pristine system measured at 1.23 V against hydrogen reversible electrode (RHE). The results suggest the simplicity of this new polymeric solution may offer a cost-effective, scalable and versatile alternative for multiple chemical modifications in oxides beyond hematite.

S8 18th Intl Symp on APMT for Structural & Multifunctional Materials & Systems

SYMPOSIUM 8: Advanced composite manufacturing technologies, hybrid processes I

Room: Coquina F
Session Chair: Akihiko Ito, Yokohama National University

8:30 AM

(ICACC-S8-026-2024) Exsolution and Coarsening in Metal-Oxide Systems (Invited)

I. Reimanis*[1]

1. Colorado School of Mines, USA

Microstructure evolution in mixed metal oxides exposed to various redox conditions is important to understand for applications such as catalysis, solid oxide fuel cells, and solar thermal water splitting. In particular, the exsolution and subsequent coarsening of transition metal particles in oxides strongly influence performance. An overview of exsolution and coarsening of Ni in yttria stabilized zirconia and yttria stabilized barium zirconate is provided. Thin films of barium zirconate provide a useful model system to examine the importance crystal orientation in these processes.

9:00 AM

(ICACC-S8-027-2024) Fluidized Bed Chemical Vapor Deposition – a versatile technique for the preparation of ceramic composites

G. L. Vignoles*[1]; N. Bertrand[1]; A. Guette[1]; G. Chollon[1]; H. Plaisantin[1]; S. Couthures[1]; A. El Mansouri[1]; T. Da Calva[1]

1. University Bordeaux, LCTS - Lab for ThermStructural Composites, France

Ceramic composites contain by definition reinforcing phases dispersed in a matrix. One of the sensitive issues of these materials is the compatibility between reinforcements and matrix, during processing and during usage, usually at high temperatures. It is therefore highly desirable in general to provide some protection to the reinforcing phase by a coating. In the case of powders or discontinuous fibers, among other methods, Fluidized-Bed Chemical Vapor Deposition (FB-CVD) is very attractive. Indeed, it is rather versatile and easy to scale up, once it has been suitably controlled. Here we will present some aspects of process engineering necessary

for a correct operation of this process, including thermal engineering and the study of fluidization and fluid transfer; then, examples of deposition of several coatings on powders and discontinuous fibers will be shown, characterized and discussed.

9:20 AM

(ICACC-S8-028-2024) Novel bioceramic/polymer composites from engineered photocurable emulsions

E. Bernardo*[1]; H. Elsayed[1]; B. Zavan[2]

1. University of Padova, Department of Industrial Engineering, Italy
2. University of Ferrara, Dept. of Translational Medicine, Italy

Hydrated calcium nitrate is an interesting precursor for bioceramics, e.g. in sol-gel formulation for bioglasses. It is not soluble in organic liquids, but its low melting point enables homogeneisation by emulsification. In this paper we discuss the feasibility of novel composites from vat photopolymerization of emulsions determined by the dispersion of droplets of molten hydrated calcium nitrate in biocompatible, bio-derived photocurable resin. The stability of the emulsions is favoured by the adoption of surfactants and slight heating of the vat (at 40-50 °C), realized by an electric-heated printing head. Calcium nitrate nano-particles are later completely transformed into carbonated hydroxylapatite simply by immersion of the as-printed scaffolds in sodium phosphate solution (in a sealed containeir, at 60 °C), at varying molarity and immersion times (from 1 to 7 days). The absence of any high temperature firing step prevents any degradation of the organic phase and leads to composites with a preudo-ductile failure. Preliminary cell tests confirm both biocompatibility and bioactivity.

9:40 AM

(ICACC-S8-029-2024) Investigation of interfaces in Porous Silicon Nitride–Zirconia–Graphene Composite

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

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

The influence of the various content of the multilayered graphene (MLG) on the structural and mechanical properties of the final bulk porous silicon nitride-zirconia (Si3N4-ZrO2) based ceramics was investigated. The ceramic composites were prepared in the form of the laminated structure with different (5-30-5 wt% and 30-5-30 wt%) MLG content by hot isostatic pressing. Homogeneous distribution of the MLGs, a completed phase transition from α to β-Si3N4 in case of 5 wt% MLG have been observed. The structural examinations revealed that the multilayered graphene and zirconia particles owing to their different sizes and shapes influenced the porous microstructure evolution and the related mechanical properties of the composites. The sandwich structures have been characterized by enhanced mechanical properties compared to reference ceramic with 30 wt% MLG. The position of the layer with higher graphene content, high ratio of α/β phase of Si3N4 and higher porosity had crucial effect on the final mechanical properties.

S9 Porous Ceramics Novel Developments and Applications

SYMPOSIUM 9: Innovations in Processing Methods & Synthesis of Porous Ceramics

Room: Coquina D
Session Chairs: Tobias Fey, Friedrich-Alexander University Erlangen-Nürnberg; Wei Zhai, National University of Singapore

8:30 AM

(ICACC-S9-001-2024) Fabrication of Functional Porous Ceramics by Additive Manufacturing combined with Colloidal Assembly (Invited)

B. Winhard[1]; L. Grassi Maragno[1]; A. Gómez Gómez[1]; K. P. Furlan*[1]

1. Hamburg University of Technology, Integrated Materials Systems Group, Germany

Micro or nanoscale resolution in 3D printing is often associate to costly equipment and long printing times. In this talk I will show the fabrication of nanostructured functional porous materials by using a smart combination of an additive manufacturing (AM) process, direct writing, with colloidal assembly of low-viscosity additive-free water-based suspensions, which enables fast printing times while reaching high-resolution in terms of 3D structural features. We demonstrate that the conventional AM "line-by-line" approach cannot be used for such low-viscosity additive-free suspensions, due to uncontrollable contact line slipping. Thereby, we proposed a "comb-strategy" to enable the 3D printing of colloidal suspensions. By optimization of the printing parameters, the printing of crack-free cm-sized homogeneous colloidal coatings is possible. Those are then transformed into functional macroporous ceramics by using atomic layer deposition. Our work highlights the advantages of combining additive manufacturing with colloidal assembly (AMCA) and atomic layer deposition (ALD) for novel applications of colloidal-based 3D printed structures.

9:00 AM

(ICACC-S9-002-2024) Geopolymer Granules for Environmental Applications

P. Colombo*[1]; M. Muracchioli[1]; G. Franchin[1]

1. University of Padova, Industrial Engineering, Italy

Wet granulation was employed to fabricate geopolymer granules with the goal of using them as a substrate for CO2 adsorption in fluidized bed columns system. A geopolymer powder was granulated using a geopolymer slurry of the same composition, to ensure obtaining a granule with the desired composition, leading to granules with a size ranging from a few tens of microns to several hundred microns. The granules, in some cases with a surface modified with APTES or mixed with zeolites, were tested for CO2 adsorption. Granules with APTES addition showed a quite good CO2 adsorption capability, better than geopolymer and zeolites granules, where the zeolites powder was found to be well distributed on the surface of the granules.

9:20 AM

(ICACC-S9-003-2024) Novel Strategies for Strength Enhancement of Reticulated Ceramic Foams: Chromia Doping and Gorilla Glass Coating

P. V. Zander[1]; D. Schrake[1]; U. Betke*[1]; M. Scheffler[1]

1. Otto-von-Guericke University, Institute for Materials and Joining Technology - Non-metallic Materials and Composites, Germany

The mechanical strength of reticulated ceramic foams made by the Schwartzwalder technique is typically low due to specific defects in the ceramic struts. General strategies for strength improvement are infiltrating the struts or recoating the outer strut surface area. In combination with transformation toughening, e. g. in Al_2O_3-ZrO_2 (ZTA) composite ceramics, a compressive strength increase up to a factor of 3 can be reached. Within this work two approaches for improving the mechanical strength of alumina foams are presented: Chromia doping of the strut material and alkali aluminosilicate glass recoating of the struts with a subsequent ion exchange step. The concept of chromia doping of alumina is the generation of an internal compressive stress inside the strut material due to the larger ionic radius of Cr^{3+} compared to Al^{3+}. By this, an improved fracture toughness of the strut material is expected, similar to ZTA ceramics. In the second approach, a recoating with an sodium aluminosilicate glass is used. The glass composition is suitable for an ion exchange of Na^+ by larger K^+ ions, which generates a compressive stress in the glass coating layer and improves its fracture toughness. This concept is well known under the brand of "Gorilla Glass". Both approaches are evaluated regarding their effect on the microstructure and the compressive strength of alumina foams.

9:40 AM

(ICACC-S9-004-2024) Sol-Gel production of BCZT for porous structures – applications, challenges and opportunities

M. Weichelt*[1]; E. Wolf[1]; S. Simon[1]; D. Köllner[1]; T. Fey[2]

1. Friedrich-Alexander-Universität Erlangen-Nürnberg, Materials Science, Germany
2. Friedrich-Alexander University Erlangen-Nürnberg, Department Material Science and Engineering, Germany

The sol-gel process is often used among the various methods of producing BCZT, as it offers precise control of composition, low temperature production and fine particle size. Here the composition Ba0.85Ca0.15Zr0.1Ti0.9 is prepared and the resulting powder is shaped in various ways before the physical and functional properties are investigated. Piezoelectric light-frame honeycomb and Kelvin cell structures were fabricated because of their suitability for applications as sensors, actuators and energy harvesters due to their high sensitivity, low acoustic impedance and improved signal-to-noise ratio. The powder produced by the sol-gel process was investigated in more detail as the piezoelectric properties of uniaxially pressed and injection moulded samples differed significantly. Therefore, the samples were heat treated and functional properties were analysed using Berlin-Court method. The SEM microstructure of the pressed samples shows an inhomogeneous microstructure resulting in d_{33} values around 42-70 pC/N. These values can be increased using another forming technique, injection moulding. Before the powder can be used in an injection moulding mass, it must be hydrophobised to make the surface water repellent. For these samples an increased d_{33} up to 288 pC/N was observed. The influence of hydrophybization on pyhsical properties and use in applications was investigated in more depth.

10:20 AM

(ICACC-S9-005-2024) Design and Characterization of Lignocellulosic-based Foams for Functional Applications

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

1. University of North Dakota, Mechanical Engineering, USA

Lignocellulosic biomass is the most abundant organic polymer on the earth. Recently, Lignocellulosic biomass-based materials attract attention as sustainable, renewable, and economical alternatives to petroleum-derived products. With the developments of conversion technologies, Lignocellulosic-based materials have a wide range of applications in biomedical products, bioplastics, building materials, insulation materials, water treatment, and energy storage, etc. In this paper, we will report on the latest development of design of biofoam by using different types of biomasses and characterize biofoam for multiple applications. We found that the properties of biofoams can be tailored by processing conditions and the selection of biomass types. The biofoam pyrolyzed at 300°C showed hydrophobic behavior which can absorb oil up to 80% of its own weight makes it a candidate of oil absorbent. The thermal conductivity test indicated that biofoam is a good insulator and some compositions are competitive to hempcrete or light concrete. The biofoam pyrolyzed at higher temperatures presented hydrophilic behavior. By further carbonization and activation, the biofoams have potential applications in water treatment and energy storage.

10:40 AM

(ICACC-S9-006-2024) Colloidal Processing Empowered by Emulsions: A Path to Hierarchical Porous Ceramics (Invited)

W. Zhai*[1]

1. National University of Singapore, Mechanical Engineering, Singapore

Hierarchical porous materials, such as bones and bamboos, exhibit exceptional mechanical efficiency and functionality. Inspired by nature, hierarchical porous ceramics with multi-scale porosity distributions have emerged, which show great potential for enhanced functional and structural properties. To unlock their potential, the development of material processing technologies to replicate the hierarchical porous structures found in nature is essential. Conventional colloidal processes, such as direct ink writing and freeze casting for porous ceramics, often yield limited porosity within a single scale. On the other hand, by utilizing emulsion inks, emulsion templating technique is well-established for porous materials through the incorporation of a secondary non-miscible solution. Combining emulsion inks with traditional colloidal processes has the potential to produce porous ceramics with hierarchical porosities from both methods. In this talk, I will present our recent progress in emulsion-based colloidal processing, specifically in direct ink writing and freeze casting of emulsion inks, to create hierarchical porous ceramics and aerogels. Our research aims to provide a simple framework for designing and applying emulsion inks in colloidal processing for a wide range of applications, from lightweight structures to tissue engineering scaffolds and high-performance acoustic absorbing materials.

11:10 AM

(ICACC-S9-007-2024) Production of Carbon Foam Made from Coal at Atmospheric Pressure (Invited)

R. A. Olson*[1]; N. Smith[1]; T. Englebert[1]

1. CONSOL Innovations, R&D, USA

CONSOL Innovations LLC (formerly CFOAM LLC) has been manufacturing vitreous carbon foam from coal in panel form for several years. The process is currently set up in batch mode as two steps, where the first step is conducted at a high pressure (about 400 psi). In order to reduce cost and increase production rates, a new process for making carbon foam is being developed that can be performed at atmospheric pressure. This advance in turn could enable the development of a continuous manufacturing process. The microstructure of foam produced at atmospheric pressure is quite different from that produced at 400 psi. In an effort to affect the microstructure and properties, a variety of additives to the coal were employed. Results of the variation in processing techniques, their impact on microstructure, as well as some of the properties and performance characteristics of carbon foam made using this new process will be discussed. This project was supported by a cooperative agreement with the Department of Energy – National Energy Technology Laboratory (DE-FOA-0002185, Area of Interest 4: Coal-Derived Carbon Foam Produced via a Continuous Process, award number DE-FE-0031992).

11:40 AM

(ICACC-S9-008-2024) Production of porous ceramics using electrostatic interaction control (Invited)

M. Cerbelaud*[1]; G. Michaud[1]; A. Aimable[1]; A. Videcoq[1]

1. IRCER, France

The self-assembly of colloids is an area of study that has developed considerably in recent years [1]. In the case of charged colloids, different arrangements are obtained as a function of electrostatic interactions, some of which are of interest for the formation of porous ceramics. In this presentation, we will focus on the use of heteroaggregation to form porous ceramics. Firstly, we will study the shaping of porous ceramics using emulsified suspensions. The stabilization of emulsions in the presence of particles, also known as Pickering emulsions, generally requires partially hydrophobic particles to penetrate the interface between the two phases of the emulsion. As ceramic particles are generally hydrophilic, they must first be surface modified, for example by adsorption of surfactants. However, it has been shown that emulsions can also be stabilized by mixed suspensions of hydrophilic particles subjected to hetero-aggregation without the addition of hydrophobic additives [2]. Using an original approach based on the coupling of simulations and experiments, we will show which mechanisms can explain the stabilization of these emulsions. We will then present a second strategy for shaping porous ceramics using a new shaping process based on microfluidics in which suspensions containing porogens heteroaggregate.

S11 Advanced Materials and Innovative Processing Ideas for Production Root Technologies

SYMPOSIUM 11: New concepts and emerging technologies for enhanced product performance II

Room: Ponce de Leon
Session Chair: Chisung Ahn, Korea Institute of Industrial Technology

8:50 AM

(ICACC-S11-014-2024) β-MoO_3 whisker as a neutron irradiation target material for radiopharmacy production (Invited)

H. Suematsu*[1]; N. Chu[1]; Y. Yang[1]; T. Do[1]; T. Nakayama[1]; K. Niihara[1]

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

^{99}Mo is a mother nuclide of ^{99m}Tc, which emits a γ-ray to be used in radiopharmacies. Currently, ^{99}Mo has been recovered from highly enriched uranium spent fuel in nuclear reactors, which have nuclear security risks. Instead, a nuclear reaction method to irradiate neutrons onto α-$^{98}MoO_3$ targets has been investigated. Comparing to conventional α-MoO_3, we have grown β-MoO_3 whiskers by an evaporation method and shown superior ^{99}Mo extraction characteristics to water because of the larger spacing between Mo-O layers and the hot atom effect. To grow the β-MoO_3 whiskers, 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.

9:20 AM

(ICACC-S11-015-2024) Electrochemical Performance and Degradation Mechanism if Rechargeable Zinc Ion Battery through Al_2O_3-Coated Zinc Anode and MnO_2 Cathode Configuration

Y. So*[1]; S. Lee[1]; S. Lee[3]; S. Lee[3]; J. Kim[2]; S. Mhin[1]

1. Kyonggi University, Republic of Korea
2. Daegu Mechatronics & Materials Institute, Republic of Korea
3. Advanced Institute of Convergence Technology, Republic of Korea

As the demand for energy storage systems (ESS) continues to grow, the development of the safe battery technologies without the risk of fire and explosion becomes increasingly important. Rechargeable aqueous zinc ion batteries (AZIBs) have gained significant attention as a promising alternative to Lithium-ion batteries due to their benefits, which include the use of water-based electrolyte, high energy density per volume, and cost-effectiveness. Several phenomena, including hydrogen evolution reaction, corrosion, and dendrite growth, structural degradation, formation of unwanted compounds lead to the degradation of the electrochemical performance of AZIBs. In this study, we investigate the effect of the MnO_2 cathode and Al_2O_3-coated Zn anode on the electrochemical performance of the AZIBs. The experimental results show that the cycle performance of a full cell consisting of an Al_2O_3-coated Zn anode and MnO_2 cathode is comparable to that observed for the bare Zn and MnO_2 configuration. Through this, it is explained that the poor cycle characteristics of AZIBs are due to the phase transformation and structural collapse, this result highlights the importance of exploring optimized materials for both anodes and cathodes in AZIBs to improve the cycle performance.

9:40 AM

(ICACC-S11-016-2024) Fabrication of MXenes-based films by in situ HF etching for electromagnetic interference shielding application

S. Nguyen*[1]; A. Okawa[2]; T. Nakayama[3]; T. Do[4]; H. Suematsu[5]

1. National Institute of Technology, Kushiro College, Department of Creative Engineering, Japan
2. Tohoku University, Institute of Multidisciplinary Research for Advanced Materials, Japan
3. Nagaoka University of Technology, Japan
4. Nagaoka University of Technology, Nuclear System Safety Engineering, Japan
5. Nagaoka University of Technology, Extreme Energy-Density Research Institute, Japan

MXene is a new family of two dimensional (2D) transition metal carbides or nitrides that has many potential applications. One of their most interest application is electromagnetic interence shielding materials for next-generation of mobile and electronic devices. However, the fabrication of MXenes is often related the use of HF to etch and delaminate 2D flakes (such as Ti3C2) from MAX phase precusors (such as Ti3AlC2), that is toxic and unfriendly with environment. This work presents an in-situ HF generating etching method, in which the HF etching media was replaced by HCl and LiF in sealed container. The etching rate can be controlled by changing the temperature of the container with an silicone bath. The size of MXene flakes, the effectiveness of the method are evaluated using SEM, TEM, and XRD, etc. After etching, the received MXenes flakes can be processed to become thin films by vacumn-assisted filtering or different methods. The products then can be used as electromagnetic interference shiedling films which is explained in-details in the paper. The presented method is very safe and quite simple, therefore is promising to the fabrication of MXenes materials and its potential products.

S16 Geopolymers Inorganic Polymers and Sustainable Construction Materials

SYMPOSIUM 16: Mechanical properties

Room: Coquina C
Session Chair: Henry Colorado L., Universidad de Antioquia

9:00 AM

(ICACC-S16-017-2024) Geopolymers for fire protection applications: Dense materials, foams and coatings (Invited)

A. Gharzouni*[1]; S. Rossignol[1]

1. IRCER, France

Nowadays, more and more studies are focusing on the search of low-carbon materials for fire protection applications. In this context, this study aims to the determination of geopolymer formulations for high temperature performance. Several formulations based on different aluminosilicates (metakaolins, laterite, volcanic ash), potassium and/or sodium silicate solutions and mineral fillers have been studied. Dense materials, highly porous foams elaborated by mechanical foaming and intumescent coatings on stainless steel were elaborated. The intumescence property was ensured with several additives. For some formulations, a scale transfer from cm^3 to m^3 was performed. Standardized tests allowed to follow the thermal evolution at high temperature. Structural and mechanical analyses were also performed. The results have shown that the selected formulations can achieve a temperature resistance up to 1400°C with no loss of mechanical properties. This is due to the formation of crystalline phases such as leucite or wollastonite whatever the scale. The foams have also shown good insulating (61 to 75 $mW.m^{-1}K^{-1}$) and mechanical performances (80 to 190 kPa). In the case of the coatings, the adhesive strength is about 3 MPa and a good fire resistance was obtained. To sum up, geopolymers with different raw materials and different porosity and thickness have shown great potential to be fire resistant materials.

9:30 AM

(ICACC-S16-018-2024) Exploring the Influence of Curing Time and Temperature on the Mechanical Properties of K- and Na-based Geopolymers (Invited)

A. C. Trindade*[1]; G. Yik[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, characterized as inorganic, ceramic polymers, blend cement and ceramic traits, and exhibit chemical, mechanical, and rheological adaptability. Controlled geopolymer formulations ($X_2O.Al_2O_{3.}4SiO_{2.}11H_2O$; X = Na/K) have been systematically studied in the past. While diverse advancements have been made using varied designs, the current focus centers on refining the control formulations by decoding time-dependent behaviors. This study evaluates the impact of aging (1-14 days) on reaction rate and strength evolution in K and Na-based geopolymer mixtures, at ambient and 50°C curing conditions. Na-based materials were found to outperform K-based counterparts over time, under both compressive and flexural loads, which may be attributed to a facilitated dissolution of smaller Na atoms. Compressive and flexural strengths reach up to 35 MPa and 7 MPa, respectively. Moreover, elevated H_2O ratios ($13<H_2O<23$) were found to gradually restrain the setting, also inducing microstructural defects, intensified by augmented weight losses at temperature curing conditions. Non-destructive (NDE) measurements were found to be a useful tool to predict setting, unveiling gradual increases in Young's and shear moduli, peaking at 10 GPa at 14 days. This investigation establishes a foundation for future mixture designs, aiming to uncover promising new pathways for this technology.

S2 Advanced Ceramic Coatings for Structural/Environmental & Functional Applications

SYMPOSIUM 2: CMAS-type corrosion and mitigation strategies I

Room: Flagler C
Session Chair: Ravisankar Naraparaju, DLR - German Aerospace Center

10:20 AM

(ICACC-S2-023-2024) Modified Melting and Crystallization Behavior in Mixtures of Calcia-Magnesia-Alumina-Silicate (CMAS) Glass and Sulfates/Seasalt

C. J. Louzon*[1]; R. Yi[1]; N. P. Padture[1]

1. Brown University, School of Engineering, USA

The evolution of gas-turbine engines has been accompanied by increased concerns about high-temperature, particulate-induced degradation in the engines. Mitigation of this degradation is achieved by utilizing protective ceramic thermal and environmental barrier coatings (T/EBCs) throughout the engine hot-section. Significant research has explored the degradation by glassy Calcia-Magnesia-Alumina-Silicate (CMAS) deposits, which form in the engine hot-section. Other studies have examined hot corrosion induced by sulfates formed in the engine. Worsening pollution is changing the atmosphere's chemistry significantly, especially in urban areas. Mixtures of environmental aerosols, including sand, seasalt, and sulfates, can be ingested into gas-turbine engines and form deposits with modified properties. It is likely that these mixed deposits react synergistically, accessing enhanced degradation mechanisms. This presentation will discuss the implications of changes to Calcia-Magnesia-Alumina-Silicate (CMAS) deposits due to the presence of multiple corrodents simultaneously. Recent results from systematic testing of mixed-deposit chemistry, melting, and crystallization will be discussed. Insights from this work can guide future studies on complex, synergistic effects in the degradation mechanisms of mixed-particulate deposits on T/EBCs in gas-turbine engines.

10:40 AM

(ICACC-S2-024-2024) Exploring the Effect of EBC Composition on CMAS Wetting Behavior

C. Luckhardt*[2]; J. L. Stokes[1]; E. J. Opila[2]

1. NASA Glenn Research Center, Environmental Effects and Coatings Branch, USA
2. University of Virginia, Materials Science and Engineering, USA

Rare-earth disilicates (REDS) are used as environmental barrier coatings for the protection of SiC-based ceramic matrix composites in the hot-section of turbine engines. REDS interactions with molten calcium-magnesium-aluminosilicate (CMAS) debris are of interest to understand molten CMAS attack and improve CMAS mitigation strategies. This work assesses the effect of REDS composition and processing on CMAS wetting by investigating wetting angle, spreading, and reactivity using a heating microscope. Substrates included freestanding atmospheric plasma spray (APS) REDS coatings (for RE = Y, La, Nd, Gd, Yb, Lu), dense phase-pure spark plasma sintered (SPS) $Yb_2Si_2O_7$, and a dense SPS two-phase mixture of 20 vol% Yb_2SiO_5 in $Yb_2Si_2O_7$. CMAS (Ca33-Mg9-Al13-Si45 in single cation mol%) was loaded as a 10 mg cylindrical rod atop the specimen surface, polished to 4000 grit, and heated in stagnant lab air to temperatures of 1250°C. The heating microscope measured the evolution of molten CMAS diameter, height, and contact angle as a function of time. Post-exposure CMAS spreading and reactivity were also examined using SEM/EDS and XRD. Wetting behavior varied with RE cation size but was found to be independent of substrate synthesis method.

11:00 AM

(ICACC-S2-025-2024) Modeling the structure of molten and glassy silicates from X-ray and Neutron Diffraction

C. J. Benmore[1]; R. Bogle*[1]; R. Weber[2]; S. K. Wilke[3]; J. Neuefeind[5]; G. Costa[4]

1. Argonne National Lab, X-ray Science Division, USA
2. MDI, USA
3. Materials Development, Inc., USA
4. NASA Glenn Research Center, USA
5. Oak Ridge National Lab, USA

Extreme temperatures can cause chemical reactions between the ceramic coating on a spacecraft and silicate deposits on the surface. To realize the nature of this interaction and improve the durability of the ceramic coating, the atomic structure of liquid and glassy Calcium-Magnesium-Aluminosilicate compositions were studied. The compositions were chosen to be similar to those deposited on turboshaft shrouds operated in a desert environment. High energy x-ray diffraction and neutron diffraction data on the molten and glassy samples were modeled using Empirical Potential Structure Refinement (EPSR). The coordination environment of each of the cations, their bond angle distributions, connectivity, and the average number of bridging oxygens are presented with a view to understanding the reactivity of the silicate melts.

11:20 AM

(ICACC-S2-026-2024) Spatially Resolved Thermal Conductivity Mapping of CMAS Reacted Environmental Barrier Coatings

E. Tiernan*[1]; M. Milich[1]; R. A. Golden[3]; G. Harrington[3]; A. L. Chamberlain[3]; P. E. Hopkins[2]

1. University of Virginia, Mechanical and Aerospace Engineering, USA
2. University of Virginia, USA
3. Rolls-Royce, USA

The extreme environment produced within gas turbine engines can be detrimental to the performance of state-of-the-art silicon carbide (SiC) turbine blades. High temperatures coupled with environmental debris create a corrosive environment which reacts with SiC, and significantly reduces the turbine blade lifetimes. To protect the blades from calcium-magnesium aluminosilicate (CMAS) corrosion, environmental barrier coatings (EBCs) are applied, along with a silicon bond coat. Ytterbium disilicate (YbDS) has become a standard coating material for protecting SiC blades. In these experiments, we use steady-state thermoreflectance (SSTR) to spatially map 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. 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.

11:40 AM

(ICACC-S2-027-2024) Impact of CMAS heterogeneity and sparsity on melt progression

B. Jun*[1]; E. H. Jordan[2]

1. University of Connecticut, Materials Science, USA
2. University of Connecticut, Mechanical Engineering, USA

Calcium-magnesium-aluminosilicates (CMAS) are largely studied in homogeneous bulk. Lab scale studies involving continuous application of CMAS generally involve compositions of liquid nitrate based precursors at specific ratios. Furnace testing typically uses homogeneous CMAS as well. With homogeneous CMAS, melting is expected to start without any of the delays associated with the formation of a lower melting phase by homogenization at the appropriate temperature and, regardless of the sparsity of initial phases that may not be contacting each other. We will discuss the delay of melting caused by the homogenization of heterogeneous CMAS, especially when CMAS deposition on a TBC is sparse. The individual components of AFRL03, and all possible combinations, are compiled in a monte carlo simulation that models the melt progression of sparsely deposited heterogeneous CMAS. Comparisons to liquid precursor homogeneous CMAS and solid powder homogeneous CMAS are made with a focus on melting behavior.

S15 8th International Symposium on Additive Manufacturing and 3-D Printing Technologies

SYMPOSIUM 15: Fused Filament Fabrication and Direct Ink Writing II

Room: Coquina H
Session Chair: Matthew Jones, Lightforce Ortho

10:20 AM

(ICACC-S15-034-2024) Fabrication of multi-material and multifunctional structures by extrusion additive manufacturing

L. Biasetto*[1]; V. Gastaldi[1]; G. Franchin[1]

1. University of Padova, Industrial Engineering, Italy

The use of 3D printing for multi-material structures is a complex endeavor with both challenges and opportunities. Combining materials like metallic alloys and ceramics adds intricacies to managing processing parameters, from initial printing to post-processing stages. However, this challenge also brings numerous possibilities, as blending different metallics and ceramics can create components with both structural and functional attributes. Our study aims to provide a comprehensive exploration of extrusion-based additive manufacturing, focusing on core-shell structure production. We will investigate diverse material combinations and their intrinsic properties. Additionally, we'll conduct an in-depth analysis of critical factors, such as nozzle optimization, ink formulation, precise printing parameter adjustments, and essential processes like de-binding and sintering. These factors significantly impact the properties of the printed structures. We will also showcase practical examples to illustrate the applications of printed core-shell structures. This will highlight the real-world implications and innovative potential of this technology in advanced manufacturing.

10:40 AM

(ICACC-S15-035-2024) Investigation of Carbon-Reinforced Acrylonitrile Butadiene Styrene 3D-Printed Honeycomb Composites

M. Ranaiefar*[1]; M. Singh[2]; J. Salem[3]; M. C. Halbig[1]

1. NASA Glenn Research Center, USA
2. Ohio Aerospace Institute, USA
3. NASA Glenn Research Center, Materials and Structures, USA

The expansive utility of polymeric 3D printing technologies and demand for high-performance lightweight structures has prompted the emergence of various carbon-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, acrylonitrile butadiene styrene (ABS) and carbon reinforced ABS variants, both carbon nanotubes (CNT) and 5 wt.% chopped carbon fiber (CF), are fabricated in a honeycomb geometry and investigated across a range of layer thicknesses and hex sizes. Microscopy of material cross-sections is conducted to evaluate the relationship between print parameters and porosity. Additionally, mechanical properties are evaluated through compression testing, demonstrating the potential of honeycomb ABS, ABS-CNT, and ABS-5wt.% CF polymer composites for novel 3D printed structures.

11:00 AM

(ICACC-S15-036-2024) A Comparative Analysis of Ceramic Additive Manufacturing: Fused Filament Fabrication vs. Vat Photopolymerization

I. Camargo*[2]; J. Verza[1]; C. A. Fortulan[3]; A. Luz[4]

1. UFSCar, Brazil
2. IFSP, Brazil
3. USP, Brazil
4. Federal University of São Carlos, Materials Engineering Department, Brazil

Vat photopolymerization (VP) and Fused Filament Fabrication (FFF) are prominent additive manufacturing (AM) technologies due to their wide availability of low-cost 3D printers, although these machines are primarily employed to manufacture polymeric parts. In the field of ceramic manufacturing, AM offers advantages such as greater design flexibility, cost-effectiveness, and reduced lead times for small-batch production. In this study, advanced ceramic parts were manufactured by VP and FFF to compare not only their ability to print different geometries but also the mechanical strength and surface quality of their sintered parts. Both processes were capable of fabricating parts with flexural strength comparable to those obtained by conventional processes. On one hand, VP distinguished by its superior surface finish, excelled in producing tiny complex features. On the other hand, FFF created parts with larger cross-sectional areas (>1000 mm2), which would be a challenge for VP due to inherent periodic detachment forces associated with the technology. These findings underscore the promise of both AM technologies in the ceramic manufacturing sector, each potentially carving out a unique niche based on their respective strengths and capabilities.

11:20 AM

(ICACC-S15-037-2024) Robocasting of Glass-Ceramic Sealant: Rheological Characterization and Optimization of a Water-based Ink by Design of Experiment

A. Baggio*[1]; F. D'Isanto[1]; D. Dalmazzo[2]; E. Santagata[4]; D. Basso[3]; D. Gaia[3]; M. Salvo[1]; F. Smeacetto[1]

1. Politecnico di Torino, Department of Applied Science and Technology, Italy
2. Politecnico di Torino, Department of Environmental, Land and Infrastructure Engineering, Italy
3. FZSoNick, Switzerland
4. Qatar University, Civil & Environmental Engineering Department, Qatar

In the framework of the SOLSTICE project (European Union's Horizon 2020, grant agreement No 963599), a novel glass-ceramic sealing material has been introduce to replace the preceding formulation, with the aim of reducing the costs associated with the joining process of a solid-electrolyte Na-Zn battery. As expected, altering the composition of the solid load has led to changes in the rheology of the paste, consequently affecting the printability of the new ink through robocasting deposition. Hence, it became necessary to readjust the ink recipe to closely replicate the rheological characteristic of the former paste. In this work, the effect of each component on the ink rheology has been investigated and optimized using the Design of Experiment (DoE), to be applied to Direct Ink Deposition. By means of a rheometer equipped with a coaxial cylinder, several recipes have been tested and one experimental-based model has been formulated for each measured parameter which was taken into account. All validated models have allowed to valuate the influence of each paste component on that specific rheological response associated with that particular model. Finally, all the collected information was used to tune properly the ink recipe, resulting in the successful replication of the rheological behavior exhibited by the previous formulation.

11:40 AM

(ICACC-S15-038-2024) Assessing Printability of Ceramic Suspensions for Direct Ink Writing by Rheology

L. O. Grant*[1]; R. Tao[1]; S. Romberg[1]; R. Maier[1]

1. National Institute of Standards and Technology, USA

Advancing Direct Ink Write (DIW) for ceramic applications necessitates robust feedstock metrology and accurate simulations. Rheological properties, including viscosity, yield stress, and viscoelastic moduli, are closely associated with DIW ink printability. In this work we examine the influence of powder morphology on rheological properties of aqueous inks with varying solids loading of ZnO powders (40-50 vol %), and we share our best practices when addressing the challenges of thixotropy and drying during measurements. Two ZnO powders with distinct morphologies are investigated: the first is a commercially available powder with a jagged, irregular morphology, and the second is an internally-produced powder with a well-controlled size distribution and spherical shape. Measurements of rheological properties relevant for printing are conducted using a rotational rheometer and a capillary rheometer. The comparative analysis of the two ZnO powder suspensions across both rheological apparatuses provides valuable insights that have the potential to improve feedstock metrology standards, increase accuracy when simulating DIW processes, and provide guidance in establishing printability metrics.

S6 Advanced Materials and Technologies for Rechargeable Energy Storage

SYMPOSIUM 6: Advanced anode and cathode materials for lithium batteries

Room: Ballroom 5
Session Chairs: Maximilian Fichtner, Helmholtz-Institute Ulm (HIU); Jason Croy, Argonne National Laboratory

10:20 AM

(ICACC-S6-028-2024) Nanocomposite Engineering of Heterogeneous Mn-based Cation-Disordered Cathodes for High-Performance and Low-Cost Lithium-ion Batteries (Invited)

D. Seo*[1]; E. Lee[2]; H. Lee[2]; J. Lee[3]

1. Korea Advanced Institute of Science and Engineering (KAIST), Republic of Korea
2. Ulsan National Institute of Science and Technology, Republic of Korea
3. McGill University, Canada

High-capacity cathodes for Li-ion batteries without Co and Ni are critical for lower $/kWh. Mn-rich cathode compounds are cost-effective alternatives. Partially cation-disordered cathodes (Sp-DRX) are promising due to their high energy densities and fast rate capability. However, they have a poor cycle life, requiring an understanding of the structure-property relationship. This study shows these cathodes have a multi-phase heterogeneous nature and presents a nanocomposite engineering strategy to modify the phase heterogeneity and enhance cycling stability. These findings expand cation-disordered cathode materials and offer insights into developing low-cost Li-ion cathodes.

10:50 AM

(ICACC-S6-029-2024) Reversible Electrochemical Lithium Cycling in a Vanadium (IV)-and Niobium (V)-Based Wadsley–Roth Phase (Invited)

H. Ji*[1]; E. Lawrence[1]; M. Davenport[2]; R. Devi[3]; Z. Cai[4]; M. Avdeev[5]; J. Belnap[1]; J. Liu[6]; H. Alnaser[1]; A. Ho[7]; T. D. Sparks[1]; G. Gautam[3]; J. Allred[2]

1. University of Utah, Materials Science and Engineering, USA
2. University of Alabama, Chemistry, USA
3. Indian Institute of Science, Department of Materials Engineering, India
4. University of California Berkeley, Department of Materials Science and Engineering, USA
5. Australian Nuclear Science and Technology Organisation, Bragg Institute, Australia
6. Oak Ridge National Lab, USA
7. Carnegie Mellon University, USA

Fast charging remains one of the greatest safety challenges in Li-ion batteries due to Li-dendrite growth occurring on graphite anodes if they are lithiated too quickly. The search for high-rate anodes has highlighted materials in the Wadsley–Roth (WR) shear phase family. The relative abundance of V compared with traditional WR compositions of Nb and W makes V-based phases attractive. However, the high voltage and poor reversibility typically associated with V redox have made V-rich WR phases less studied than Nb- and W-rich phases. Here, we show that a new V-rich Wadsley–Roth phase, $V_7Nb_6O_{29}$, achieves excellent rate capability and 80% capacity retention after 228 cycles with a relatively low average voltage of 1.76 V vs Li/Li^+ compared with other V-rich WR phases. Single-crystal X-ray diffraction reveals a P4/m space group with repeating $2 \times 2 \times\infty$ and $3 \times 3 \times\infty$ blocks of V^{4+} and Nb^{5+} octahedra. Combined neutron pair distribution function analysis, X-ray absorption spectroscopy, and density functional theory calculations show that V redox is the primary source of capacity and that cycling stability is provided by the stable octahedral coordination adopted by V^{4+} in the material.

11:20 AM

(ICACC-S6-030-2024) Activation and Stabilization of Solid-State Oxygen Redox in Antifluorite-Type Li_5FeO_4

H. Kobayashi*[1]; Y. Nakamura[3]; Y. Yokoyama[2]; I. Honma[3]; M. Nakayama[2]

1. Hokkaido University, Japan
2. Nagoya Institute of Technology, Dept. Materials Sci. & Eng., Japan
3. Tohoku University, Japan

Antifluorite Li_5FeO_4, which is firstly reported in 1999, has revisited recently as an earth-abundant Fe-based high-capacity cathode, having the theoretical capacity over 300 mAh g^{-1} using both cationic Fe and anionic O redox. Although the recent works actually showed only one electron redox reaction (173 mAh g^{-1}) with Li_5FeO_4 due to an irreversible structure change, very recently, we have demonstrated two electron redox reactions (346 mAh g^{-1}) by applying cation-disorder treatment to Li_5FeO_4, forming a metastable cubic phase. However, the cyclability of the solid-state oxygen redox is still poor, due to simultaneously occurring O_2 gas evolution. In this work, we succeed in suppressing the O_2 gas evolution by earth-abundant p-block element doping into cubic Li_5FeO_4, enhancing both specific energy density and cyclability. The oxygen spices are covalently bonded to the p-block element, raising an oxidation potential of the O_2 gas release.

11:40 AM

(ICACC-S6-031-2024) Electrochemical K intercalation into polyanionic materials tailored by systematic substitution

J. Kim*[1]

1. Stevens Institute of Technology, Chemical Engineering & Materials Science, USA

Effective grid integration of renewable power generation requires stationary energy storage. Batteries made of earth-abundant elements, such as sodium (Na) and potassium (K), have emerged as alterna-tives to lithium (Li)-based systems for grid. Unlike Na-ion batteries, K-ion batteries, especially their cathodes, are yet to be developed, necessitating materials design principles for systematic investiga-tion. $KVPO_4F$ is one of the most energy-dense polyanionic cathodes. Although promising, its electrochemical properties show room for improvement for practical applications. In this talk, I will demon-strate how K-K interactions affects electrochemical properties of $KVPO_4F$ by introducing titanium to substitute vanadium or oxygen to substitute fluorine. 50% Ti-substituted $KVPO_4F$ exhibits good capacity retention and promote K diffusion by expanding K diffusion pathways. Oxygenated $KVPO_4F$ also shows stable capacity retention and good rate capability. I will also discuss a thermodynamic origin of this improved K kinetics. Our results suggest that judicious selec-tion of cation and anion compositions can be an effective approach to engineer K-ion cathode materials with a desirable electrochemical response.

12:00 PM

(ICACC-S6-039-2024) Ceramic all-solid-state batteries based on garnet LLZO – manufacturing and optimization (Invited)

M. Finsterbusch*[1]; M. Mann[1]; M. Rosen[1]; V. Kiyek[1]; C. Schwab[1]; D. Fattakhova-Rohlfing[1]; O. Guillon[1]

1. Forschungszentrum Juelich, IEK-1, Germany

Full cell fabrication of all-solid-state batteries using only a LLZO based electrolyte faces several challenges, both on materials and processing level. While great improvements in energy density and safety are expected, such full cells featuring Li-metal anodes and high- capacity cathodes still need to be demonstrated. Advanced sintering techniques like FAST/SPS can be used to manufacture purely inor-ganic LLZO/LCO based cathodes without secondary phases and capacities of > 4 mAh/cm2 at a fraction of the energy and time necessary in oven-based processes. The comparison to freely sintered cathodes gives detailed insight into the capacity fading mechanisms and enables new recycling strategies, which take place at the component level. Additionally, we were able to demonstrate modified Li-metal anodes on LLZO with high critical current densi-ties of over 5 mA/cm2 and high plating/stripping capacities of over 27 mAh/cm2 for the first time, pushing the material into the indus-trial relevant region. To further improve cell performance, hybrid approaches using polymer-based electrolytes lead to high energy densities and long cycle life, demonstrating the successful combi-nation of the advantages of both materials classes. These examples show that in case of garnet based all-solid-state batteries, great gains can still be expected if proper materials and process design is used.

S11 Advanced Materials and Innovative Processing Ideas for Production Root Technologies

SYMPOSIUM 11: Recycling and reuse processes

Room: Ponce de Leon

Session Chair: Sungwook Mhin, Kyonggi University

10:20 AM

(ICACC-S11-017-2024) Preparation and characterization of Porous Silicate Particles Derived from Waste Rice Husks via Emulsion Approach (Invited)

R. V. Virtudazo*[1]; A. Arcasa[1]; S. Manlupig[1]; J. Cahigao[1]; E. d. Magdaluyo[2]; E. MIrasol[3]; M. Fuji[4]; I. B. Arugay[1]

This study focused on the preparation and characterization of porous silicate particles derived from agricultural waste rice husk and process via emulsion approach. Waste rice husks as a renew-able silica source were collected locally from a rice milling plant in Kolambugan, Lanao del Norte, Philippines, and undergone chem-ical and heat treatments for the extraction of silica. At first, the rice husks were pre-burned using the rice husk carbonizer. Then, rice hull ash was calcined at 700°C after then it was dispersed in sodium hydroxide to dissolve the silicate and produce a sodium sili-cate solution. After that, emulsified and then calcination at 450 °C. Physicochemical characterizations of synthesized porous silicate powder were analysed. XRD analysis confirmed the presence of an amorphous structure, while FTIR analysis showed SiO_2 structure. FE-SEM surface images revealed distinct globular morphology. While N_2 adsorption desorption isotherm showed nano porous characteristic based on BET surface area and BJH pore size distri-bution. The results showed that the bio-silicate from rice husks not only solves disposal problems but also conserves natural resources, and can be an alternative to commercially available silicate powders wherein this could be a good candidate for thermal management material application.

1. Mindanao State University-Iligan Institute of Technology, Department of Materials and Resources Engineering and Technology, Research Center for Advanced Ceramics, Philippines
2. University of the Philippines, Philippines
3. Mariano Marcos State University, Department of Materials Science and Engineering, Philippines
4. Nagoya Institute of Technology, Japan

10:50 AM

(ICACC-S11-018-2024) Nanosecond Pulse powered Electrocoagulation process (NSP-EC) for wastewater treatment: Fundamentals and applications

A. I. Martinez Sanchez*[1]; Y. Takimoto[2]; H. Furuno[2]; T. Nakayama[2]

1. Nagaoka University of Technology, Science of Technology Innovation, Japan
2. Nagaoka University of Technology, Japan

This study investigates the fundamentals and applications of the Nanosecond Pulse powered Electrocoagulation process (NSP-EC) as a wastewater treatment alternative. The present research is divided into two phases. First, an NSP-power source was used to supply current to Aluminum electrodes in a pure water medium to investigate the influence of the pulse width and voltage in the coagulant particle size. Examination of scanning electron microscope (SEM) imagery suggests that a narrower pulse width engenders the miniaturization of in-situ-produced coagulant precursors. The second phase centers on assessing the effects of current density, pulse width and pH on the efficiency of Chemical Oxygen Demand (COD), Total Nitrogen (TN) and Total Phosphorus (TP) removal from artificial swine wastewater through nanosecond pulse powered electrocoagulation (NSP-EC) process. Experiments were conducted with electrodes fabricated using commercial Aluminum, Iron, Carbon, and Copper plates. The results suggest that a narrower pulse width improves the contaminant removal efficiency attributed to the enhanced reactivity of the coagulant precursors due to the miniaturization of particle size.

S13 Development & Applications of Adv Ceramics & Composites for Nuclear Fission/Fusion Energy Sys

SYMPOSIUM 13: Joining and coating technologies for reactor components

Room: Ballroom 4
Session Chair: Takaaki Koyanagi, Oak Ridge National Laboratory

10:20 AM

(ICACC-S13-030-2024) Brazing of SiC/SiC composites for advanced nuclear energy systems (Invited)

V. Chaumat*[1]; O. Gillia[1]; C. Lorrette[2]

1. Univ. Grenoble Alpes, CEA, Liten, DTCH, France
2. Université Paris-Saclay, CEA, Service de Recherche en Matériaux et Procédés Avancés, France

Owing to their remarkable physical and mechanical properties at high temperature and under irradiation, SiC/SiC composites developed at CEA are of great interest for advanced nuclear energy systems. Brazing is a promising technique for the joining of complex structures of SiC/SiC composites to be used in these systems. More than 30 years have been devoted to the brazing of sintered SiC and SiC/SiC composites at CEA, involving both scientific and technological aspects. This research activity is presented with a focus on nuclear fuel cladding for fission reactors. Brazing SiC-based materials presents two main problems, especially for high temperature applications requiring brazes with high melting points. Firstly, the strong reactivity between SiC and these brazes may lead to degradation of SiC and composites during brazing. Secondly, the difference in thermal expansion coefficient between SiC and braze induces high residual stresses that may be detrimental for the integrity of the assemblies. Therefore, CEA developed the BraSiC® process and associated non-reactive brazes (BraSiC® alloys based on silicon and/or silicides) well suited to the brazing of SiC allowing formation of strong SiC/braze interfaces with no interfacial reaction product and good mechanical properties of the joints created. The obtained results concerning ATF fuel cladding have been achieved through a collaborative work between CEA and Framatome.

10:50 AM

(ICACC-S13-031-2024) Interdiffusion barriers for improved stability of Accident Tolerant Coated Cladding in Pressurized Water Reactors (Invited)

M. Cabrioli[2]; M. K. Grosse[3]; F. Di Fonzo*[1]

1. X-nano, Italy
2. Istituto Italiano di Tecnologia, Center for Nano Science and Nano Technologies, Italy
3. Karlsruhe Institute of Technology, Institute for Applied Materials, Germany

Chromium-coated zirconium-based alloys are on the validation track for industrial application in light water reactors, specifically Pressurized Water Reactors (PWR), as best candidate for a cost-effective near-term evolutionary option for coated Accident Tolerant Fuel (ATF) claddings. Despite their excellent performance during normal operation and the improvements granted under Loss Of Coolant Accident (LOCA) conditions compared to uncoated cladding, interdiffusion at coating/cladding interface leads to fast consumption of the coated system by combined melting and oxidation/reduction processes. Here we present a novel barrier layer capable of completely preventing interdiffusion between Cr and Zr both during normal operation and under LOCA conditions. Transient experiments demonstrated that the failure of coated-cladding under oxidation by steam at high temperatures (in the range 1200 – 1600 °C) is shifted significantly to higher temperatures with respect to the benchmark Chromium-coated zirconium alloy. On these bases, the interdiffusion barrier presented in this work represents a promising, viable option for improved stability of coated ATF cladding from normal operation, through LOCA and towards beyond design basis accident conditions.

11:20 AM

(ICACC-S13-032-2024) Laser assisted joining of SiC/SiC for nuclear applications

M. Ferraris*[1]; M. De Maddis[2]; D. Basile[2]

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

Several technologies and materials have been proposed for joining of SiC/SiC components for nuclear reactors. Among them, laser assisted joining seems to give promising results as a pressure-less, localized heating joining technology suitable for SiC/SiC operating in a nuclear (and other) environment. The present work reports on preliminary results on laser assisted joining of SiC/SiC; several joining materials have been used to join SiC/SiC by a diode laser (LDF 4000-40, Laserline GmbH) operating in the infrared (1020-1060 nm). Morphology and micro-structure of joined SiC/SiC tubes will be discussed. Acknowledgements Part of the research leading to these results has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 101059511 - Project SCORPION (SiC Composite Claddings: LWR Performance Optimisation for Nominal and Accident Conditions)

11:40 AM

(ICACC-S13-033-2024) High temperature ductile amorphous oxide coatings on structural steels: A key technology for next generation fission and fusion power plants

A. Stinchelli[1]; G. Leonardis[1]; M. Beghi[2]; F. Di Fonzo*[1]

1. X-nano, Italy
2. Politecnico di Milano, Department of Energy, Italy

We present our recent results on high temperature ductile amorphous coatings (DACs) and their key enabling role for heavy liquid metal fast reactors and fusion power plants. The prototypical DAC is amorphous alumina (a-Al2O3) fabricated by Pulsed Laser Deposition (PLD). This material exhibits a unique elastoplastic response with a yield stress as high as 4 GPa (tensile and compressive) and a plastic deformation as high as 7% in tension and 100% in compression. SEM in-situ micromechanical tests confirm this elasto-plastic behaviour even for volumes of several hundred microns cube. Besides, a-Al2O3 is stable untill 700 °C, with near zero permeability to gases (H2 isotopes in particular) and inertness with respect to heavy liquid metals. Heavy ion irradiation demonstrated integrity up to 450 dpa. In order to increase reliability and maximum operational temperature, we developed a new formulation stable up to 950 °C. Given the remarkable characteristics and performance of these coatings, especially when applied to structural steels, they emerge as key enablers for the advancement of next generation heavy liquid metal cooled nuclear test reactors and fusion power plants. Our research aims to drive progress in these fields, ensuring the safety, reliability, and efficiency of sustainable future nuclear technologies.

S16 Geopolymers Inorganic Polymers and Sustainable Construction Materials

SYMPOSIUM 16: Sustainable materials and novel applications

Room: Coquina C
Session Chair: Cristina Leonelli, University of Modena and Reggio Emilia

10:20 AM

(ICACC-S16-019-2024) Heavy metal removal properties of porous geopolymers by reactive metal foaming

A. Ozer*[1]; A. Qadeer[1]; W. M. Kriven[2]

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

Geopolymers encompass a diverse range of materials with numerous potential applications because of their exceptional characteristics such as, compressive strength, adsorbance, fire and heat resistance, waste encapsulation, construction materials, restoration and conservation, etc. The chemical precipitation and adsorption are the most used ones, as geopolymers can be used due to the ease of operation, higher removal efficiency, cost-effectiveness and adsorbent disposal. In this work, a novel family of arsenic adsorbent that is extremely effective and relatively economical, which uses porous sodium-based geopolymers doped with graphene oxide (GO) nanoparticles where the composite is a robust mechanical support for targeted metal sequestration. Highly porous supporting material, such as geopolymer doped with graphene oxide nanoparticles is proposed for metal (hydr)oxide nanoparticle impregnation in order to alleviate these issues. However, no research has been done conclusively in order to show that the base media porosity and pore size distribution have a significant impact on the metal (hydr)oxide sequestration. The characterization of geopolymers, powders to be added such as GO and Si was done by XRD and SEM, for phase and morphological structure, respectively. The adsorption efficiency was evaluated by XRF and ICP studies, as well as adsorption coefficient measurements to understand the model.

10:40 AM

(ICACC-S16-020-2024) Controlling the phase development in the alkali activation of waste glass

G. Tameni*[1]; D. C. Lago[2]; J. Kraxner[2]; E. Bernardo[1]

1. University of Padova, Industrial Engineering, Italy
2. Centre for Functional and Surface Functionalized Glass, Italy

Alkali activation can be considered as a reliable strategy to reuse a vaste range of inorganic waste materials which are normally landfilled. Whereas the process is quite simple, a clear understanding of the molecular mechanisms of hardening, especially in the case of waste glass, is challenging. In fact, there is no intensive dissolution and poly-condensation of silicate, aluminate, and alumino-silicate species; strong bonds, in any case, are established according to condensation reactions occurring at thin, hydrated surface layers. Condensation bridges are created along with secondary phases, the latter involving reactions of dissolution products, just between themselves or with the support of the atmosphere. These secondary phases may result soluble or not. This hypothesis is supported by NMR and ICP-OES analysis on cold consolidated and boiled samples. Specific attention is paid to boro-alumino-silicate glass that is mild activated (2.5M) just using metallic hydroxides, such as LiOH, NaOH or CsOH, alone or combined with alternative basic compounds, such as ammonium hydroxide (NH_4OH), in different proportions. The structures of the secondary phases are studied as a function of the dimension of cations and the anion affinity to them.

11:00 AM

(ICACC-S16-021-2024) Cold Sintering of Geopolymers: A Novel Approach for Dense Aluminosilicates (Invited)

L. Lattanzi*[1]; A. Conte[1]; P. Colombo[1]; A. X. Sin[2]

1. University of Padova, Industrial Engineering, Italy
2. ITT Italia s.r.l., Italy

Geopolymers are an emerging class of aluminosilicate compounds which are inorganic, amorphous and and possess a continuous 3D structure. Their network is based on connected SiO_4 and AlO_4 tetrahedra sharing interstitial oxygens, formed by alkali activation of aluminium-silicate sources (metakaolin, fly ash). To achieve a stable structure, positive ions (Na^+, K^+) balance the negative charge of AlO_4^-. Geopolymers exhibit different attractive properties, e.g., high temperature resistance, strong interfacial bonding ability, good corrosion resistance. In this work, we successfully produced geopolymer dense bodies using Cold Sintering. Cold Sintering is a recent low energy sintering technique where powders are densified with the aid of a liquid phase (a water solution, in general) under a (high) external pressure and limited heating conditions (below 350°C). In this scenario, several applications could benefit by this approach (Cold Sintering of geopolymers), such toxic waste encapsulation, matrices for composites, etc.

11:30 AM

(ICACC-S16-022-2024) Understanding the molecular mechanism of cold consolidation of glass by 'weak' alkali activation (Invited)

G. Tameni[1]; D. C. Lago[3]; L. Daniel[2]; S. Ashbrook[2]; D. Galusek[4]; E. Bernardo*[1]

1. University of Padova, Department of Industrial Engineering, Italy
2. University of St Andrews, School of Chemistry and Centre of Magnetic Resonance, United Kingdom
3. Centre for Functional and Surface Functionalized Glass, Italy
4. IIC SAS, Joint Glass centre, Slovakia

Alkali activation is interesting for the development of sustainable construction materials, especially starting from reactive waste-derived alumino-silicate feedstock. A challenge concerns the amount of activators, in the form of concentrated aqueous solutions of alkali hydroxides, silicates and aluminates. Strong and chemically durable components derive from extensive dissolution of starting materials, followed by condensation reactions, realizing highly interconnected, 'zeolite-like' gels. This contribution aims at disclosing unprecedented opportunities arising from alkali activation of glass. Fine powders (<100 μm), in very high amounts, can be collected as industrial waste. Suspended in alkaline solution of limited molarity (e.g. ≤3M NaOH), glass undergoes partial dissolution, followed by gelation, upon drying at 40-80 °C. This gelation is attributable to condensation reactions occurring at surface hydrated layers, leading to strong bonds (Si-O-Si, Al-O-Si etc.). Silicates, borates and aluminates, from glass dissolution, may combine with alkaline ions, in new phases. These extra phases may be leached out, upon immersion in boiling water or acidic attack; the latter attacks, however, do not compromise the integrity of the bonds between particles. The process may be applied to te definition of a new range of porous glass components, applicable even beyond constructions.

S19 Molecular-level Processing and Chemical Engineering of Functional Materials

SYMPOSIUM 19: Energy-Related Matters II

Room: Ballroom 3
Session Chair: Thomas Konegger, TU Wien

10:20 AM

(ICACC-S19-024-2024) Waste to Product: Green production processes for a circular economy (Invited)

A. Weidenkaff*[1]

1. Fraunhofer IWKS, Germany

The transformation to a future green circular economy will be based on green materials and ecologic innovations. The development of renewable materials for renewable energy converters requires sustainable large-scale production from secondary raw materials. The decision making for future resilient energy systems has to be based on environmental aspects as well as on performance criteria defined by a holistic life cycle assessment. In this lecture, an efficient circularity of materials with a programmable lifetime and regeneration will be introduced as a suitable approach. The design of circular high-performance materials uses theoretical predictions and the criticality analysis of applied elements to improve the cycle life of future energy converters such as thermoelectric generators and refrigerators, batteries, electrolysers, fuel cells, plasmalysers, hydrides and solar water splitting cells.

10:50 AM

(ICACC-S19-025-2024) In situ formation of non noble transition metal nanoparticles in a micro-/mesoporous Si-C-N-O(H) ceramic support for water oxidation

M. Ben Miled*[1]; S. Celerier[2]; A. Habrioux[2]; O. Masson[1]; S. Bernard[3]

1. IRCER - CNRS UMR 7315, France
2. Institut de Chimie des Milieux et Matériaux de Poitiers IC2MP UMR7285, France
3. CNRS, IRCER, France

Hydrogen is considered as a promising energy carrier to assure the needs of humanity. Its combustion in a fuel cell (FC) emits only water and does not involve any noise pollution. The use of H_2 in a fuel cell requires it to be obtained at a very high level of purity, which can be easily achieved by water electrolysis. The PDC (Polymer Derived Ceramics) route is a method of developing ceramics that uses pre-ceramic polymers as precursors. The coordination of these precursors with metal complexes makes it possible to grow metal nanoparticles. The nanocomposites produced are expected to : (i) display long-term stability stability due to stronger nanoparticle-matrix interaction and high corrosion resistance ; (ii) expose more active sites; (iii) avoid active sites aggregation during electrochemical process leading thereby to a constant catalytic activity and ; (iv) have practical applicability. This oral communication discusses our recent works on the in situ growth of non-noble transition metal nanoparticles in an amorphous Si-C-N-O(H) ceramic network at very low temperatures (300-700 °C). The large specific surface area of the materials associated with the nanometric size (20-50 nm) of the particles made it possible to boost OER performances, which suggests very promising prospects for the development of a nion exchange membrane electrolysers.

S8 18th Intl Symp on APMT for Structural & Multifunctional Materials & Systems

SYMPOSIUM 8: Advanced composite manufacturing technologies, hybrid processes II

Room: Coquina F
Session Chair: Ivar Reimanis, Colorado School of Mines

10:20 AM

(ICACC-S8-030-2024) Chemically vapor deposited YAG-alumina eutectic system with ordered structures (Invited)

A. Ito*[1]

1. Yokohama National University, Graduate School of Environment and Information Sciences, Japan

Directionally solidified eutectics or melt-growth composites are known as bulk composites in the eutectic systems, and they have been investigated as high-temperature structural ceramics since the early reports on $Y_3Al_5O_{12}$–Al_2O_3 (YAG–alumina) and $GdAlO_3$–Al_2O_3 (GAP–alumina) bulk composites with their excellent high-temperature strength and durability. Although these bulk composites exhibited outstanding mechanical strength and heat resistance, their application as a ceramic coating is limited because a melt-solidification process was believed to be the only way to form ordered structures in eutectic systems. Attempts to melt the surface of ceramic components or apply a melt onto base components have not been successful. Chemical vapor deposition (CVD) is a versatile method of crystal growth from the vapor phase; however, there is no report on the formation of ordered structures in eutectic ceramic systems via conventional CVD process. We demonstrated a chemically vapor deposited eutectic system (CDE) of YAG–alumina composite films with ordered structures, where YAG phase with rod and lamella structures grew unidirectionally in α-alumina phase. Here, we report the effects of the Y/Al molar fractions in the precursor vapor on the formation of ordered structure on various sapphire substrates for the YAG–alumina CDE films.

10:50 AM

(ICACC-S8-031-2024) New perspective on the texture evolution mechanism of Si_3N_4 ceramics:Effect of additive content

Y. Shi*[1]

1. Wuhan University of Technology, China

The effect of additive content on the texture evolution of Si_3N_4 ceramics prepared by hot-pressing at 1750 °C was investigated. When the amount of additive was insufficient, the liquid converted some of the α-Si_3N_4 powder into a few elongated β-grains and numerous isometric β-grains during heating. Afterwards, the deficient residual liquid caused a few isometric β grains to be randomly elongated and coarsened, resulting in a strong texture degree of 0.126 f_L during holding. As the additive content was moderately increased, the texture degree decreased to 0.108 owing to the formation of more randomly elongated grains, which significantly weakened the texture. Moreover, when the additive was abundant, the superfluous liquid phase facilitated the elongation and coarsening of all β grains, resulting in a strong texture of 0.213 f_L. The IOP and MRD values demonstrated a similar tendency for the texture degree in Si_3N_4 ceramics. Therefore, the texture evolution mechanism provides a new understanding of more accurately regulating the texture microstructure in Si_3N_4 ceramics.

11:10 AM

(ICACC-S8-032-2024) Microstructures and mechanical properties of B_4C–SiC–rGO composites prepared using spark plasma sintering

L. Hu[1]; Q. He*[3]; W. Wang[2]

1. Wuhan Business University, China
2. Wuhan University of Technology, China
3. Wuhan University of Technology, The State Key Laboratory of Advanced Technology for Materials Synthesis and Processing, China

In this study, B_4C–SiC–rGO composites with different SiC contents were prepared by self-assembly polymerization process and spark plasma sintering at 1800 °C for 5 min under a uniaxial pressure of 50 MPa. The microstructure evolution with SiC addition of the B_4C–SiC–rGO composites were investigated. The optimal values for flexural strength (545.25 ± 23 MPa) and fracture toughness (5.72 ± 0.13 MPa*$m^{1/2}$) were obtained simultaneously when 15 wt.% SiC was added to 5 wt.%–GO reinforced B_4C composites (BS15G5). The introduction of the SiC phase and rGO into the B_4C ceramics produced high-density interfaces, and these interface structures were the key factors enhancing the mechanical properties of the composites. The twin structures of SiC and B_4C observed in the composites improved their fracture toughness. Crack deflection and crack bridging caused by the SiC grains as well as rGO bridging and rGO pull-out were observed on the crack propagation path.

FS1 Bioinspiration/Green Processing & Related Technologies of Advanced Materials

Focused Session 1: Bioinspiration, Green Processing, and Related Technologies of Advanced Materials

Room: Ponce de Leon
Session Chair: Zhaoyong Zou, Wuhan University of Technology

1:30 PM

(ICACC-FS-001-2024) Convergent Biological Designs for Advanced Materials (Invited)

D. Kisailus*[1]

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

For millions of years, organisms have derived specific sets of traits in response to selection pressures that serve as guideposts for optimal biological designs. A prime example is the evolution of toughened structures in disparate lineages within plants, invertebrates, and vertebrates. Extremely tough structures can function much like armor, battering rams, or reinforcements that enhance the ability of organisms to win competitions, find mates, acquire food, escape predation, etc. Natural systems have developed well-orchestrated strategies 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 frequently superior to mechanical properties exhibited by engineering materials. Some comparative analyses of multiscale structures have pinpointed which design principles have arisen convergently, suggesting an optimized solution for separate evolutionary paths. This is required for survival under extreme conditions. In this work, we describe a few of these systems that show convergent design and describe how controlled syntheses and hierarchical assembly using organic scaffolds lead to these integrated macroscale structures. We describe their function and its translation to biomimetic materials used for engineering applications.

2:10 PM

(ICACC-FS-003-2024) Bioinspired synthesis of crystalline nanomaterials through peptoid-based approaches (Invited)

C. Chen*[1]

1. Pacific Northwest National Lab, USA

In nature, biominerals (e.g. bones) are examples of hierarchical composite materials whose formation and functions are controlled over multiple length scales by high information content biomacromolecules. Inspired by nature, many biomimetic approaches have been developed for the design and synthesis of crystalline nanomaterials. These approaches are attractive because they generate complex, functional materials under mild aqueous synthetic conditions. Despite the advances in developing new approaches for bioinspired materials synthesis, the rules of designing macromolecules that lead to the predictable synthesis of crystalline nanomaterials remain unclear. As one of the most advanced peptidomimetics, peptoids offer tremendous opportunities for bioinspired materials synthesis. In this presentation, two peptoid-based approaches will be discussed for controlled synthesis of crystalline nanomaterials. The first involves the design and synthesis of surfactant-like peptoids for controlling formation and morphogenesis of inorganic nanomaterials. The second approach exploits self-assembling peptoids for controlled synthesis of hierarchical composite nanomaterials. A combination of in situ imaging (e.g. in situ TEM) and molecular simulations are used to elucidate the important roles of peptoids and particle attachments during the peptoid-directed formation of crystalline nanomaterials.

FS3 Nanostructures and Low-Dimensional Materials for Chemical Sensors

Focused Session 3: Nanostructures and Low-Dimensional Materials for Chemical Sensors

Room: Flagler A
Session Chairs: Ho Won Jang, Seoul National University;
Ji-Wook Yoon, Jeonbuk National University

1:30 PM

(ICACC-FS3-001-2024) Real-time taste detection using rationally designed graphene channels for electronic tongue (Invited)

H. Jang*[1]

1. Seoul National University, Republic of Korea

The detection of ions and molecules in liquids has been receiving considerable attention for the realization of the electronic tongue. Solution-gated field-effect transistors (SFETs) with high sensitivity are useful for detecting ions and molecules by reading electrical transconductance. However, to date, ionic and molecular sensors that employ SFETs have limitations, such as the lack of a dynamic on–off function and low selectivity. We develop rationally designed graphene SFETs as taste sensors. The integration of the microfluidic channel to the graphene SFET exhibits dynamic on–off functions by controlling injection and withdrawal of solutions. The graphene SFET device exhibits high selectivity toward sourness, sweetness, and saltness when coated with Nafion as a molecular sieve and appropriate receptors. The dynamic on–off functions and high selectivity of SFETs with tailored graphene channels have a high potential for advancing as a platform for electronic tongues by integrating the separate SFETs as an array for simultaneous sensing of multiple targets.

2:00 PM

(ICACC-FS3-002-2024) Surface-Modification is Crucial in Resistive Type Gas Sensing (Invited)

H. Kim*[1]

1. Hanyang Univ, Republic of Korea

In the IOT era with trillion sensors, there have been a variety of gas sensors, being classified into ~20 kinds. Resistive type gas sensors have been intensively explored, however, several characteristics need to be attained: high selectivity to target gas, stability, humidity-resistance, and temperature-independence, etc. We found that the manipulation of sensor surfaces is a crucial factor for enhancing the sensing performances. First, a variety of beam-irradiation techniques, in terms of electron beam, helium ion-beam, xenon ion beam, proton beam, laser beam has been examined to investigate the beam-induced change of sensor surface and the accompanying enhancement of sensing capabilities. In addition to microstructural defects, the rough surfaces have been regarded as improving the gas sensing. Impurity doping on the surface will change the surface properties. Metal catalysts located on the sensor surfaces will indirectly enhance the selectivity, by modulating the chemical bonding. Metal catalysts will be installed in a more sophisticated manner, on various materials, including MOFs. Some functional materials, such as RGO, will be installed on the sensor surface to enhance the sensing by modulating the carrier concentration, in regard to the formation of heterostructures. Some layers, such as an amorphous carbon layer, indirectly controlled the sensing behaviors of the underlying sensor surfaces.

2:30 PM

(ICACC-FS3-003-2024) Heterogeneous Integration of Atomically Thin Wafer-Scale 2D Materials for Sensing Device Applications (Invited)

Y. Jung*[1]

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

In this talk, I will discuss recent efforts in my group on exploring viable manufacturing strategies to assemble wafer-scale 2D transition metal dichalcogenide (TMD) layers of heterogeneously tailored components on arbitrary substrates for sensing device applications. Specifically, we grew multi-component 2D TMD layers of controlled orientation – i.e. horizontal or vertical layer alignments – on a large wafer scale (> cm^2) via a modified chemical vapor deposition process. We, then, peeled off the as-grown wafer-scale 2D TMD layers from their original growth wafers within water preserving their intrinsic structural/chemical integrity, and subsequently integrated them onto substrates of virtually unrestricted kinds and shapes in a heterogenous manner. The underlying principle for this water-assisted 2D layer delamination will be discussed in the framework of thermodynamic interfacial energy and water-driven capillary force mechanisms. A range of novel sensing device applications enabled by these atomically-thin wafer-scale materials assembled on unconventional substrates were demonstrated, including multi-dimensionally stretchable toxic gas sensors, humidity-responsive soft actuators as well as bio-degradable transient sensors.

3:20 PM

(ICACC-FS3-004-2024) Design of ferroelectric ε-WO_3 for selective detection of acetone (Invited)

J. Yoon*[1]

1. Jeonbuk National University, Republic of Korea

Acetone, a byproduct of lipolysis, is excreted via breath due to its high volatility. Thus, breath acetone analysis using portable sensors could be a non-invasive, always accessible technique to monitor fat burning during exercise/dieting. However, implementing this technology remains challenging due to the lack of sensing materials that can detect acetone selectively. Herein, we suggest ferroelectric ε-WO3 as a novel sensing material for selective detection of acetone. Acetone is a polar gas with a high dipole moment of 2.88 D. Thus, the key idea is to facilitate a strong interaction of polar acetone to the sensing surface using polarized ferroelectric domains. The ε-WO3 was prepared by doping Nb and Ti into γ-WO3 by ultrasonic spray pyrolysis. The acetone selectivity of the ε-WO3 sensors was significantly higher than the values reported in the literature, and the acetone response was also sufficiently high to monitor breath acetone at ppb levels. The correlation between the ferroelectric properties and gas sensing characteristics was investigated by measuring the spontaneous polarization, and the acetone adsorption ability of the ε-WO3 was evaluated using thermal desorption spectroscopy. This work will provide a novel strategy for designing highly selective oxide semiconductors and facilitate the development of a new class of portable breath acetone sensors for monitoring fat burn efficiency.

3:50 PM

(ICACC-FS3-005-2024) Atomic layer deposition to materials for gas sensing applications (Invited)

N. Pinna*[1]

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

Atomic layer deposition is a thin film deposition technique based on self-terminated surface reactions. Contrarily to most of the thin film deposition techniques, it is not a line of sight deposition technique due to the sequential introduction of the gaseous precursors and because the reactants can only react with surface species. The precursors can thus diffuse into porous structures and the conformal coating of high aspect ratio structures can be achieved. Because of these peculiarities atomic layer deposition is an attractive technique for fabricating materials to be applied in resistive gas sensors. The presentation focuses on materials for resistive gas sensor devices in which the sensing material is elaborated using atomic layer deposition, in at least one step of the fabrication. It will be shown that atomic layer deposition has proven to be well-suited for the elaboration of compact thin films, nanostructures and heterostructures to be applied for the detection of a variety of analytes such as toxic compounds, pollutants, explosives, etc. The chemical and physical properties of the sensing layers will be discussed in parallel to the gas sensing mechanisms in an attempt to develop clear structure–property correlations.

4:20 PM

(ICACC-FS3-006-2024) Low Dimensional Ceramic Chemiresistive Gas Sensors for Medical Diagnosis (Invited)

O. K. Varghese*[2]; D. Waligo[2]; B. Kandel[2]; M. Paulose[1]

1. University of Houston, Department of Physics, USA
2. University of Houston, Department of Physics & Texas Center for Superconductivity, USA

Metal oxide semiconductors are renowned for their ability to change electrical resistance significantly as a function of concentration of chemical species in the environment. This property, commonly called chemiresistive behavior, has been utilized for the development of gas sensors for over 60 years. Many of these ceramic materials are inexpensive and offer mechanical and chemical stability compared to non-ceramic semiconductors. Nevertheless, problems such as inability to distinguish a specific analyte from an environment of many chemicals and insufficient sensitivity to very low concentration analytes (parts per million to billion or trillion level) are common problems impairing the commercialization prospects of ceramic sensor technologies. Low dimensional metal oxides are promising in this regard, as they offer ways to address these problems. They possess tremendous surface area and enable the surface states and microstructure to have a strong control over the electrical response. Consequently, the sensors based on nanostructured oxide semiconductors are increasingly abeing applied for the detection of low concentration gases and volatile organic compounds in human breath that carry information about body's health state. This presentation is aimed at discussing the advances in this non-invasive technology focusing on the sensing performance of the novel materials developed recently in our laboratory.

S2 Advanced Ceramic Coatings for Structural/Environmental & Functional Applications

SYMPOSIUM 2: CMAS-type corrosion and mitigation strategies II

Room: Flagler C

Session Chairs: Seongwon Kim, Korea Institute of Ceramic Engineering and Technology (KICET); Gustavo Costa, NASA Glenn Research Center

1:30 PM

(ICACC-S2-028-2024) Development of EB-PVD (Gd,Y) Zirconate solid solution coatings as CMAS resistant TBCs (Invited)

R. Naraparaju*[1]; P. Mechnich[2]; U. Schulz[2]

1. DLR - German Aerospace Center, Materials Research, Germany
2. DLR - German Aerospace Center, Institute of Materials Research, Germany

(Gd, Y) Zirconate solid solution having multiple compositions within the range of 50Y2O3-30Gd2O3-20ZrO2 to 25Y2O3-50Gd2O3-25ZrO2 coatings were deposited employing electron beam physical vapor deposition (EB-PVD) technique using a jumping beam approach on dual ingots of GZO and Y2O3. The obtained coatings have exhibited dual (YSZ fluorite type+ Y2O3 type) phases for Gd2O3 rich and a single phase (yttria type) for ytrria rich solid solution. High-temperature CMAS infiltration tests at 1350°C and 1400°C have shown that yttria-rich solid solution induces very quick reaction kinetics and exhibits the protection behaviour. A reaction layer consisting of apatite and garnet forms a continuous layer at the interface and crystallizes the CMAS residue. The comparison with GZO and 65YZ shows that yttria-rich (Gd, Y)ZO exhibits superior CMAS resistance at high temperatures after longer times.

2:00 PM

(ICACC-S2-029-2024) Reactive crystallization in multiple rare-earth zirconates and a model CMFAS melt

C. S. Holgate*[1]; S. Berens[1]; N. Basilyan[1]; C. G. Levi[1]

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

Improving the efficiency of gas turbine engines requires discovering increasingly durable materials that can withstand higher temperatures. One challenge is the degradation of thermal and environmental barrier coatings by molten silicates. The complex thermochemical interactions involve the dissolution, diffusion, and reaction of at least five cations. Therefore, fully understanding this problem through experimental testing alone is impractical. Computational thermodynamics tools can greatly accelerate this understanding, and subsequently inform coating discovery and design. However, the available databases are incomplete and often incorrectly predict experimentally-observed behavior. This gap motivates systematic experiments to probe the underlying thermodynamics relevant to coating-melt interactions. This work investigates the equilibrium reactive crystallization between three candidate thermal barrier coating materials ($La_2Zr_2O_7$, $Gd_2Zr_2O_7$, and $Y_2Zr_2O_7$) and a model CMFAS melt ($C_{23}M_9F_9A_{19}S_{40}$) at 1300°C. The three coating oxides show distinct crystallization behavior, with different evolutions of apatite, garnet, and polymorphs of reprecipitated zirconia as more of the zirconate dissolves. The experimental results are compared to the predicted reaction behavior from Thermo-Calc's TCOX database. Implications for coating design and rare-earth selection is discussed.

2:20 PM

(ICACC-S2-030-2024) Mitigation of Slag Corrosion for Gasifier and Burner Components in IGCC(Integrated Gasification Combined Cycle) Power Plants

S. Kim*[1]; M. Nam[1]; Y. Oh[1]; S. Lee[1]

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

Gasifiers and burners are indispensable components of IGCC (Integrated Gasification Combined Cycle) power plants, operating under harsh conditions characterized by high temperatures, elevated pressures, and exposure to corrosive environments. Corrosion by slag from raw coal fuel, which is composed of calcia, alumina, and silica, has been a common challenge in these components. Effective strategies are essential to mitigate slag corrosion, focusing on materials selection and functional surface treatments. In this study, corrosion behaviors are identified encountered in gasifiers, burners, and related components during IGCC power plant operation. We explore mitigation approaches, primarily from a materials perspective as well. Special emphasis is placed on the most promising strategies and their detailed components for tackling slag corrosion in gasifier and burner systems.

2:40 PM

(ICACC-S2-031-2024) CMAS Resistance of Xenotime Mineral-inspired Rare Earth Phosphates for Environmental Barrier Coatings

P. Stack*[1]; E. Opila[1]

1. University of Virginia, USA

Recent work demonstrates that multicomponent rare-earth (RE) disilicates offer promise as thermal/environmental barrier coatings (T/EBCs) for SiC-based composites. Reduced thermal conductivity is attributed to increased phonon scattering caused by variations in RE mass and size, enabling T/EBC capability. However, exposure of RE disilicates to molten calcium-magnesium-alumino-silicates (CMAS) results in rapid degradation of the RE disilicate. Additionally, the separation of rare earths, only to recombine them for improved thermal properties, makes this an expensive concept. Naturally occurring xenotime ($REPO_4$) minerals containing mixed $(Y,Gd,Dy,Er,Yb)PO_4$ offer promise as a more economical T/EBC if rare earth separation is not required. Here we present CMAS resistance properties for individual and synthetic mineral xenotime rare earth phosphates synthesized from RE_2O_3 and H_3PO_4 and densified by spark plasma sintering. Thermochemical stability in molten deposits was determined at 1300°C and compared to conventional RE disilicates. Viability of xenotime minerals as a T/EBC is discussed.

3:20 PM

(ICACC-S2-032-2024) Erosion Performance and CMAS Degradation of T/EBC Material Systems (Invited)

D. E. Wolfe[3]; C. DeSalle*[1]; J. A. Reiss[1]; M. Schmitt[2]; P. E. Albert[1]; A. K. Rai[4]

1. Penn State ARL, USA
2. HAMR Industries LLC, USA
3. Pennsylvania State University, USA
4. UES, 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 and SEM/EDS. Coatings are also exposed to sand erosion and correlated to relative mass loss. These results are holistically compared to establish the process-structure-property-performance relationships associated with leading compositions and deposition techniques.

3:50 PM

(ICACC-S2-033-2024) Effectiveness of YFeSi oxide as a CMAS-resistant layer for EBCs

Z. Stein*[1]; J. E. Förster[2]; U. Schulz[2]; R. Naraparaju[2]; S. Raghavan[1]

1. Embry-Riddle Aeronautical University, Aerospace Engineering, USA
2. DLR - German Aerospace Center, Institute of Materials Research, Germany

Aircraft aero-engines operate within demanding extreme environments and conditions. Ingestion and subsequent melting of sand (CMAS) particulates are detrimental to the overall lifetime and effectiveness of high temperature ceramic coated components, such as turbine blades. Enhancing turbine engine efficiencies necessitate more robust material systems. Compositions based on silicon carbides permit higher operating temperatures, but are susceptible to water vapor oxidation. Environmental barrier coatings (EBCs), such as silicates, limit these oxidant species from reaching the composite, but are prone to CMAS-related degradation. A novel YFeSi oxide layer deposited atop Y-based PVD mono- and disilicate bi-layer as well as Yb-based plasma sprayed EBCs was assessed for its effectiveness in hindering CMAS infiltration using SEM, EDX, and high-energy synchrotron XRD data. These EBC systems were exposed to CMAS and annealed at 1300°C for 1h, 1350°C for 1h and 1400°C for 1h. The results highlight YFeSi as an effective CMAS-resistant layer, enhancing the durability of EBCs and facilitates reusability in carbide-based and other novel ultra-high temperature systems.

4:10 PM

(ICACC-S2-034-2024) Molten silicate interactions with $La_2Hf_2O_7$ and two-phase $La_2Hf_2O_7/HfO_2$ environmental barrier coatings

S. Berens*[1]; A. R. Ericks[1]; C. S. Holgate[1]; F. W. Zok[1]; C. G. Levi[1]

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

Hf-based coatings have received interest as candidate topcoat materials in environmental barrier coating systems given their high temperature stability and resistance to H_2O-mediated volatilization in the gas turbine environment. Microcracking driven by anisotropic thermal expansion and attack by molten silicates, especially basic melts, preclude implementation of monoclinic HfO_2. A potential improvement is adding a second reactive phase to (1) refine HfO_2 grain size to prevent microcracking and (2) promote formation of a kinetically-passivating reaction layer. $La_2Hf_2O_7$ pyrochlore is uniquely stable with m-HfO_2 and contains sufficient $REO_{1.5}$ to promote reactive crystallization. This study investigates the silicate interactions with $La_2Hf_2O_7$ and a 30 vol% $La_2Hf_2O_7$/m-HfO_2 composite at 1400°C to assess the formation and stability of reaction layers through equilibrium and coating-silicate reaction couple experiments. While $La_2Hf_2O_7$ does rapidly react to form dense reaction layers, grain boundary penetration dominates after sufficient time, and melt deeply infiltrates the compact. Likewise, grain boundary penetration destabilizes the $La_2Hf_2O_7$ /m-HfO_2 composite. Implications for composite coating design will also be discussed.

4:30 PM

(ICACC-S2-035-2024) CMAS-type corrosion of Yttrium-Aluminum-Garnet (YAG)

P. Mechnich*[1]

1. DLR - German Aerospace Center, Institute of Materials Research, Germany

Yttrium-Aluminum-Garnet (YAG, $Y_3Al_5O_{12}$) is considered an attractive material for thermal and environmental barrier coatings for next generation gas turbine engines. Low-thermal conductivity, relatively high thermal expansion, good steam corrosion resistance, and favorable low diffusivity make YAG a promising coating material for metal components as well as ceramic-matrix composites. A key environmental thermal-chemical load in operation is recession by ingested airborne mineral dust particles, commonly referred to as CMAS corrosion. The interaction of YAG by CMAS has been reported in several studies in the past years, where some ambiguities regarding the chemical stability of YAG against were appearing. The presentation deals with a detailed study of different kinds of YAG materials (powders, plasma-sprayed coatings, single crystals) and their thermal-chemical response on CMAS-type attack, also by means of in situ experiments, and will address possible difficulties during interpretation of results.

S10 Modeling and Design of Ceramics and Composites

SYMPOSIUM 10: Modeling and design of ceramics and composites

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

1:30 PM

(ICACC-S10-001-2024) Compositional and microstructural design of environmental barrier coatings for SiC_f/SiC composite (Invited)

J. Wang*[1]

1. Institute of Metal Research, CAS, Advanced Ceramics and Composites Division, China

Emerging engine hot-section components are focused on SiC_f/SiC composite with environmental barrier coating (EBC) that can withstand harsh thermal and chemical attacks against combustion environment. Typical EBCs provide reliable protections to SiC_f/SiC components below the surface temperature of 1300°C. However, the present EBCs demonstrate limited capability of corrosion resistances to hot steam and molten $CaO-MgO-Al_2O_3-SiO_2$ (CMAS) deposit at higher temperatures, as well as the phase instability and amorphization during coating fabrication. To address these critical challenges, strategic design are adopted to discover new rare-earth silicate EBCs through multilevel optimizations, including both the multi-RE-component (or high entropy) modification and microstructure regulation. The advancement of new multi-RE-component silicate EBCs may support the requirements of future SiC_f/SiC composite engine components.

2:00 PM

(ICACC-S10-002-2024) Approximation and definition of state variables for a comprehensive description of damage for ceramic matrix composites (Invited)

E. Baranger*[1]

1. Université Paris-Saclay, CentraleSupélec, ENS Paris-Saclay, CNRS, LMPS, France

Over the past decades, non-linear models describing damage and fracture of architectured materials have highly progressed, taking into account varied scenarios of degradation under complex multi-axial loadings. Many of these models remain expansive to numericaly handle and difficult to understand. In parallel, full-field measurement has brought a large quantity of data. A challenging task for the researcher is extracting information from that data quantity. The extraction relies on state variables and the associated energetic potentials. This paper will present the automatic definition of state variables in two situations. The first situation corresponds to a local case where the objective is simplifying an existing constitutive relation. As an example, an anisotropic tensorial damage model developed is simplified to a scalar damage model. Several choices of approximations and errors will be discussed. The second situation corresponds to a structural case where the objective is to simplify fields. The developed point of view relies on local pattern extraction associated with a PUM/GFEM method. After the introduction of this method, an automatic building method is evaluated. This project has received funding from the European Research Council (ERC) under the European Union's Horizon 2020 research and innovation programme under grant agreement No. 101002857.

2:30 PM

(ICACC-S10-003-2024) Machine learning accelerated computational design of oxide catalysts using "Center-Environment" features: From spinel to perovskite (Invited)

Y. Liu*[1]

1. Shanghai University, Materials Genome Institute, China

Computational design of materials with targeted properties provides a rational design principle for costly experimental developments. It is, however, a formidable task for first-principles (FP) calculations to explore the numerous hypothetical materials as well as their properties. Thanks to the surge of data science of the fourth research paradigm, machine learning becomes a powerful tool to accelerate the computational materials design at low cost. In this talk, we introduce the development and applications of machine learning models with the attention-focused composition-structure informed "Center-Environment" features using FP computational data (FP-ML/CE). The FP-ML/CE approach was first used to predict the stability of spinel oxides and extend to perovskite oxides by transfer learning, suggesting potential candidates of novel oxides. Furthermore, the catalytical performance of oxygen evolution reactions of perovskite oxides were studied using surface "Center-Environment" features (SCE) by the FP-ML/SCE approach. Taking the advantages of the accuracy of computation and efficiency of ML methods, such combined FP-ML approach becomes a powerful acceleration strategy to explore enormous candidates commonly required in computational materials design.

3:20 PM

(ICACC-S10-004-2024) Machine learning approaches for property predictions of silicon nitride ceramics

R. Furushima*[1]; Y. Nakashima[1]; Y. Zhou[1]; Y. Maruyama[1]; K. Hirao[1]; T. Ohji[1]; M. Fukushima[1]

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

Reducing measurement time for properties of ceramic products greatly contributes to accelerating their research and development. Machine learning approach helps to reduce the measurement time by predicting specific properties from alternative data without direct measurements of them. It is widely recognized that precise property prediction through the machine learning requires a substantial amount of data. Consequently, the application of machine learning has been limited in specific materials, like engineering ceramics, that demand long time for fabrication of a single sample. In this study, we present typical examples where machine learning is employed to precisely predict various properties of silicon nitride. These examples of machine learning approaches are conducted with a relatively small number of samples. These results suggest that precise property predictions can be accomplished through ingenuities attempted, even for ceramic materials where a large amount of data is not obtained.

3:40 PM

(ICACC-S10-005-2024) Development of Deep Neural Network Interatomic Potentials for Advanced Ceramics

K. Ghaffari*[2]; S. Bavdekar[1]; D. Spearot[2]; G. Subhash[2]

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

Due to recent unprecedented innovations in computing power, data-driven methods like Machine Learning (ML) have risen in popularity in the field of solid mechanics. Specifically, 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 further investigations. This work details the development of an NN-based IP for boron carbide (B4C), specifically the training data generation, model selection, and model validation. The breadth of computational and experimental literature available on B4C allows for development and thorough validation of the MLP. Preliminary results indicate a run-time speed increase of nearly two orders of magnitude in NN-based IP shock simulations as compared to traditional ReaxFF-based simulations. This increase in efficiency can radically improve the predictability and accuracy of computational investigations previously unattainable with conventional approach.

4:00 PM

(ICACC-S10-006-2024) Shock Simulations of B4C with Machine Learning and Empirical Force Fields

K. Ghaffari*[2]; S. Bavdekar[1]; D. Spearot[2]; G. Subhash[2]

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

Due to recent unprecedented innovations in computing power, data-driven methods like Machine Learning (ML) have risen in popularity in the field of solid mechanics. Specifically, 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. In particular, ceramics with complex bonding structures require multiple energy contribution terms to accurately capture the chemistry of the system. For example, the unique icosahedral structure of boron carbide (B4C) and its associated reactivity had been best represented empirically by the ReaxFF potential. This force field formulation has 14 energy contribution terms and numerous fitting parameters, making it computationally costly despite its empirical nature. In this work, we develop an ML interatomic potential (MLIP) for B4C, and compare it to the available ReaxFF potential with specific focus on shock compression simulations. Preliminary results indicate increased accuracy and a 8-fold increase in computational efficiency in the MLIP as compared to ReaxFF. Such dramatic speedups can unlock new simulation time- and length-scales previously not possible.

4:20 PM

(ICACC-S10-007-2024) Optimizing Deep learning Training for Scientific Imaging of Fiber-reinforced Composites

A. Badran*[1]; B. Provencher[2]; J. Kroll[3]; M. Marsh[2]

1. General Electric Aerospace Research, USA
2. Comet Technologies Canada, Canada
3. University of Texas Arlington, USA

Deep learning models have revolutionized scientific imaging by enhancing pixel-wise labeling accuracy. However, U-Net and related architectures often face implementation challenges due to insufficient training optimization studies. This topic explores U-Net training using x-ray CT images of fiber reinforced composites, analyzing the impact of training data volume, and data augmentation. We employ seven-fold replications to address training process variability. Variance in training and the complexity of images are explored. Increased training data volume significantly improves model accuracy and speed, reducing variance among replicates. Notably, data augmentation substantially enhances performance, especially with limited ground truth data. High data augmentation coefficients are recommended for scientific imaging semantic segmentation models. Future work on quantifying image complexity will guide minimum required training data volumes for specific tasks in scientific imaging.

S12 Design and Applications of Nanolaminated Ternary Transition Metal Carbides/Nitrides and Borides, Their solid solutions and 2D Counterparts

SYMPOSIUM 12: Design of novel compositions and manufacturing methods

Room: Ballroom 3
Session Chairs: Miladin Radovic, Texas A&M University;
Konstantina Lambrinou, University of Huddersfield

1:30 PM

(ICACC-S12-001-2024) Twelve Years In: How are MXenes Doing? (Invited)

M. Barsoum*[1]

1. Drexel University, Materials Science and Engineering, USA

At this juncture there is no doubt that MXenes have captivated the world's interest and with good reason. The combination of hydrophilicity and metal-like conductivity – a new combination – has proven to be quite useful. The number of potential applications is quite numerous and quite diverse. The use, or addition, of MXenes has been shown to boost some properties sometimes to record levels. In other situations, they allow for new applications for 2D materials like electromagnetic shielding. Their chemical diversity is also a huge attractor. As MXenes get more popular, they are accompanied by hype. In this perspective, I will review the field and try to differentiate between work done in the name of science that has little chance of applicability and other work that may lead to applications. The fundamental, and quite difficult problem to solve, with MXenes is that because we are dealing with essentially a few – 2, 3, 4 - of highly reactive transition metals layers interleaved with C or N, it will be very difficult indeed to render them oxidation resistant. It follows that actual applications where MXenes are placed in open, aqueous-based systems will be few and far between. Hyping such applications - while not simultaneously solving the stability conundrum for times commensurate with the time of use in said applications – is irresponsible and hurts the credibility of the field in general including applications that are possible.

2:00 PM

(ICACC-S12-002-2024) High Quality MXenes for Electrocatalysis (Invited)

K. Liang*[1]

1. Ningbo Institute of Materials Technology and Engineering, China

Electrocatalysis is regarded as the core of future clean energy conversion technologies. In particular, the HER involved in water splitting is the most studied, in which the precious metal-based electrocatalysts remain the most efficient. Thus, exploring earth-abundant element-based electrocatalysts with high efficiency and stability for the involved electrochemical reaction is of great importance. Transition metal carbides/nitrides (MXene), a novel growing family of 2D nanomaterials, demonstrates superiorities such as outstanding electrical conductivity, abundant terminal groups, unique layered-structure, large surface area, and hydrophilicity, making it to be a potential candidate material for electrocatalysis. In this talk, I will provide an overview of the fundamental properties and synthesis routes of pure MXene, functionalized MXene and their hybrids, highlights the state-of-the-art progresses of MXene-based applications with respect to electrocatalysis.

2:30 PM

(ICACC-S12-003-2024) MNenes for Electrochemical Energy Storage and Conversion (Invited)

A. Djire*[1]

1. Texas A&M University, Artie McFerrin Department of Chemical Engineering, USA

The large-scale shift towards renewable energy technology requires the ability to reliably convert and store electrochemical energy. In recent years, two-dimensional (2D) materials have been explored as low-cost, high-performing catalysts and electrodes for these conversion and storage processes. Of these 2D materials, a special class of carbides and nitrides, known as MXenes, have gained popularity due to their high tunability, conductivity, and high surface area-to-volume ratio. From these MXenes, the carbide form has been the most vastly studied, but the nitride MXene (MNene) has been theorized to perform better due to their higher electronic conductivity. In this presentation, I will present on the oxygen-assisted molten salt fluoride etching technique developed in my lab for phase-pure MNenes. Along with this, I will provide evidence of the enhanced capabilities of the MNenes compared to carbides, sulfides, and oxides, when applied in the nitrogen reduction reaction (NRR), hydrogen evolution reaction (HER), carbon dioxide reduction reaction (CO_2RR), and supercapacitor applications. In each of these fields, the enhanced performance is investigated using in-situ/operando spectroelectrochemical techniques. I will wrap up by discussing the future directions in the field of MXenes that require further attention for the advancement of 2D materials in electrochemical energy storage and conversion systems.

3:20 PM

(ICACC-S12-004-2024) Termination Species and Chemical Bonding in the Energy Storage Materials MXenes (Invited)

M. Magnuson*[1]

1. Linköping University, Department of Physics, Chemistry and Biology (IFM), Sweden

The electronic structures and the adsorption sites of the termination species in the 2D ceramic materials MXenes were investigated by the polarized synchrotron radiation methods X-ray absorption near edge structure (XANES), Extended X-ray absorption fine structure (EXAFS), and resonant photoemission (RPES). These techniques are shown to be particularly useful for detecting the detailed electronic structures that yield anisotropic information about the symmetry of the termination species at the interfaces. Valence band studies using synchrotron radiation reveals how the structures react to increasing temperature and gas adsorption. By varying the termination species and adsorbed molecules, constituting elements and structures of MXenes, a change of the electron population cause a change of covalent bonding between the laminated layers, that enables tuning of the macroscopic properties of the materials. The role of temination species at the interfaces and their local symmetries at different adsorption sites are discussed. Calculated spectra using density-functional theory (DFT) including core-to-valence dipole matrix elements yield consistent spectral functions of experimental data.

3:50 PM

(ICACC-S12-005-2024) MXene Derived Carbides As Precursors For Ultra High Temperature Ceramics

S. Nemani*[1]; Y. Im[6]; N. Gilli[2]; B. Sapkota[4]; A. Kumar[3]; A. Vohrees[1]; L. Silvestroni[5]; R. Klie[4]; N. Chawla[3]; B. Anasori[6]

1. Indiana University--Purdue University, Mechanical Engineering, USA
2. CNR-IMM, Ceramics Engineering, Italy
3. Purdue University, Materials Science, USA
4. University of Illinois, Physics, USA
5. CNR, ISTEC, Italy
6. Indiana University – Purdue University, Mechanical and Energy Engineering, USA

$Ti_3C_2T_x$ MXene is a two-dimensional (2D) refractory carbide which has high solution processability, high aspect ratios, and exhibits one of the highest stiffness (~330 GPa) among solution-processable 2D materials. We have investigated a one-pot, surfactant-free, aqueous mixing method to develop homogeneous ZrB_2-Ti_3C_2 MXene green bodies. We present the phase transformation of Ti_3C_2 MXene to TiC_y at the grain boundaries upon sintering, role of cationic species on texturing, the interface interactions between the ZrB_2-TiC_y grains and their densification mechanisms. A nominal relative density of ~96% is achieved when 0.5 wt.% Ti_3C_2 MXene is added to ZrB_2 and spark plasma sintered at 1900C with 50 MPa pressure in inert atmospheres. The microstructure evolution, stability, and their effect on micromechanical properties of the resulting UHTCs will be presented and discussed. This study lays the groundwork for 2D MXenes to be used as template precursors for 2D carbides and their implementation as compatible materials for high-temperature applications.

4:10 PM

(ICACC-S12-006-2024) Non-conventional synthesis and characterization of carbonitride MAX phases

N. Kubitza*[1]; C. Birkel[2]

1. Technical University Darmstadt, Eduard-Zintl-Institute, Germany
2. Arizona State University, USA

The vast majority of MAX phase chemistry is represented by solely carbide-based syntheses, whereas nitride and carbonitride phases are significantly underrepresented. This can be related to a more demanding synthesis procedure due to the high stability and low diffusion rate of nitrogen-containing compounds. However, by combining non-conventional solid-state methods, such as microwave-assisted heating, the liquid ammonia method, or the so-called “urea-glass method”, with conventional solid-state preparation techniques, it is possible to approach the field of nitrogen-containing MAX phases using an efficient two-step synthesis process. First, nitrogen-containing precursors are synthesized, where microwave and liquid ammonia-based methods provide binary transition metal nitride precursors, whereas the urea-glass sol-gel method results in ternary transition metal carbonitrides. Afterwards, the precursors are mixed with elemental precursors to ensure “MAX” stoichiometry. Subsequent heat treatment finalizes product formation. The combination of these methods enabled the synthesis of new materials such as $V_2GaC_{1-x}N_x$, $Cr_2GaC_{1-x}N_x$, and $V_2GeC_{1-x}N_x$, respectively. Particularly, the latter example impressively shows how the nitrogen content in the MAX phase can specifically influence materials properties, such as magnetism or transport behavior, and thus strongly supports the potential of these types of materials.

S13 Development & Applications of Adv Ceramics & Composites for Nuclear Fission/ Fusion Energy Sys

SYMPOSIUM 13: Fuel, cladding, assembly, and core evolutions and performance modeling

Room: Ballroom 4
Session Chair: Koroush Shirvan, Massachusetts Institute of Technology

1:30 PM

(ICACC-S13-034-2024) Nuclear fuel performance simulations of gas evolution, swelling and creep informed by lower length scale investigations (Invited)

A. D. Andersson*[1]

1. Los Alamos National Laboratory, USA

Fission gas evolution, swelling and creep in nuclear fuels are controlled by atomic scale diffusion processes that are governed by the chemistry of the fuel, temperature, and irradiation conditions. In order to facilitate accelerated qualification of new fuel types, mechanistic models have been developed for these processes in several fuel types, e.g. UO_2 and UN. This was accomplished by first investigating atomic scale defects and diffusion processes using density functional theory (DFT) and empirical potential calculations. The results of these calculations are then used to parametrize cluster dynamics models describing the response to irradiation. Finally, the gas evolution, swelling and creep rates are captured by use of the rates predicted by cluster dynamics simulations in model equations governing the macro-scale response of the fuel pellet. The engineering scale fuel pellet behavior is predicted by the Bison fuel performance code. This approach will be showcased by select examples for UO_2 and UN. Both the lower length scale and engineering scale predictions for these two fuel types are compared to available experimental data. To demonstrate the ability to reduce uncertainties of the mechanistic models, a Bayesian framework was developed to calibrate the model to experimental data for UO_2.

2:00 PM

(ICACC-S13-035-2024) Multiscale modeling of Silicon Carbide Cladding

G. Singh*[1]; P. Xu[1]

1. Idaho National Laboratory, USA

The engineering scale properties and performance of Silicon carbide (SiC) fiber-reinforced SiC matrix composite are based on individual properties of the constituents (fiber, matrix and interphase), the composite architecture and the microstructure. The recent efforts on multiscale modeling of SiC composite cladding for predicting the engineering scale properties from the microstructure and lower length-scale properties, obtained through the post-irradiation examination, will be presented.

2:20 PM

(ICACC-S13-036-2024) Empirical and Constitutive Surrogate Damage Modeling Techniques Capturing Progressive Failure Mechanisms in Nuclear Grade Silicon Carbide Composites

J. Kosmata*[1]; H. Shatoff[1]; P. Mijatovic[1]; G. Jacobsen[1]; L. Capolungo[2]; R. Lebensohn[2]; K. Spilker[2]

1. General Atomics, Nuclear Technologies and Materials, USA
2. Los Alamos National Laboratory, USA

Silicon carbide (SiC) ceramic matrix composites are being developed by General Atomics Electromagnetic Systems for use in nuclear reactors due to their advantageous temperature and radiation tolerance. The combination of a SiC matrix with internal SiC fiber reinforcement, (SiC_f/SiC_m) provides pseudoductility and crack arresting features to the SiC ceramic material resulting in a structure with an improved ability to withstand reactor normal operation and accident scenarios. However, current modeling capabilities struggle to accurately predict material behavior during irradiation due to its complex fiber weave structure and failure mechanisms. Two methods of modeling (SiC$_f$/SiC$_m$) ceramic matrix composite behavior will be presented: a finite element based modeling method utilizing empirically derived ceramic matrix composite geometry and empirically derived properties, and a constitutive modeling method capturing material performance via homogenization and incorporation of key failure mechanisms in a representative volume element. These models predict formation of and capture effects of microcrack formation and propagation within these materials under a variety of loading configurations. How the development of these models fit within the greater accelerated fuel qualification (AFQ) framework will be discussed.

2:40 PM

(ICACC-S13-037-2024) Physics-Informed Artificial Intelligence for Guided Wave Non-Destructive Evaluation of Ceramic Cladding

J. Harley*[1]; G. Subhash[2]

1. University of Florida, Department of Electrical and Computer Engineering, USA
2. University of Florida, Mechanical and Aerospace Engineering, USA

Nuclear fuel cladding is the first external barrier in preventing the radioactive fission products from escaping the fuel pellet assembly. Silicon carbide fiber-reinforced, silicon carbide matrix (SiC$_f$-SiC$_m$) composites have emerged as a highly viable candidate for this cladding. Inspection of these materials remains challenging. Available ultrasonic nondestructive evaluation is not effective in assessing the quality of cladding tubes due to their high curvature, multiscale anisotropy, and heterogeneity. Standard inspection is based X-ray computed tomography and optical or electron microscopy, which require specimens to be sectioned, are slow, and are not amenable to online monitoring. We present an approach, amenable to on-line monitoring, based on physics-informed machine learning and guided waves, measured using a laser Doppler vibrometer system. With current analysis methods, the ultrasonic spatial-spectral anisotropic and heterogeneous relationships within the material systems that define defects are difficult to extract. Our physics-informed machine learning framework extracts knowledge directly from experimental data without an underlying analytical basis. The proposed framework learns material characteristics from the wave equation, allowing the framework to adapt to many scenarios. We discuss example scenarios and demonstrate how material properties are extracted.

S15 8th International Symposium on Additive Manufacturing and 3-D Printing Technologies

SYMPOSIUM 15: Direct Writing and Multi-Materials I

Room: Coquina H
Session Chair: Alberto Ortona, SUPSI

1:30 PM

(ICACC-S15-039-2024) Ceramic Additive Manufacturing Technologies as Enabler for Multifunctional Components (Invited)

T. Moritz*[1]; U. Scheithauer[1]; E. Schwarzer-Fischer[1]; S. Weingarten[2]

1. Fraunhofer IKTS, Processes/Components, Germany
2. AMAREA Technologies GmbH, Germany

Ceramic additive manufacturing methods are on the way to reach a productive level. The advantages of these processes are particularly evident in the manufacture of multifunctional components. The article is dedicated to the development of ceramic components with the property combinations electrically conductive/electrically insulating via oxide and non-oxide materials, which were built via additive manufacturing routes. The processes used are Vat Photopolymerization (VPP) and Multi Material Jetting (MMJ). The advantages and disadvantages of both processes for multi-material processing are presented and underpinned with current development results and components made by both techniques for different applications. Whereas VPP deposits material layer-wise and cures it by means of light of a defined wavelength, MMJ uses a dispensing unit to deposit thermoplastic microdroplets at specific points and solidifies them just by cooling. This method also makes it possible to build up functionally graded structures in a targeted manner. The VPP process is applied to translucent ceramic particles, i.e., to titania and zirconia. In contrast, the MMJ technology can be used for opaque powders like dark ceramics and metal powders too.

2:00 PM

(ICACC-S15-040-2024) Towards Additive Manufacturing of Thin-walled "Magnetocaloric Structures" *WITHDRAWN*

V. Sharma*[1]; R. Hadimani[1]; H. Zhao[1]; R. Barua[1]

1. Virginia Commonwealth University, Department of Mechanical and Nuclear Engineering, USA

Magnetic refrigeration is an energy-efficient, environmentally friendly alternative to conventional vapor-compression cooling technology. A key challenge in manufacturing magnetocaloric devices is the lack of fabrication methods for shaping the brittle caloric alloys into thin-walled channeled structures with ideal heat transfer properties while maintaining the material's functionality. To this end, a Direct-Ink-Writing (DIW) extrusion-based additive manufacturing (AM) method has been developed to 3D print porous magnetocaloric structures with complex architecture. The novelty of our research effort lies in the initial material feedstock that consists of magnetocaloric powders (up to ~ 90 wt%) and a sacrificial polymer binder in a multi-solvent system. The solvents control ink rheology, while the polymers bind magnetic particles, retaining the regenerator structure. A two-step sintering process removes the binder and, subsequently, promotes grain growth and densification of the final finished part. Key fundamental factors about the AM process development such as powder morphology, ink viscosity, printing speed, and sintering conditions affect the function and strength of the printed part will be discussed. Overall, this study cost-effective strategies for creating magnetocaloric heat exchange structures, potentially removing one of the main barriers to commercialization of magnetic cooling technology.

2:20 PM

(ICACC-S15-041-2024) Preparation and characterization of 3D printed ceramic electrolyte for batteries

M. Faral*[1]; A. Laventure[1]; M. Dollé[1]

1. University of Montreal, Chemistry, Canada

Recent advances in 3D printing not only bring interest in the design of all-solid state battery components, but also support the optimization and study of shaping properties. Thanks to its flexibility, this technique offers the possibility of creating customizable shapes that allow a new approach, not permitted by conventional manufacturing methods. However, challenges remain in optimizing ink formulations, printing parameters, and performance to develop and maintain a printable structure before considering its integration into a system. Due to the limited information in the literature, the objective of this work is to investigate the parameters for 3D printing a ceramic electrolyte. The first part of the study consists in evaluating the limitations and feasibility of printing a solid composite electrolyte with a complex architecture. Thus, the direct-ink writing technique will be discussed, including the development of a ceramic ink formulation using a sacrificial polymer. Other aspects will be addressed such as ink printability via rheology, processing and print fidelity via direct-ink writing. Then, their properties will be discussed using localized ionic conductivity characterization methods. These tests are performed to evaluate the impact of the processing technique on the system properties.

2:40 PM

(ICACC-S15-042-2024) Direct Write Additive Manufacturing (DWAM) and Testing of Batteries for Aerospace Applications

Z. J. Tuchfeld*[1]; A. S. Almansour[3]; R. M. Sullivan[3]; D. Dornbusch[3]; M. Singh[2]; M. C. Halbig[3]

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

There are critical needs for light weight and compact energy storage systems which can be made on-demand and can store energy and rapidly discharge the energy for aircraft mission cycles of charging, take-off, and landing, as well as for emergencies. Design limitations of conventional battery manufacturing can be overcome by utilizing advanced materials synthesis and 3D additive manufacturing. In this presentation, electrochemical modeling guided design and direct write additive manufacturing (DWAM) of lithium iron phosphate (LFP) and lithium titanate (LTO) battery electrodes were optimized. Various ink formulations were developed, and sintering parameters were optimized. Optical microscopy and profilometry analysis of printed electrodes were carried out. DWAM method was optimized to yield 20+ uniform layers of both LFP and LTO. In addition, coin cell testing was conducted on half-cell configurations for materials sintered at different temperatures to improve the electrochemical performance. From these tests, it was found that sintering at 550° C was ideal for LFP. Also, carbon coated LFP particles outperform those without carbon coating. Additionally, TGA, SEM, and XRD analyses were performed to further analyze thermal stability, moisture content, and purity.

S16 Geopolymers Inorganic Polymers and Sustainable Construction Materials

SYMPOSIUM 16: Sustainable materials and novel applications

Room: Coquina C
Session Chair: Claus Rüscher, Leibniz University Hannover

1:30 PM

(ICACC-S16-023-2024) Geopolymer based Adsorbents for effective adsorption and selective separation of CO_2 and pollutants (Invited)

V. Medri*[1]; E. Papa[1]; E. Landi[1]; D. Frascari[2]; D. Pinelli[2]; M. Minelli[2]

1. National Research Council of Italy, ISSMC (former ISTEC), Italy
2. University of Bologna, DICAM, Italy

Geopolymers are increasingly studied as alternative, economical and eco-sustainable solid adsorbents for the removal of pollutants from liquid or gaseous systems. Optimized stoichiometry, phase composition, and structural and textural properties give the ability to tailor geopolymers for specific adsorption purposes. The development of geopolymer systems for separation and purification applications will be presented, with the focus on 1) adsorption and separation of carbon dioxide from concentrated sources of CO_2 (e.g. flue gas), in the context of a carbon capture and storage (CCS) strategy; 2) removal of ammonium or phosphates from wastewater with the aim of preventing eutrophication and recovering critical raw materials. The goal includes the correlation between material formulation, properties and performances for the targeted application, thus leading to the identification of the most suitable geopolymer-based material. Research activities are funded in the framework of the GEA project - Prot. 20229THRM2 by PRIN 2022 PROGETTI DI RICERCA DI RILEVANTE INTERESSE NAZIONALE.

2:00 PM

(ICACC-S16-024-2024) Environment Friendly Ceramics for Sustainable Building Construction (Invited)

S. Mittan*[1]

1. M\S Roop Chand Virender Pal, Field Operations, India

The construction industry is a significant contributor to environmental degradation consuming vast amounts of resources and energy. To address this concern there is a growing need for sustainable building materials that minimize the environmental footprint. This research paper explores the potential of environment friendly ceramics as a promising alternative for building construction. Environment friendly ceramics are characterized by their low energy consumption during production and their durability making them an attractive option for sustainable construction. This paper examines the environmental benefits of utilizing recycled and low-impact ceramic materials such as fly ash-based ceramics and waste-derived ceramics in building applications. These materials not only reduce waste but also decrease greenhouse gas emissions compared to conventional construction materials. Furthermore, this study investigates the structural integrity, thermal performance and cost-effectiveness of environment friendly ceramics in building construction. Through a comprehensive analysis this research provides insights into the feasibility and advantages of adopting ceramics as an eco-conscious choice for constructing environmentally responsible buildings.

2:30 PM

(ICACC-S16-025-2024) New applications, advantages, and current limitations of Phosphate cements (Invited)

H. A. Colorado L.*[1]

1. Universidad de Antioquia, Colombia

This investigation research presents the phosphate cements, with his advantages, applications, and current limitations. Phosphate cements, also known as chemically bonded phosphate ceramics are inorganic materials extensively used in bioengineering, nuclear waste stabilization, building, and in other structural applications. The main advantages are their suitable chemistry and capacity to involve as raw material hazardous wastes, and their high mechanical and fire-resistant characteristics in some compositions. There are still current limitations which will are discussed in this presentation.

S18 Ultra-High Temperature Ceramics

SYMPOSIUM 18: Processing-Microstructure-Property Relationship

Room: Coquina A
Session Chairs: Helmut Riedl, TU Wien; Lavina Backman, U.S. Naval Research Laboratory

1:30 PM

(ICACC-S18-031-2024) In-situ micro-mechanical evaluation of graded ultra-high temperature ceramic matrix composites (UHTCMCs) (Invited)

K. Detwiler*[4]; H. Gross[1]; E. E. Boakye[2]; T. Key[2]; A. Rossi[3]; C. P. Przybyla[4]; M. Cinibulk[4]; L. M. Rueschhoff[4]

1. Strategic Ohio Council of Higher Education, USA
2. UES Inc., Materials Science, USA
3. University of Dayton Research Institute, USA
4. Air Force Research Laboratory, Materials and Manufacturing Directorate, USA

UHTCs offer desirable oxidation behavior for high-temperature, extreme environment aerospace applications. However, UHTC mechanical performance is limited due to their low fracture toughness and thermal shock resistance. One such way to take advantage of the oxidation performance while maintaining mechanical integrity

is to reinforce UHTCs with continuous ceramic fibers to form a UHTCMC. Specifically, the UHTCMC matrix layers can be graded in such a way that surface layers that would be exposed to oxidants are richer in UHTC The intermediate layers have a decreasing ratio of UHTC to provide a gradual change in the coefficient of thermal expansion as an attempt to decrease risk of interlaminar stresses and spallation of the oxidation-resistant UHTC layers. This logic was employed to produce graded UHTCMCs that had surface matrix layers richer in HfB_2 with decreasing ratio balanced with C matrix, reinforced with C fibers. In-situ mechanical testing in an SEM is a tool that can be used to evaluate mechanical response of constituents. Specifically, in-situ flexure testing was completed in the SEM to observe the method of crack propagation and effectiveness of the matrix grading. It was observed that the UHTC rich layers did not preferentially delaminate, and instead micro-cracks propagated through multiple plies through the width of the flexure bar.

2:00 PM

(ICACC-S18-032-2024) Microstructural and Mechanical Properties of Ultra-High Temperature Ceramics Produced through the Bound-Ceramic FFF Additive Manufacturing Process

E. Faierson*[1]; P. Collins[1]

1. Iowa State University, USA

Utilizing the Fused Filament Fabrication (FFF) additive manufacturing process with feedstock comprised of ceramic particles in a polymer binder is a relatively new technique for fabricating ceramic parts. This study investigated several Ultra-High Temperature bound-ceramic filament feedstocks, incorporating ceramics such as SiC, B_4C, TiB_2, and novel ceramic-composite powders. FFF process parameters were developed to facilitate fabrication of suitable specimens. After fabrication the samples were consolidated using debinding and sintering techniques. Processing-Microstructure-Mechanical property relationships were investigated and optimized in order to reduce defects and obtain the best mechanical properties in the samples after sintering. The ability to fabricate novel ceramic lattice structures using bound-ceramic FFF technology was also investigated.

2:20 PM

(ICACC-S18-033-2024) Densification, Thermal Properties, and Grain Boundary/B4C TEM Characterization of Zirconium Diboride (ZrB2) with Carbon Additions

Y. Zhou*[1]; W. Fahrenholtz[1]; G. Hilmas[1]

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

Commercial Grade B ZrB2 powder with carbon additions up to 1.5 wt% were fabricated by hot-pressing. Phenolic resin was used as carbon source, the char yield was measured prior mixing with ZrB2. The particle size of the starting powders was measured by laser light scattering method and further confirmed by SEM observation. Impurity elements contents were examined using inert gas-fusion method. The starting powders were hot-pressed at 2150°C with a pressure of 32 MPa. Bulk densities were measured by Archimedes method. Morphology of the cross section was observed by SEM. EBSD was taken and the images were used for grain size calculation. The effect of carbon content on thermal conductivity was also analyzed. Finally, grain boundaries were characterized using TEM to identify impurity phases.

2:40 PM

(ICACC-S18-034-2024) High-Temperature Kinetic Behavior of Boride Compounds When Exposed to CMAS

T. Y. Ansell*[1]; L. Raschke[1]; C. Vorbroker[1]

1. Naval Postgraduate School, Mechanical and Aerospace Engineering, USA

Ever-increasing temperatures within gas turbine engines require alternative ceramic materials for thermal and environmental barrier coatings (E/TBC). The current trend in the field has been to investigate yttrium mono- or di-silicates, Y_2SiO_5 and $Y_2Si_2O_7$, respectively as replacements for yttria-stabilized zirconia. At temperatures exceeding 1300 °C, however, both silicates break down and lose their effectiveness as T/EBCs in the presence of CMAS deposits. This necessitates a similar investigation of other ultra-high temperature ceramics. In this work, we looked at the effect of CMAS on bulk borides (HfB_2, ZrB_2, and a compositionally complex diboride, HEB) at high temperatures. AFRL-02 powder was placed on the borides and was then heated, in air, up to either 1300 or 1600 °C for one or ten hours. The resultant microstructural changes, oxide scale formation, and material breakdown were studied. HfB_2 survived ten hours at 1600 °C with most of the microstructure oxidizing but some remaining boride phase remaining. ZrB_2 similarly resisted CMAS attack at 1600 °C but only for one hour; completely oxidizing after ten hours at that temperature. The HEB samples saw relatively rapid oxidation completely transforming at temperatures lower than 1600 °C.

3:20 PM

(ICACC-S18-035-2024) Exploring the phase space of transition metal diborides: From binary to quaternary PVD coatings (Invited)

H. Riedl*[1]; L. Zauner[2]; A. Bahr[2]; T. Glechner[2]; A. Hirle[2]; S. Richter[2]; C. Fuger[2]; R. Hahn[2]; T. Wojcik[1]; J. Ramm[3]; O. Hunold[3]; S. Kolozsvári[4]; P. Polcik[4]

1. TU Wien, Institute of Materials Science and Technology, Austria
2. TU Wien, Christian Doppler Laboratory for Surface Engineering of high-performance Components, Austria
3. Oerlikon Surface Solutions AG, Liechtenstein
4. Plansee Composite Materials GmbH, Germany

Transition metal diboride (TMB) coating materials are the subject of growing research interests. They are considered potential candidates in future industrial applications ranging from thermo-mechanical purposes in UHT environments to electrochemical needs. Exploring the phase space of novel TMBs poses specific challenges: (i) susceptible compositional and structural variety during physical vapor deposition (PVD), (ii) anisotropy in their mechanical properties as well as brittle nature, and (iii) formation of non-adherent and volatile oxide scales. Within this talk, we want to address these specific challenges on a step-wise alloying concept from binary to quaternary group IV to VI transition metal diborides. The anisotropic nature and impact of B-rich tissue phases on the intrinsic fracture toughness of superhard binary $TiB_{2\pm z}$ and $WB_{2\pm z}$ are elaborated as a starting point. Subsequently, different alloying routes (e.g. TM-X-$B_{2\pm z}$, TM-X-Si-$B_{2\pm z}$) will be discussed to enhance the ductile character and oxidation resistance. Here, Si and disilicide alloying routes have been proven highly effective in providing long-term oxidation resistance up to 1200 °C (e.g. < 350 nm oxide scale after 10 h). To describe all these relations comprehensively, we correlated the synthesis parameters with structural and morphological evolution using XRD, HR-TEM, APT, ERDA, and micro-mechanical testing methods.

3:50 PM

(ICACC-S18-036-2024) Interdiffusion in the WB2 – ZrB2 system

Y. Zhou*[1]; S. Filipovic[1]; W. Fahrenholtz[1]; G. Hilmas[1]

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

The interdiffusion behavior in the WB2-ZrB2 was studied using diffusion couples. WB2 and ZrB2 powders were synthesized by boro-carbothermal reduction. The materials used for WB2 synthesis were commercial WO3, B4C, and carbon black. WB2 was synthesized at 1600°C in vacuum for 2.5 hours. The synthesized powder was densified by spark plasma sintering at 1700°C in vacuum under a pressure of 50 MPa for 10 minutes. For ZrB2, the starting materials were ZrO2, B4C, and carbon black. Synthesis was performed at 1650°C in vacuum for 1 hour, and the densification was accomplished by spark plasma sintering at 2050°C in vacuum under a pressure of 50 MPa for 10 minutes. Polished surfaces of WB2 and ZrB2 ceramics were used to make diffusion couples that were annealed at 2000 – 2200°C for different periods of time. The morphologies and elemental concentrations near the WB2 - ZrB2 interfaces were characterized using scanning electron microscopy and energy dispersive spectroscopy. Interdiffusion flux profiles were constructed based on the EDS line scans, and the interdiffusion coefficients were calculated. Finally, Arrhenius plots of the diffusions for each element were established and the corresponding activation energies were calculated.

4:10 PM

(ICACC-S18-037-2024) Aligned Boron Nitride Nanotube Polysiloxane Ablative Nanocomposite Laminate for Thermal Protection Systems

P. B. Patel*[2]; S. Kim[4]; J. Dai[1]; L. Acauan[1]; J. Buffy[3]; J. Koo[4]; B. L. Wardle[1]

1. Massachusetts Institute of Technology, Aeronautics and Astronautics, USA
2. Massachusetts Institute of Technology, Mechanical Engineering, USA
3. Techneglas, USA
4. The University of Texas at Austin, Walker Department of Mechanical Engineering, USA

For thermal protection systems, aerospace polysiloxane ultra high temperature resin (UHTR) is often utilized as ablative material. It is beneficial for the material to possess characteristics such as low thermal conductivity, low density, high thermal resistance, as well as the ability to form a strong and stable char. In order to provide better mechanical properties, fiber-reinforced UHTR composites are used in the aerospace industry due to their high strength-to-weight ratio. Nanofibers, like boron nitride nanotubes (BNNTs), have excellent properties such as high specific strength, low density, thermal stability, and radiation shielding capabilities. This makes them ideal for reinforcing UHTR, compared to traditional microfibers, and for space applications where radiation protection is necessary. Current methods for integrating BNNTs into resins often result in nanocomposites with voids and agglomerated and randomly oriented BNNTs, creating a low packing density, which results in low improvements in mechanical and multifunctional properties. To overcome this shortcoming, a novel bulk nanocomposite laminating (BNL) process has been used in this study which combines a BNNT orientation and densification step with a resin polymer infiltration process. The BNNT/polysiloxane composite manufactured will be characterized and tested for mechanical and ablative properties.

4:30 PM

(ICACC-S18-038-2024) Investigating Thermal and Optical Properties in Rare Earth Zirconates for Radiative Barrier Coatings

W. Riffe*[1]; H. B. Schonfeld[3]; V. Champagne[4]; S. Zare[3]; P. E. Hopkins[2]; D. Clarke[4]

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, Materials Science, 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, laser radiometry, and spectroscopic ellipsometry to elucidate temperature-dependent thermal conductivities and optical properties of rare earth zirconates. With a new laser-based metrology, thermal conductivities and emissivities of ceramics over 2000 °C can be measured. Understanding these trends is important for RBCs that need to endure cycling to operating temperatures. Through ellipsometry, the lifetimes of optical phonons can be understood. Anharmonic scattering dominates thermal transport at high temperatures so measuring lifetimes at temperature is important to understand fundamental energy transport in RBCs. By investigating pertinent physical scattering mechanisms and optical properties, we deconvolute key design considerations for next generation RBCs.

S1 Mechanical Behavior and Performance of Ceramics & Composites

SYMPOSIUM 1: Sustainable manufacturing, joining, and repair approaches of ceramics

Room: Coquina E
Session Chairs: Dietmar Koch, University of Augsburg;
Aurélien Debelle, Andra

1:30 PM

(ICACC-S1-037-2024) Sustainable ceramic matrix composites (SCMC) by combining life cycle assessment (LCA), multicriteria analysis and digitalization (Invited)

A. Schneller*[1]; D. Koch[1]; D. Schüppel[1]; T. Schneider[1]; L. Wietschel[1]; J. Riesner[1]; A. Thorenz[1]; A. Tuma[2]

1. University of Augsburg, Institute for Materials Resource Management, Germany
2. University of Augsburg, Institute of Business Administration, Germany

The demand for the ecological footprint (e.g. CO2) of materials from the political and social sphere is becoming more and more imperative. At the same time, for energy and resource demanding CMC materials there are almost no studies on LCA. The innovation of this study for developing SCMC materials is that LCA is combined with Pareto analysis. The latter identifies processes that have a major impact on, e.g. environmental indicators. The combination of both methods allows the representation of the contribution of relevant impact categories (e.g. global warming potential, water consumption, human carcinogenic toxicity) to the damage categories (human health, ecosystem quality, resources). It shows, i.a. the influence of curing and tempering temperatures during CMC processing on different impact categories. It is a targeted approach to environmental improvement by allocating limited resources to areas where they have the greatest impact. Approaches for the digitalization of CMC manufacturing processes and their linkage to an end-to-end networked and intelligent process chain are shown. This offers the opportunity to unlock further resource and energy efficiency potential. In combination with the LCA and Pareto analysis, this approach makes it possible to identify system correlations and synergy effects along the whole SCMC manufacturing process.

2:00 PM

(ICACC-S1-038-2024) Pareto Efficient manufacturing of C/C-SiC via LSI: An overview of energy saving potentials and the associated performance loss

D. K. Schüppel*[3]; A. Schneller[2]; O. M. Avci[2]; F. Halter[1]; L. Wietschel[1]; D. Koch[2]

1. University of Augsburg, Institute for Materials Resource Management, Germany
2. University of Augsburg, Institute for Materials Resource Management MRM, Materials Engineering, Germany
3. Composites United e. V., Ceramic Composites, Germany

Much development work in the field of ceramic matrix composites (CMC) in recent decades has focused mainly on improving mechanical, chemical and thermal properties and on optimizing cost-effective production processes. Within the framework of Composites United e. V. and its industrial members, various joint research projects are now starting which also deal with ecological, economic and circular economy aspects of CMC. These aspects have hardly been investigated so far but are attracting growing interest in politics and industry due to the increasing awareness of environmental impacts. The project team of the CU EcoCeramic collaborative research project is now focusing on these issues using carbon fiber reinforced silicon carbide (C/C-SiC) as an example to show what the environmental footprint of C/C-SiC manufacturing looks like through the liquid silicon infiltration (LSI) route. On the one hand, the key performance indicators (KPIs) are considered. On the other hand, realistic future scenarios are calculated, such as the use of regenerative process energy or the further development of manufacturing technology. In addition, energy savings trough in manufacturing processes are correlated to their mechanical behaviour. So-called Pareto-efficient points help to ensure optimal, energy-saving production.

2:20 PM

(ICACC-S1-039-2024) Yttrium aluminosilicate glass-ceramic used to join SiC/SiC composites

C. Malinverni*[1]; M. Salvo[1]; M. Zietara[2]; J. Maier[3]; C. Prentice[4]; M. Farnham[4]; V. Casalegno[1]

1. Politecnico di Torino, DISAT, Italy
2. International Centre of Electron Microscopy for Materials Science, AGH University of Science and Technology, Poland
3. Fraunhofer Institute for Silicate Research ISC, Center for High Temperature Materials and Design HTL, Germany
4. Archer Technicoat Ltd, ATL, United Kingdom

Over the last years, the steelmaking industry has been the subject of interest in the context of the environmental transition to improve production processes to meet low carbon emission targets and to reduce energy inputs through the use of renewable sources. Ceramic matrix composites (CMCs) are high-performance materials with excellent thermo-mechanical properties that can operate at very high temperatures and in harsh environments, e.g. corrosion and combustion. The EU CEM-WAVE project (G.A. 958170) will develop CMCs to be used in radiant tube furnace components for this energy-intensive sector. In this work, a yttrium aluminosilicate glass-ceramic was used to join two different SiC/SiC CMCs. Two different joining processes were developed. The joints were morphologically characterized by SEM, analyzed by means of XRD, and mechanically tested by single-lap offset shear tests at room temperature, where the failure was caused by the delamination of the composite, showing excellent adhesion between the YAS glass-ceramic and the composite. The research carried out to write this article was funded under the CEM-WAVE project. This project has received funding from the European Union's Horizon 2020 research and innovation program under grant agreement No. 958170. This document only reflects the authors' view. The European Commission is not responsible for any use that may be made of the information it contains.

2:40 PM

(ICACC-S1-040-2024) Improving brazed CMC joints via surface texturing using atmospheric-pressure plasma jet and reactive ion etching

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1. Politecnico di Torino, DISAT, Italy
2. National Research Council – Institute of Condensed Matter Chemistry and Technologies for Energy (CNR-ICMATE), Italy
3. Institute for Plasma Science and Technology, National Research Council of Italy, Italy

Ceramic Matrix Composites (CMCs) are one of the promising engineering materials because of their exceptional thermo-mechanical characteristics and reduced weight. The integration of CMC components to similar or dissimilar materials is a common practice and the joining process is crucially important. In particular, achieving robust and reliable brazed connections in CMCs remains a challenge due to their inherent characteristics, including low wettability and chemical inertness. To surmount these constraints and enhance the performance of brazed joints, surface preparation assumes a fundamental role. Specifically, surface texturing has the capacity to establish a mechanical anchoring system through interlocking when infiltrated by the bonding agent, thereby elevating the mechanical strength of the joined structures. This innovative approach refines the microscopic surface of CMCs without compromising wettability by liquid brazing alloys, thereby promoting the success of brazing operations. In this study, we explore the novel application of surface texturing, employing both atmospheric-pressure plasma jets and reactive ion etching, to modify the surface properties of C/C, C/SiC and SiC/SiC prior to brazing. Our evaluations focus on assessing how the altered surfaces impact the CMC/ braze adhesion, as well as the resulting enhancements in joint mechanical strength.

3:20 PM

(ICACC-S1-041-2024) Repair Concepts for Reduced Reject Rates of CMC Structures -Machining of Damaged Volumes and Repair Inlays (Invited)

R. Goller*[1]; S. Unseld[1]; T. Bratzdrum[3]; J. Moosburger-Will[3]; A. Rösiger[2]; D. Koch[3]

1. Technical University of Applied Sciences, Mechanical Engineering, Germany
2. University of Applied Sciences, Mechanical Engineering, Germany
3. University of Augsburg, Institute for Materials Resource Management MRM, Materials Engineering, Germany

Ceramic Matrix composites (CMC) are more and more used in aircraft engines and satellite structures. As the production numbers grow rapidly and these components have a very high value, scrap has to be avoided and used components should be overhauled for reuse. An essential step of the repair concept comprises the machining of the damaged areas. For this a volumetric scan and analysis of the damaged areas will be performed and digitized to enable a targeted removal of the damage pattern by mechanical machining. The use of new diamond tools and machining methods, which have been developed for this purpose, will be demonstrated. 3D measurements of the machined areas are used to create digital twinns, which are the basis for the fabrication of short- or long-fiber reinforced ceramic and pre ceramic inlays. In preparation for backfilling and repair the surface and the geometry of the machined area and inlays are characterized.

3:50 PM

(ICACC-S1-042-2024) Repair concepts for reduced reject rates of CMC structures – development of inlays and compounds

D. Koch*[1]; T. Bratzdrum[1]; J. Moosburger-Will[1]; S. Unseld[2]; A. Rösiger[2]; R. Goller[2]

1. University of Augsburg, Institute for Materials Resource Management MRM, Materials Engineering, Germany
2. Technical University of Applied Sciences Augsburg, Mechanical Engineering, Germany

Ceramic Matrix Composites (CMC) are promising candidates for application under severe conditions. For broader application quality issues like prevention of scrap and repair and overhaul of parts after use for a second life become more and more important. For repair of damaged volumes in nonoxide fiber reinforced composites two "dental" strategies have been developed. First, machined inlays which consist of the original material are fixed with a bonding system. Second, a compound system out of short fibers, fillers, and preceramic polymers is filled into the cavities and then pyrolyzed and siliconized. These heat treatments are performed locally via inductive heating. The challenges of these two methods are evaluated considering composition, inductive coupling, conversion and shrinkage during heat treatment, microstructural properties, and bonding with the pristine substrate. Finally, non-destructive and mechanical testing proof the quality of the proposed local repair methods.

4:10 PM

(ICACC-S1-043-2024) Multimaterial and functionally graded structures through ceramic additive manufacturing (Invited)

S. M. Allan*[1]; M. Schwentenwein[2]; S. Nohut[2]

1. Lithoz America, LLC, USA
2. Lithoz GmbH, Austria

Co-processing of materials through additive manufacturing can enable parts with increased functionality. Lithography-based ceramic manufacturing (LCM) provides an opportunity to produce high resolution parts in a variety of materials combinations. Materials combinations that have been investigated include hydroxyapatite (HA)/tricalcium phosphate (TCP), porous/dense alumina, porous/dense HA, alumina/zirconia-toughened-alumina (ZTA), and copper (Cu)/glass ceramic. The challenges encountered and strategies for addressing issues in co-processing will be discussed. Results to date show great potential for multi-material and functionally-graded printing, and highlight significant areas for further research.

4:40 PM

(ICACC-S1-044-2024) Optimized microstructure for enhanced properties of novel ecofriendly green plants wastes hybridized ultrafine grained Al7Si2Cu0.5Ni eco-composites

K. C. Nnakwo*[1]

1. Nnamdi Azikiwe University Awka Nigeria, Metallurgical and Materials Engineering, Nigeria

The utilization of eco-friendly green plant waste materials as reinforcing agents in aluminium alloy-based composites represents a sustainable and environmentally responsible approach to material development. The primary objective of this research is to explore the reinforcing characteristics of Irvingia wombolu shell nanoparticulates (IWSNp)/carbon nanotubes (CNTs) hybrid on the electrical and thermo-mechanical properties of Al-7Si-2Cu-0.5Ni eco-composite. The CNTs were synthesis from Rice Husk. The IWSNp and CNTs were prepared using a sol-gel technique. The IWSNp and CNTs hybrid were in the ratio 2:0.5, 2:1, 2:2, 0.5:2, and 1:2. The eco-composites were inoculated by Al-Nb-V-Zr master alloy and subjected to thermo-mechanical treatment (cold worked/homogenized/hot worked/aged at 450ºC and 480ºC for 2-12h) to produce a high strength, super electrical and thermal conductivity ultrafine-grained Al7SiCu0.5Ni/IWSNp/CNTs eco-composite. The microstructure evolution and Phase compositions of the eco-composites were analyzed using Optical Microscope, Scanning Electron Microscope (SEM), X-ray Diffractometer (XRD), and Energy Dispersive Spectroscopy (EDS).

5:00 PM

(ICACC-S1-045-2024) Evaluation of Microstructure Evolution and Mechanical Properties of Al-10wt%Zn-1.63Si Irvingia gabonensis Particulates Alloy Composites

J. L. Chukwuneke*[2]; K. C. Nnakwo[1]

1. Nnamdi Azikiwe University Awka Nigeria, Metallurgical and Materials Engineering, Nigeria
2. Nnamdi Azikiwe University, Mechanical Engineering, Nigeria

The current study was motivated by the recent search for the development and application of locally sourced eco-friendly agricultural waste as a viable reinforcement for high strength Al-Zn-Si-based composites. In this experimental study, the Al-10wt% Zn-1.63Si/Irvingia gabonensis nano-particulates composites were fabricated using stir-casting techniques. The Irvingia gabonensis nano-particulate was added in concentrations of 1, 3, 5, and 7 wt %. The chemical constituents of the Irvingia gabonenesis and the developed composite samples were analyzed using X-ray fluorescence (XRF). The microstructure evolution and mechanical properties of the developed composites were investigated using scanning electron microscopy (SEM) and an X-ray diffractometer (XRD).

S3 21th Intl Symp on Solid Oxide Cells Materials Science & Technology

SYMPOSIUM 3: Simulation, testing and degradation / Progress in SOC development

Room: Ballroom 1-2
Session Chairs: Tae Ho Shin, Korea Institute of Ceramic Engineering & Technology; Alex Morata, Catalonia Institute for Energy Research (IREC)

1:30 PM

(ICACC-S3-032-2024) Investigating Durability of Solid Oxide Electrolysis Cells (Invited)

O. A. Marina*[1]; L. Le[1]; R. Springer[1]; C. Coyle[1]; S. Belko[1]; J. Bao[1]

1. Pacific Northwest National Lab, USA

To become a commercial technology, SOECs need to demonstrate at least a 40,000-60,000 hours life. In this presentation, we will discuss our systematic performance studies of Ni-YSZ and LSCF-GDC electrodes for over 6,000 hours in a broad range of experimental conditions, while varying temperature, steam concentration, steam utilization, current, voltage, operating pressure, Cr and S contaminants, and applying dynamic operation in order to map potential degradations and establish their impact on device life. Tests were conducted in either potentiostatic or galvanostatic mode and at the open-circuit voltage (OCV) to separate effects of electrochemical aging from materials thermal aging. Multiple repeats were used to eliminate the occasional artifacts and atypical trends. Steam concentration was varied from 10 to 99% and steam utilization was adjusted to 30-85%. Electrochemical characteristics, such as current, voltage, and area specific resistances were measured continuously over time. In each test, the ohmic and polarization resistances were obtained using electrochemical impedance spectroscopy. Following each test termination, extensive post-test characterization of Ni-YSZ and LSCF microstructures using SEM/EDS, EBSD, and STEM was performed. Advanced machine learning were applied to provide the quantitative estimation on the probability of electrode microstructure variation under different operating conditions.

2:00 PM

(ICACC-S3-033-2024) Developing higher performing solid oxide cells through simulation (Invited)

H. W. Abernathy*[1]; W. K. Epting[1]; T. Yang[1]; Y. Lei[1]; F. Xue[1]

1. US DOE National Energy Technology, USA

Through the US Department of Energy's Reversible Solid Oxide Fuel Cell (R-SOFC) and H2NEW program, the National Energy Technology Laboratory (NETL) has developed a comprehensive solid oxide cell (SOC) multiphysics performance degradation modeling framework. The framework uses microstructurally resolved information about an SOC to predict its lifetime performance under different operating modes (SOFC, SOEC, R-SOC) and subjected to selected degradation modes (e.g., coarsening, Cr poisoning, cation interdiffusion, cracking/delamination). By varying the operating conditions and the composition and structure of the electrodes, one can optimize the cell based on a desired metric, such as degradation rate or lifetime power/hydrogen production. The simulations are used to explore more efficiently a larger multi-dimensional parametric space and to find conditions and compositions that are not currently practically accessible, but that could be used to guide SOC development. The talk will review the current capabilities of the modeling framework and will present case studies that provide lessons on SOC electrode design and operation.

2:30 PM

(ICACC-S3-034-2024) Stable operation of Metal-Supported Electrolysis Cells for 7000 hours

J. Zamudio Garcia*[1]; M. P. Klitkou[2]; Å. H. Persson[1]; B. Sudireddy[1]; A. Hagen[2]; P. Hendriksen[1]

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

Solid oxide electrolyser technology has the potential to play a key role in energy conversion devices for large-scale power-to-X applications. Recently, Metal-supported solid oxide cells (MS-SOC) have attracted great attention due to their lower costs and increased mechanical robustness when compared to ceramic fuel electrode and electrolyte-supported configurations, leading to expected longer lifetime under dynamic operating conditions. The present study investigates for the first time the long-term durability of MS-SOCs in electrolysis mode, operating under galvanostatic conditions for 7000 h in 50 % H_2O/H_2 at -0.5 A cm^{-2} at 650 °C. The cell voltage over time showed a very low degradation rate of ~ 1.5 %/1000 hours. The EIS analysis under load during the durability test unveiled that the principal increase in total resistance was attributed to polarization contributions, while only a slight increase in ohmic resistance was observed. The cross-sectional SEM images revealed very low oxidation of the metal-support (MS) due to the formation of a continuous 250 nm thick chromia scale, which acts as a protective barrier for further corrosion. Additional corrosion experiments at high steam content (90% H_2O/H_2) suggested that the infiltration of Ni-CGO active catalysts into the MS prevents chromium evaporation. In contrast, uncoated samples exhibit breakaway corrosion after 500 h exposure.

2:50 PM

(ICACC-S3-035-2024) Degradation behavior of CFY based SOC stacks with different types of interconnect protection coatings

V. Sauchuk*[1]; S. Rothe[1]; S. Megel[1]; N. Trofimenko[1]; M. Andritschky[2]; D. Heitzinger[3]; M. Kusnezoff[4]

1. Fraunhofer IKTS, Germany
2. Minho University, Portugal
3. Miba Group company, Austria
4. Fraunhofer IKTS, Germany

The alloy CFY, containing more than 90 ma. % of chromium, is used as a metallic interconnect (MIC) in the MK35x series stacks developed by IKTS. Due to high reactivity and oxidizing ability of chromium, CFY interconnect must be provided with a protective layer to guarantee the long-time operation of the fuel cell stack. A CuNiMn spinel compound applied by wet powder spraying of the paste served as a basis for the development of protective coatings. The pastes can be used for both protective and contact layers without significant change of the stack parameters. The physical vapor deposition method was used to apply the thin metallic films with precisely pre-set stoichiometry on the MIC surface followed by their in-situ oxidation during the stack conditioning at operation temperatures. The MICs with perovskite coating applied by atmospheric plasma spraying were tested for comparison. It was found, that tested materials or combinations behave differently depending on operation mode: SOFC and SOEC. The degradation in stacks is found to differ from the annealing results on some variants of model samples. The mechanisms causing the difference in degradation behaviors of the coatings are discussed based on the results of structure analysis, scanning electron microscopy, area specific resistance measurements and thermodynamic aspects of formation and stability of oxide phases.

3:30 PM

(ICACC-S3-036-2024) Recent Highlights on Solid Oxide Cell (SOC) developement, modelling and characterization (Invited)

J. Mougin*[1]

1. CEA, Liten, France

Hydrogen can play a key role, as a resource for industrial processes, a fuel for transportation, and a vector for high-capacity and/or long-distance electricity storage. A sharp increase in the share of hydrogen in the overall energy mix is expected. It will be necessary to increase the production capacity of low carbon hydrogen, primarily through electrolysis. At EU level, it is targeted to produce 10 million tons of H2 per year by 2030, plus 10 million tons of imports. However, the production of low carbon hydrogen is currently not competitive with conventional production routes from fossil fuels. The efficiency of the electrolysers is a key parameter to lower the cost of H2. Solid Oxide Cell (SOC) technology presents the highest efficiency and is therefore an highly promising technology to produce hydrogen at low cost. In addition, it still has a significant potential for innovation, making it possible to gain in performance and lifetime. The status of development of the technology will be presented, as well as the remaining challenges. CEA is working on SOCs technology since more than 20 years. The current R&D activities will be detailed, with a particular emphasis on cells development, modelling and characterization, aiming to understand and improve performance and durability. Finally, the cells developments will be put in perspective with their integration in stacks and modules.

4:00 PM

(ICACC-S3-037-2024) Elcogen SOC technology: Status and perspectives (Invited)

M. Rath*[1]; S. Pylypko[1]; M. Noponen[1]; M. Skjøth-Rasmussen[1]; E. Ounpuu[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 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 above 60 MW in fuel cell mode equaling to 260 MW capacity in electrolysis mode.

4:30 PM

(ICACC-S3-038-2024) Progress and Challenges on Solid Oxide Fuel Cell and Stack development for Mobility Use (Invited)

T. Shiomi*[1]; M. Sugino[1]; M. Abdul Jabbar[2]; Y. Furuya[2]; N. Dale[2]; P. Singh[3]

1. Nissan Motor Co., Ltd., EV System Laboratory, Japan
2. Nissan, USA
3. University of Connecticut, Materials Science and Engineering, USA

The research and development of SOFC (Solid Oxide Fuel Cell) has been expanding to various applications such as high temperature steam electrolysis and mobility use. To install SOFC-powered system on a vehicle, the development of metal supported cell and

stack is desirable to realize smaller volume, lower cost and more rapid start-up etc. Among lots of technical challenges, the improvement of both power density and durability is a key challenge and various efforts need to be focused. In a past presentation, AFA, alumina forming alloy, as an interconnector of a SOFC repeating unit, was proved to be very high resistive to chemical degradation under SOFC operating conditions and to be able to realize low interface resistance between AFA interconnectors by applying diffusion bonding. In this research, further progress to apply AFA as an interconnector of a metal supported-SOFC repeating unit is presented. Firstly, welding is applied to the joints between metal-supported cell and AFA interconnect. Then these metal joint methods are confirmed as a large size repeating unit. Finally further challenges to realize metal-supported cell/stack for automotive use are discussed.

5:20 PM

(ICACC-S3-039-2024) SOEC for Highly Efficient Hydrogen and E-Fuel Production (Invited)

B. Reiter*[1]

1. AVL List GmbH, SOFC/SOEC Systems, Austria

The production of hydrogen from renewable sources shows a promising path to long-term de-fossilization, now adopted by many countries in their national strategic vision. The business case depends on application in the transportation, stationary and industrial sectors. Cross-sectoral approach is critical, interconnecting user profiles to ensure energy security, despite intermittency. The energy efficiency of the hydrogen production process is of utmost importance to achieve the cost of hydrogen required for industrial and energy applications. SOEC represents a particularly interesting technology to improve the efficiency of hydrogen and e-fuels production. In the 2030 time horizon, the cost of H2 production by SOEC is estimated to be 15-25% lower than that of PEM electrolysis. Approximately 70% of the cost are OPEX and 30% CAPEX. In the future, SOEC has the potential to reach lower CAPEX than PEM electrolysis. AVL has developed a complete Power-to-Liquid plant concept in which the SOEC process is thermally coupled to the Fischer-Tropsch process. Via this thermal coupling, the production efficiency can be increased by ~30% compared to the route via PEM or AL electrolysis. Currently, AVL and IWO are building up the demonstration plant "IFE – Innovation flüssige Energie" in Austria based on this technology to proof the feasibility and efficiency of this process. Commissioning of the demonstration plant is planned in 2023.

5:50 PM

(ICACC-S3-040-2024) Co-SOEC system development for optimized SNG production

R. Schauperl*[1]

1. AVL List GmbH, Research & Innovation, Austria

Power-to-Gas (PtG) technologies represent a key technology for developing low-carbon energy systems with a high share of fluctuating electricity production from renewable sources, such as wind and solar, since surplus power can be stored by means of chemical energy carriers, typically in form of hydrogen or methane. These gases can be used as CO_2 neutral fuels, or can be re-transformed to electricity when needed. Among others, the advantage of methane over hydrogen is the already widely existing infrastructure, since methane can be fed into natural gas networks or be used as fuel for existing gas fired power plants or natural gas vehicles. The project HydroMetha was a strongly horizontal and vertical integrated joint undertaking consisting of a development service provider (AVL List GmbH, coordinator), research institutes (Energieinstitut JKU, Fraunhofer IKTS, Montanuniversität Leoben), and the Austrian small business companies Repotec and Prozess Optimal. The project has been start with a specification definition centrally, followed by parallel development activities of the key technologies CO_2+H_2O Co-Electrolysis with Solid Oxide Cells (Co-SOEC) and catalytic methanation. Subsequently, these two key technologies were coupled to a 15 kW_{el} functional unit via simulation. The presentation will outline how the key objectives of this system have been addressed and achieved. Finally, an outlook for this technology is presented.

6:10 PM

(ICACC-S3-041-2024) Viability of CO_2-Neutral Vehicles Utilizing Carbon-Based Fuels

S. Barnett*[1]; T. Schmauss[1]

1. Northwestern Univ, USA

The viability of CO_2-neutral transportation using hydrocarbon or alcohol fuels, in which the CO_2 product is captured on-board the vehicle, is examined. Solid oxide fuel cells are proposed for energy conversion because they react fuels with pure oxygen, enabling on-board CO_2 capture in a reasonable volume after compression to a moderate pressure of 250 bar; net storage volume is substantially less than of equivalent hydrogen tanks or batteries. During vehicle refueling, captured CO_2 can be off-loaded and subsequently used in fuel production with renewable resources, or sequestered, thereby maintaining carbon-neutrality. Alternatively, these Carbon-Capture Fuel Cell Vehicles (CCFCVs) can be part of a CO_2-negative pathway by using biofuels and sequestering the captured CO_2. Storage volume and weight are analyzed for a range of vehicle types, comparing the proposed transportation platform with those of internal combustion, Li-ion battery, and hydrogen fuel cell vehicles (H2FCV); the results suggest that this is the best available option for long-range vehicles. The well-to-wheels efficiencies for fossil fuels and biomass-to-wheels efficiencies for biofuels are estimated using literature data. The results indicate that the CCFCV can yield efficiencies of ~ 40%, nearly twice that of the H2FCV, ~22%.

S6 Advanced Materials and Technologies for Rechargeable Energy Storage

SYMPOSIUM 6: Advanced anode and cathode materials for lithium and multivalent batteries

Room: Ballroom 5
Session Chairs: Badri Narayanan, University of Louisville;
Derrick Fam, Institute of Materials Research and Engineering

1:30 PM

(ICACC-S6-032-2024) Research and Development of Mn-Rich Cathodes at Argonne National Laboratory (Invited)

J. R. Croy*[1]

1. Argonne National Laboratory, USA

The increasing demand for Li-ion batteries has brought about the realization that diversity in materials will be critical to ensure supply chain security, lower costs, and wide-spread adoption of energy storage technologies. In this regard, enabling new materials as viable options for Li-ion cathodes is of particular significance. More specifically, substantially increasing the use of earth-abundant materials in vehicle technologies is now viewed as a necessary path forward to ensure sustainability. Earth-abundant materials are attractive options in terms of sustainable technological development as they have the potential for positive impact with respect to increasing the availability of raw materials, ultimately lowering costs and increasing accessibility of electric vehicles. The Vehicle Technologies Office (U.S. DoE) has recently established a consortium of seven national laboratories to address the challenges of enabling more sustainable cathodes. This consortium, named "Earth-abundant Cathode Active Materials", or EaCAM for short, is focusing efforts across a wide range materials and technology readiness levels. In this presentation, we will discuss ongoing efforts within the EaCAM program aimed at the design, synthesis, and characterization of Mn-rich cathodes as alternative, next-gen materials.

2:00 PM

(ICACC-S6-033-2024) Oxygen defect engineering for advanced battery materials (Invited)

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

1. Tohoku University, IMRAM, Japan

Oxygen defect formation in cathode active materials is important phenomena for safety, stability and electrochemical performance. Although sophisticated in-situ/operando experiments were examined to understand the mechanism of oxygen release from cathode active materials, equilibrium properties of oxygen defect formation are not studied well so far. In this work, equilibrium oxygen release behavior of Li(Ni,Co,Mn)O2 (NCM) are investigated by thermogravimetry and coulometric titration. In addition, reduction of transition metals by oxygen release was evaluated by X-ray absorption spectroscopy. Above experiments revealed that NCMs with high valent Ni easily release lattice oxygen, suggesting that high valent Ni in NCMs destabilize lattice oxygen. Moreover, partial molar enthalpy of oxygen which represents necessary energy of oxygen vacancy formation (oxygen release) was calculated from thermodynamic analysis. It was revealed that oxygen release by Co reduction requires more than 2 eV, while that by high valent Ni requires only 0.5 eV. This supports our hypothesis that high valent Ni destabilizes lattice oxygen. The methodology developed in this work is applicable not only to Li-ion battery cathodes but also to post Li-ion batteries such as Na-ion, K-ion, Mg-ion and Ca-ion battery cathodes.

2:30 PM

(ICACC-S6-034-2024) Recent Results and Progress with Multivalent Batteries (Invited)

M. Fichtner*[1]

1. Helmholtz-Institute Ulm (HIU), Solid State Chemistry, Germany

Reversible batteries based on the shuttling of multivalent ions such as Mg, Ca and Al offer the potential to provide both high volumetric energy densities and a high degree of sustainability due to the abundant and non-toxic raw materials used. The fact that metallic anodes can be used is a conceptual advantage of these systems, as this allows for high storage capacities. The talk will address recent results on Mg and Ca batteries, which show that stable systems with high cyclic stability can be achieved by selective control of the interfaces and the storage materials, allowing good ion mobility of the shuttle ion. Therefore, it may be necessary to use tailored organic or organometallic insertion cathode materials, since oxide systems suffer from the low ion mobility of divalent ions in the host.

S7 18th Intl Symp on Functional Nanomaterials & Thin Films for Sustainable Energy Harvesting

SYMPOSIUM 7: 18th International Symposium on Functional Nanomaterials and Thin Films for Sustainable Energy, Environmental and Health Applications

Room: Coquina B
Session Chair: Germán Salazar Alvarez, Uppsala University

1:30 PM

(ICACC-S7-013-2024) Topochemical fluorination of indate-based Ruddlesden-Popper-type oxide, structural-optical characterizations, and photocatalytic hydrogen evolution

S. Perween*[1]; K. Wissel[1]; Z. Dallos[7]; M. Weiss[2]; Y. Ikeda[1]; S. Vasala[3]; S. Strobel[4]; P. Schützendübe[5]; P. M. Jeschenko[6]; U. Kolb[7]; R. Marschall[2]; B. Grabowski[1]; P. Glatzel[3]; O. Clemens[1]

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2. University of Bayreuth, Department of Chemistry, Germany
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4. University of Stuttgart, Institute of Inorganic Chemistry, Germany
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6. Max Planck Institute for Medical Research, Heidelberg, Germany
7. Technical University of Darmstadt, Institute for Applied Geosciences, Germany

Hydrogen (H_2) generated by photocatalytic water splitting can provide an alternative to non-renewable fuels to increase sustainable energy use. There is a growing interest in the exploitation of wide bandgap oxide semiconductors with the ability to use sunlight to bring about photocatalytic reactions such as the generation of hydrogen from water or hydrocarbons. Amongst the materials reported, Ruddlesden–Popper–type (RP) materials have attracted particular interest because of their superior photocatalytic activity, visible-light response, and unique optical and electronic properties. In the present work, we report a non-oxidative topochemical fluorination of novel indate-based RP-type oxide materials, $LaBaInO_4$ to $LaBaInO_3F_2$ which shows a strong expansion perpendicular to the perovskite layers, accompanied by a strong tilting of the octahedra in the ab plane. The structural impact on the optical properties has been studied. Both the compounds, oxide as well oxyfluoride, show photocatalytically active for H_2 evolution. Additionally, we provide the ab initio density functional theory calculations in support of the structural-optical properties obtained experimentally, and therefore we concluded that the fluorination of perovskite-related indate materials could be used to design novel photocatalysts for solar energy harvesting.

1:50 PM

(ICACC-S7-014-2024) Enhancing Photocatalytic Performance of Silicon Photocathode Using MoS_2-Based Cocatalysts

Y. Sung*[1]; J. Ting[2]

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

Due to its abundance and suitable band gap, silicon (Si) is a desirable photocathode for hydrogen evolution reaction (HER). However, its HER kinetics and charge recombination issue required further attention. To overcome these limitations, we fabricate Si photocathode having a molybdenum sulfide (MoS_2)-based cocatalyst to form a heterostructured catalyst. The heterostructure is synthesized using a hydrothermal method. The obtained samples are examined for the material characteristics using SEM, TEM, XRD and UV-vis. The electrochemical performance is evaluated via linear sweep voltammetry (LSV) and electrochemical impedance spectroscopy (EIS), both under light illumination and in the dark, and photocurrent measurement. The effect of the cocatalysts is discussed and addressed.

2:10 PM

(ICACC-S7-015-2024) Polymer-derived SiOC ceramics: A potential catalyst support controlled by the sintering temperature and carbon content

J. Liu*[1]; D. Giuntini[1]; R. Riedel[2]

1. Eindhoven University of Technology, Mechanical Engineering, Netherlands
2. TU Darmstadt, Materials Science, Germany

The hydrogen (H_2), as the ultimate clean energy carrier with extremely high energy density, can be produced by electrochemical water splitting. However, the water splitting technology still faces some practical challenges. In addition to high production costs, the water splitting catalysts result in the limited catalytic activity and energy conversion efficiency as well as stability. In this work, a series of silicon oxycarbide (SiOC) ceramics with varying carbon content from ca. 10 wt.% to ca. 40 wt.% were prepared by thermal pyrolysis of four commercially available polysiloxanes and subsequent spark plasma sintering (SPS) at 1200 °C, 1400 °C, and 1600 °C. The results showed that the high carbon content led to a porous microstructure, and for SiOC with ca. 40 wt. % carbon content, its porosity and specific surface area at 1600 °C reached 34 % and 262 m^2/g, respectively. The electrochemical behavior of materials was evaluated. It was shown that SiOC has a certain degree of electrocatalytic activity, and the sample with 10 wt.% carbon content obtained at 1200 °C exhibited an overpotential of 450 mV vs. RHE at 10 mA/cm^2 in acid medium. These results provide platform for developing transition metal-modified SiOC catalysts towards the H_2 evolution reaction (HER) and for expanding polymer-derived ceramics (PDCs) to a wider range of functional applications.

2:30 PM

(ICACC-S7-016-2024) Multi-Element MOF for Photo-Fenton Process

J. P. Budianto*[1]; J. Ting[1]

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

Photo-Fenton (PF) is an advanced oxidation process that utilizes light-induced generation of highly reactive hydroxyl radicals to effectively degrades pollutant in wastewater. In this study, we have used multi-element MOF as a catalyst in PF. The multi element MOF is consider as a promising catalyst due to the incorporation of multiple transition metal ions in the MOF lattice, creating a highly active and synergistic catalytic environment. The multi element MOF was synthesized using a co-precipitation method. To evaluate the performance of multi element MOF, degradation test of methylene blue is conducted under solar simulator lamp and 98.32% of methylene blue was removed after 60 min. This work provides an overview of the synthesis and characterization of multi element MOF catalyst and an insight of multi element MOF can be used to degrade pollutant in PF process.

S8 18th Intl Symp on APMT for Structural & Multifunctional Materials & Systems

SYMPOSIUM 8: Design-oriented manufacturing and processing

Room: Coquina F
Session Chair: Motoyuki Iijima, Yokohama National University

1:30 PM

(ICACC-S8-034-2024) Control of orientation direction 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. At the same time, this indicates that the orientated direction cannot be actively controlled. We have developed a method for controlling magnetic field direction using ion doping was developed. In this study, the effect of substitution with rare earth ions on the orientation direction was investigated for hydroxyapatite(HAp), tungsten bronze structure $(SrCa)_2NaNb_5O_{15}$ (SCNN), and zeolite. Among them, in HAp and SCNN, substitution with rare earth element Eu was effective for c-axis orientation. Until then, orientation could only be achieved using a rotating magnetic field, but ion exchange has made it possible to align the c-axis in a static magnetic field. It was also found that the response in a magnetic field and sinterability were affected. Regarding zeolites, we are investigating not only the effects of rare earths but also the effects of transition elements. It is also effective in lowering the magnetic flux density, and high orientation at 2T is also possible.

2:00 PM

(ICACC-S8-035-2024) Designing novel dielectric composites with high thermal conductivity via cold sintering

J. Mena Garcia*[1]; A. Ndayishimiye[1]; Z. Fan[1]; M. Mervosh[1]; S. Perini[1]; W. Li[1]; B. Poudel[1]; S. Priya[1]; B. Foley[2]; J. Gaskins[2]; C. Randall[1]

1. The Pennsylvania State University, USA
2. Laser Thermal, USA

In order to fulfill the demands of more bandwidth in 5G and 6G communication technology, new dielectric substrates that can be cofired into packages and devices that have low dielectric loss and improved thermal conductivity are desired. The motivation for this study is the design of composites with low dielectric loss (tan δ) and high thermal conductivity (κ), while still limiting the electrical conductivity, for microwave applications involving high power and high frequency. This work describes the fabrication of high density electroceramic composites with a model system dielectric material for Cold Sintering, namely Sodium Molybdate $Na_2Mo_2O_7$ (NMO) and a filler with higher thermal conductivity such as hexagonal Boron Nitride (hBN). Physical properties of the composites were characterized as a function of filler vol.%, temperature and frequency. Understanding of the variation in measured properties is achieved through analyzing the respective transport mechanisms.

S9 Porous Ceramics Novel Developments and Applications

SYMPOSIUM 9: Engineered Porous Architectures Enabled by Additive Manufacturing Technologies

Room: Coquina D
Session Chairs: Kaline Furlan, Hamburg University of Technology; Paolo Colombo, University of Padova

1:30 PM

(ICACC-S9-009-2024) Highly porous 70S30C bioglass scaffolds from additive manufacturing of novel silicone-based emulsions

V. Diamanti[1]; H. Elsayed[1]; F. M. Stabile[2]; E. Bernardo*[1]

1. University of Padova, Department of Industrial Engineering, Italy
2. Univesidad Nacional de La Plata, Department of Chemical Engineering, Argentina

70S30C (70 mol% SiO2, 30 % CaO) bioglass is one of the most promising results of sol-gel processing of ceramics for bone tissue engineering. We discuss the feasibility of this material directly from silicone-based blends, used as feedstock for additive manufacturing of highly porous scaffolds (porosity >80 %). These scaffolds are first shaped, by vat photopolymerization or direct ink writing, and later ceramized by firing at only 700 °C, in flowing nitrogen or in air. A uniform SiO2/CaO distribution is achieved according to an emulsification step: droplets of molten hydrated calcium nitrate or of concentrated calcium nitrate solutions, in water, are homogeneously dispersed in blends of H44 commercial polysiloxane and photocurable acrylate resin, with the help of surfactants. Vat photopolymerization is adopted for scaffolds with complex geometry, offering a distrinctive control of the porosity (practically identical to that of reference geometrical models). Photopolymerization is exploited also for direct ink writing, as a way to consolidate reticulated scaffolds, soon after extrusion, based on water-containing blends. The latter component is useful as 'templating' agent, since it favours the obtainament of spongy struts. In all cases, the obtained scaffolds feature a remarkable strength-to-density; preliminary cell tests confirm both biocompatibility and bioactivity.

1:50 PM

(ICACC-S9-010-2024) Tailored macroscopic periodic cellular structures with optimized microstructure for tissue engineering

S. Simon*[1]; E. Wolf[1]; M. Weichelt[1]; T. Fey[1]

1. Friedrich-Alexander University Erlangen-Nürnberg, Department Material Science and Engineering, Germany

Tuning the classical replica technique for ceramics with additive manufacturing of polymers is a recently developed method for the production of advanced cellular ceramics with periodic structure. Exploiting the advantages of a simple and low-cost template creation process with structural flexibility is a promising approach to the design and fabrication of scaffolds for bone tissue engineering. As there is demand on tailored periodic structures for the biomaterial sector, we are investigating three different types of biomaterials: hydroxyapatite, Bioglass 45S5® and BCZT. Periodic cellular Kelvin cells were fabricated with different macroporosities and microstructures optimized for bioactivity. In addition to porosity, their compressive strength and cell viability tests are the main properties that were investigated. This research demonstrates that this advanced fabrication process, which allows tuning of material, porosity, structure, strength, electrical response and bioactivity, has great potential for manufacturing highly complex and patient-specific bone replacement materials.

2:10 PM

(ICACC-S9-011-2024) Digital light processing of MOFs functionalized mullite complex architectures for CO_2 capturing systems

A. Bertero*[2]; B. Coppola[2]; J. Schmitt[3]; N. Tanchoux[3]; P. Trens[3]; H. Kaper[1]; P. Palmero[2]; T. Jean Marc[2]

1. UMR 3080 CNRS/Saint-Gobain Research Provence, Laboratoire de Synthèse et Fonctionnalisation des Céramiques, France
2. Politecnico di Torino, Applied Science and Technology, Italy
3. UMR 5253 Pôle Chimie Balard Recherche, Institut Charles Gerhardt, France

Carbon capture and storage (CCS) is a valuable strategy to limit global warming. The current study develops a novel CCS system, based on mullite substrates fabricated by Digital Light Processing (DLP) functionalized with Metal Organic Frameworks (MOFs). Among porous ceramics, mullite ($3Al_2O_3$ $2SiO_2$) was selected as an ideal refractory material. As geometry plays a crucial role on the gas flow properties and CO_2 capture efficiency, DLP was selected to shape complex interconnected ceramic architectures with high resolution and dimensional precision. Mullite architectures based on triply periodic minimal surface (TPMS) frameworks with accurately designed macroporosity and gas permeability were realized. The tailored microporosity of mullite TPMS allows a controlled MOFs growth on their surface, conferring selective CO_2 adsorption properties. MOFs exhibit a robust 3D structure, high specific surface area and good interaction with guest molecules, showing a great potential in several gas separation processes. CO_2 adsorption properties of the functionalized TPMS monoliths were tested: samples showed an interesting CO_2 adsorption capacity, the regeneration efficiency leads to reusable systems. This study demonstrates how the combination between ceramics additive manufacturing and MOFs technology could set the stage for the manufacturing of efficient CCS systems.

S8 18th Intl Symp on APMT for Structural & Multifunctional Materials & Systems

SYMPOSIUM 8: Green manufacturing, global environmental issues and standards

Room: Coquina F
Session Chair: Surojit Gupta, University of North Dakota

2:20 PM

(ICACC-S8-036-2024) Bubble formation in ice crystals by pulsed electron beam irradiation for remote sensing on Europa

H. Suematsu*[1]; H. Ito[1]; T. Kikuchi[1]; G. Imada[2]; T. Do[1]; T. Nakayama[1]

1. Nagaoka University of Technology, Extreme Energy-Density Research Institute, Japan
2. Niigata Institute of Technology, Department of Engineering, Japan

Europa is known to be covered by ice and NaCl which are exposed to electron beam irradiation in the magnetosphere surrounding Jupitar. Radiation effect of NaCl to form color center has been studied and is the reason of the brown color. On the other hand, electron irradiation to ice can cause vacancy clusters with size of 3-35 nm. However, there are no results by pulsed electron irradiation to simulate the electron burst environment on Europa. In this study, ice crystals were irradiated at room temperature by a pulsed electron beam accelerator (ETIGO-III) with a peak energy of 4 MeV and a peak current of 5 kA for 100 ns. After one shot of irradiation, bubbles with an average size of 182 μm were detected. From the comparison between the bubble and the vacancy densities, the bubbles were likely to be formed by the diffusing and clustering of vacancies.

2:40 PM

(ICACC-S8-037-2024) The oxidation of Zircaloy-4 in cesium compounds and steam at 800-1100°C

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2. Nagaoka University of Technology, Japan

During severe accidents in light water reactors, radioactive cesium isotopes will be formed and released from fuel to the reactor coolant system. The form of cesium could be cesium hydroxyl (CsOH) or cesium molybdate (Cs_2MoO_4) [1, 2]. At certain temperatures, both CsOH and Cs_2MoO_4 exist in the gas phase and are diffused into zirconium oxide (ZrO_2). The reaction between CsOH and ZrO_2 may happen, forming zirconate compounds, such as CsZrO3 [1], however, the behavior of Cs_2MoO_4 during the oxidation of zircaloy has not been clearly understood. Moreover, both CsOH and Cs_2MoO_4 may be released through zirconium oxide before the cladding failure. The aims of this study are to investigate the effect of cesium compounds on the oxidation process of zircaloy, and the possible chemical reactions between Cs compounds with zircaloy at high temperature, for improving safety measures and emergency response procedures. The results show that the oxidation rate of zircaloy in the gaseous CsOH environment was faster than that in the crystallite Cs_2MoO_4 at 800°C. Besides, the oxidation depths at temperatures higher than 1000°C in gaseous Cs_2MoO_4 were shorter than that in steam. The decomposition of Cs_2MoO_4 may have already taken place, forming Cs_2O and MoO_3. Under steam condition, the formation of redox pairs MoO_2/MoO_3 and Mo/MoO_2 allowed the zircaloy cladding to be maintained longer than under normal steam oxidation conditions.

S9 Porous Ceramics Novel Developments and Applications

SYMPOSIUM 9: Computational Techniques in Porous ceramics

Room: Coquina D

Session Chairs: Swantje Simon, Friedrich-Alexander-Universität Erlangen-Nürnberg; Ulf Betke, Otto-von-Guericke-University Magdeburg

2:30 PM

(ICACC-S9-012-2024) Using machine learning to classify pore types in ceramic replica foams

J. Dreyer[1]; T. Fey*[1]

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Porous ceramics are promising materials for the energy transition, for use in lightweight structures, as support materials for catalysts or heat exchangers. The surface to volume ratio gives them an advantage over dense materials. The most commonly used industrial process is the replication process based on heterogeneous polymer foams or templates. Burning out the struts leaves hollow struts with the typical triangular structure of polymer foams. Using µCT measurements, the microstructure can be displayed as a stack of 2D cross-sectional images, which are used for evaluation. However, current software cannot separate the different types of pores. Using machine learning, a neural network based on the Eff-Net architecture is employed. The necessary training network is generated by Liner. AI software using 2,800 training images of a 30ppi Al_2O_3 foam with up to 10,000 training iterations and data augmentation. The recognition accuracy for the strut pores was over 87%, while that for the foam pores was 99%. This is then used in the next stage of the Tensorflow-based neural network to identify the strut pores in arbitrary ceramic replica foams. Here, 1200 images of another ceramic foam and a 30ppi PU foam are analysed. The network identified 16.86% material, 81.91% foam pores and 1.23% strut pores for the ceramic. For the PU foam only material (3.9%) and foam pores (96.1%) were identified.

2:50 PM

(ICACC-S9-013-2024) Examining porosity distribution in hydroxyapatite-zirconia fiber bioceramic composite via a multimodal nanoscale-to-microscale 3D tomography analysis

M. Slama*[1]; M. Krols[1]; D. Drdlik[2]; J. Cihlar[2]

1. TESCAN Group, Czechia
2. Ceitec, Czechia

Balancing material composition and porosity is crucial for mechanical properties and bioactivity. In our previous work, conducted with CEITEC and BUT in Brno, Czech Republic, hydroxyapatite was reinforced with ZrO_2 fibers to enhance the material's fracture toughness. The composite material was prepared using the electrophoretic deposition method (EPD). The EPD process allows easy adjustment, leading to deposits with varying levels of porosity. Therefore, conventional methods like Archimedes are insufficient as they only provide information about total porosity values, lacking size distribution information. 3D tomography methods, particularly microCT (X-Ray Microscopy), stand out for non-destructive, high-precision porosity size distribution analysis. The limitation of such methods is resolution. To determine porosity levels on a nanometer scale, other methods such as FIB-SEM tomography are more suitable. Nevertheless, 3D FIB-SEM tomography is limited in the analyzed volume and by the maximum size of detected porosity, and it is a destructive method. Through a combination of these 3D tomography techniques, we can evaluate the overall distribution of porosity in a multiscale, from the microscale to the nanoscale. The multimodal characterization approach combines multiscale information and will provide us with unique data.

3:30 PM

(ICACC-S9-014-2024) Artificial microstructure reconstruction algorithm and stiffness property estimation of ceramic foam

V. Deshpande*[1]; R. Piat[1]

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Numerical studies to determine structure-property relationships of ceramic foam require a statistically significant number of microstructure samples. Manufacturing and micro-CT scanning of such samples is a costly and tedious process. An alternate route is to generate the samples numerically through reconstruction algorithms. This work describes a novel reconstruction algorithm that generates equivalent microstructures with the same statistical correlation functions as the real ones obtained from CT scanning. The microstructure of the studied material looks like polydisperse pores interconnected to each other. Based on Young-Torquato method, this algorithm creates a random distribution of pores as an initial microstructure and in each iteration, it is perturbed by randomly changing the position of pores till convergence is achieved. Further, effective stiffness coefficients of the reconstructed and the real samples are calculated numerically and compared with experimental measurements. The deviation of stiffness coefficients in tension and in shear for reconstructed samples as compared to the real ones is 1.8 % and 5.2% respectively. The same as compared to measurements is 10 % and 20 % respectively. The results show that reconstruction algorithm produces microstructures that are not only statistically equivalent to the real ones but also have realistic stiffness properties.

S15 8th International Symposium on Additive Manufacturing and 3-D Printing Technologies

SYMPOSIUM 15: Direct Writing and Multi-Materials II

Room: Coquina H
Session Chair: Tassilo Moritz, Fraunhofer IKTS

3:20 PM

(ICACC-S15-043-2024) Matrix First: Additive manufacturing for complex 3D ceramic matrix composite structures (Invited)

S. Bottacin[1]; M. Pelanconi[1]; G. Bianchi[1]; A. Rosa[1]; A. Ortona*[1]

1. SUPSI, Department of Innovative Technologies, Switzerland

This work focuses on the computational design and additive manufacturing of complex ceramic matrix composite structures, reinforced with continuous fibres, placed in any direction and volume fraction. Depending on the chosen matrix material, different AM techniques like PBF, BJ and SLA can be used to produce it. Fibre bundles are then sticked into the cavities of the matrix. The printed and thermally treated samples are highly complex architectures like TPMS which can subsequently be densified by PIP, CVI, RMI. They can be used where lightweight, high temperatures components are needed. Samples with different fibre volume fractions, were produced and characterized, results validate the concept, but further work needs to be done in order to optimize their performances.

3:50 PM

(ICACC-S15-044-2024) Direct ink write and weatherability of Lunar highlands regolith simulants inks

A. Marnot*[1]; B. Brettmann[1]

1. Georgia Institute of Technology, Chemical and Biomolecular Engineering, USA

Using Direct-ink-write (DIW) 3D printing in conjunction with Lunar regolith simulants is of particular interest to promote ISRU in the upcoming Artemis missions. The wide formulation design parameters of DIW inks make this 3D printing method highly customizable for the unique environmental demands of the Lunar surface. However, there is a lack of guidelines and best practices in formulation design for these unique environments. In our work we have elected to utilize UV-curing solidification post-extrusion to facilitate operation in extreme cold and vacuum. Inks are composed of high loadings (60 vol%) of solid particles suspended in a photopolymer binder and are extruded and cured at -30°C. The evolution of porosity, tracked across the prints and at the binder-particle interface, and tensile strength are evaluated following thermal cycling between Lunar day/night temperatures. This work aims to further guide ink formulation design for both new materials and new challenging environments.

4:10 PM

(ICACC-S15-045-2024) Hybrid additive manufacturing for the fabrication of freeform ceramics

A. De Marzi*[1]; K. Huang[1]; P. Colombo[1]; G. Franchin[1]

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A common drawback associated with most AM approaches rises from the layer-by-layer construction: printing of complex geometries possessing overhangs or small features requires the addition of support structures, excess or sacrificial material in order to avoid structural collapse. Moreover, the piling up of layers results in the presence of multiple interlayer interfaces and a stair-stepping effect on the surface of the part, leading to reduced strength and mechanical response of the printed parts. The combination of multiple manufacturing techniques into a unique hybrid system could represent a novel solution to overcome the aforementioned limitations. Here, we present a hybrid extrusion-photopolymerization process (UV-DIW) in which a photocurable suspension is extruded through a nozzle and consequently cured by an external UV source. The full potential of the UV-DIW setup will be demonstrated by coupling it with a 6-axis robotic arm and fabricating complex, freeform shapes using preceramic precursors as well as suspensions of transparent and dark ceramic particles. Specifically, polysiloxane-, silica- and silicon nitride-based inks were prepared and used to produce different lattice structures; ink reactivity, particles surface properties and liquid-particles interaction forces were found to be the most critical parameters to control the inks' ceramic yield, flow behavior and freeform abilitability.

4:30 PM

(ICACC-S15-046-2024) Water-based additive manufacturing of ceramics by Laser-Induced Slip Casting (LIS)

A. Zocca*[1]; H. Schubert[1]; T. Mühler[2]; J. Guenster[1]

1. BAM Bundesanstalt für Materialforschung und -prüfung, Advanced Multi-materials Processing, Germany
2. QEP3D GmbH, Germany

The Laser-Induced Slip Casting is an additive manufacturing technology specifically developed for ceramic materials using water-based ceramic slurries. The process takes place layer-by-layer in a similar fashion as top-down vat photopolymerization, selectively consolidating each layer by means of a laser energy source positioned on the top. Contrary to vat photopolymerization, in which the consolidation is achieved by selectively cross-linking a ceramic-filled resin, LIS uses water-based slurries with a low amount of organic additives (typically < 5 wt%) as feedstocks. In LIS, a green body is formed by local evaporation of water which causes the suspension to collapse forming a cast, following a mechanism similar to slip casting. Only a small content of organic additives is needed to effectively disperse the ceramic particles and to increase the green strength. The technology is very versatile and can be applied to all ceramic systems that can be dispersed in water. One of the main advantages is that even dark materials such as silicon carbide can be processed without issues related to light scattering and absorption. The presentation will discuss strengths and limitations of LIS compared to other AM technologies and will highlight the latest results for alumina and for silicon carbide ceramics.

4:50 PM

(ICACC-S15-047-2024) Exploring the Potential: Wireless Embedded Ceramic Sensors using Additive Manufacturing

N. Reed*[1]; J. B. Shivakumar[2]; S. C. Perry[3]; K. Coote[1]; E. Rojas-Nastrucci[2]; D. Kim[1]

1. Embry-Riddle Aeronautical University, Aerospace Engineering, USA
2. Embry-Riddle Aeronautical University, Electrical Engineering and Computer Science, USA
3. Embry Riddle Aeronautical University, Mechanical Engineering, USA

Additive manufacturing (AM) has enabled and accelerated the development of structural components with embedded sensors. The unique AM process allows for extensive access to the internal structure of complex parts, something unattainable with traditional manufacturing methods. However, despite these advances, creating ceramic structures with embedded sensors remains a significant challenge. The advantageous temperature resistance and stiffness of ceramic parts present distinct challenges when attempting to process a structure to incorporate an embedded sensor. In this work, we explore the development of ceramic structures embedded with additively manufactured sensors to accurately measure the internal properties of the substrate at elevated temperatures. Utilizing the fused deposition modeling (FDM) process, a wireless ceramic sensor is printed, which is layered with additional sensing elements and an antenna. After the sensor is completely encased and fired, the finished sensor is added to a digital light processing (DLP) ceramic substrate during the fabrication process. This integration, once cured, fully encases and bonds the sensor to the substrate, creating a unified structure. After post-processing, the completed sample can perform wireless sensing while retaining the beneficial thermal and mechanical properties of the underlying ceramic material.

S6 Advanced Materials and Technologies for Rechargeable Energy Storage

SYMPOSIUM 6: All-solid-state batteries VI

Room: Ballroom 5
Session Chairs: Valerie Pralong, CNRS ENSICAEN;
Rick Laine, University of Michigan

3:20 PM

(ICACC-S6-035-2024) Operando X-ray CT Analysis on All-solid-state Batteries (Invited)

Y. Orikasa*[1]; M. Matsumoto[1]; Y. Sakka[1]; C. Zhong[1]; H. Yamashige[2]

1. Ritsumeikan University, Department of Applied Chemistry, Japan
2. Toyota Motor Corporation, Japan

All-solid-state batteries (ASSB) have advantages for safety and rate capability because of nonflammability and high transport number of carrier ion. Solid electrolytes might mechanically surpress dendrite formation. However, the hardness of solid electrolytes disturbs contact between solid electrode active materials and solid electrolytes during charge–discharge. In addition, voids in electrodes and electrolytes of ASSB do not contribute the electrochemical reaction. To improve the practical performance of ASSB, morphological structure including the contact state should be optimized. X-ray computed tomography (CT) is one of powerful tool to analyze morphology of ASSB electrodes and electrolytes. In this presentation, we introduce the resent results from X-ray CT analysis of ASSB. The porosity of the composite electrode and contacted state of the active materials shows pressure dependency, which influences charge–discharge capacity. In addition, the mechanical contact between silicon particle and solid electrolyte during charge-discharge have been tracked by operando X-ray CT measurements.

3:50 PM

(ICACC-S6-036-2024) Low-temperature sintering and in situ formed protective layer for oxide-based composite cathode in all-solid-state Li batteries (Invited)

C. Lin[1]; M. Ihrig[2]; K. Kung[1]; H. Chen[1]; O. Guillon[2]; S. Lin*[1]

1. National Cheng Kung University, Materials Science and Engineering, Taiwan
2. Forschungszentrum Juelich, IEK-1, Germany

Oxide-based all-solid-state Li battery (ASSLiB) with sufficient ionic conductivity and good chemical and electrochemical stability is a promising system for ASSLiB development. Problems in oxide-based composite cathodes are often caused by cathode thermal stability, contact between particles, and unwanted interfacial reactions. Most of the cathode materials cannot withstand the conventional sintering temperature of oxide electrolytes (>1000 °C). Lack of contact between particles and formation of low Li-ion conductivity interphases lead to high interfacial impedance in composite cathodes. To solve the problems, a low-temperature sintering technique and a design of in situ forming protective layer were proposed in this work. Low-temperature sintering was carried out through field-assisted sintering technology/spark plasma sintering (FAST/SPS). Interfacial reaction which provides driving force for chemical bond formation was induced through doping in cathode. By selecting suitable dopant, the interfacial reaction product acts as a protective layer preventing further interfacial reactions between cathode and electrolyte during sintering and cycling. The concept and the method provide an efficient way of solving interfacial problems in ASSLiB composite cathode, and the material design procedure could be promoted to other composite cathode systems.

4:20 PM

(ICACC-S6-037-2024) Laser-induced melting of tin-iron-soda-silicate glass on solid electrolyte

F. Sato*[2]; T. Honma[1]

1. Nagaoka Univeristy of Technology, Department of Materials Science and Bioengineering, Japan
2. Nagaoka University of Technology, Doctoral Program in Engineering, Japan

Oxide-based all-solid-state sodium batteries (ASSBs) are expected to be next-generation batteries. However, the high interface resistance between the electrode and solid electrolyte is one of the barriers to the practical use of ASSBs. Transition metal ions in glass have strong absorption in the UV to IR wavelength region, and optical energy is converted to thermal energy by multi-phonon relaxation. By this mechanism, a temperature field can be induced by laser irradiation of the material, and liquid phase formation and crystallization proceed depending on the temperature range. Laser processing also allows the glassy active material to adhere to the solid electrolyte faster than the typical sintering process. The tin-iron-soda-silicate anode glass $55SnO-15Na_2O-6.75Fe_2O_3-(30-y)SiO_2$ glass powder was mixed with polyimide binder to synthesize the anode paste. The anode paste was applied on a solid electrolyte substrate of about 10 μm thickness by a screen-printing method. The sample was laser irradiated at wavelength 1064 nm, power 3 W. As a result, the anode layer was densified and adhesion with the solid electrolyte. The adhesion of the anode layer and solid electrolyte layer by laser irradiation was the first discovery, which not only solved the problem of mismatch in processing temperatures, which cannot be achieved by conventional heating in furnace, but also enabled high-speed processing.

4:40 PM

(ICACC-S6-038-2024) A New, Energy-Saving Precursor Route for Synthesizing $Li_7La_3Zr_2O_{12}$ & $LiCoO_2$ Mixed Cathode for Solid State Batteries

V. Kiyek*[1]; C. Schwab[1]; M. Finsterbusch[1]; D. Fattakhova-Rohlfing[1]; O. Guillon[1]

1. Forschungszentrum Juelich, IEK-1, Germany

The demand for safe, high energy density batteries has increased recently, while all-solid-state batteries (ASSB) are one of the major battery systems discussed. The high energy density can be achieved by using metallic Li as anode. Safety can be guaranteed by implementing an oxide ceramic electrolyte. $Li_7La_3Zr_2O_{12}$ (LLZO) is one of the most discussed solid electrolytes and chemically stable against Li metal. Among the most significant challenges in this regard is the high energy required for the synthesis and processing of ceramics, due to several high temperature steps over 1000°C for several hours. In this study, we propose a single heat treatment for processing functional mixed cathodes including the formation of LLZO from precursors and the usage of $LiCoO_2$ (LCO) as active material. The proposed method relies on a tape casting process of a slurry containing LLZO precursors and LCO powders. During heating, LLZO phase is formed in-situ, avoiding the need for separate synthesis (involving a long calcination step) of LLZO powder prior to shaping and sintering. Our results show that this strategy is highly effective in producing high-quality mixed cathodes, achieving a high relative density of up to 95% and a level of performance comparable or even better to that of conventional methods already tested for the manufacturing of ASSBs.

S13 Development & Applications of Adv Ceramics & Composites for Nuclear Fission/Fusion Energy Sys

SYMPOSIUM 13: Material technologies for accident tolerant fuel cladding and core structures for light water reactors

Room: Ballroom 4
Session Chair: Gyanender Singh, Idaho National Laboratory

3:20 PM

(ICACC-S13-038-2024) Improving the coolant compatibility of SiC/SiC composites for light water reactors via the design of multilayered protective coatings (Invited)

S. Huang*[1]; N. Goossens[1]; S. Mráz[2]; J. M. Schneider[2]; C. Sauder[3]; M. K. Grosse[4]; M. Steinbrück[4]; G. Greaves[5]; J. A. Hinks[5]; J. Vleugels[1]; K. Lambrinou[5]

1. KU Leuven, Materials engineering, Belgium
2. RWTH Aachen University, Materials Chemistry, Germany
3. CEA, DRMP, France
4. Karlsruhe Institute of Technology, Institute for Applied Materials, Germany
5. University of Huddersfield, School of Computing and Engineering, United Kingdom

The prospective use of SiC/SiC composite accident-tolerant fuel (ATF) claddings in light water reactors is hindered by the mediocre compatibility of SiC with water (nominal operation) and steam (accident scenarios). SiC losses in water have been attributed to the formation of water-soluble species (e.g., SiO_2), whilst processing-induced SiC defects such as stacking faults & twins have been identified as inherent weaknesses that accelerate its deterioration in hydrogenated water. Moreover, the formation of SiO_2 scales on the SiC surface in steam endangers the reliable use of SiC/SiC composites above the melting point of SiO_2 (1720°C for β-cristobalite) due to the reaction between molten SiO_2 and SiC that produces gaseous species (SiO & CO). The HORIZON SCORPION project targets the improvement of the SiC compatibility with water and steam via the design and testing of multilayered coating systems. A candidate coating material with promising compatibility with both water and steam is ytterbium disilicate ($Yb_2Si_2O_7$). This work reports on the fabrication of phase-pure $Yb_2Si_2O_7$ via pressure-assisted densification of oxide powders in a spark plasma sintering (SPS) facility. The $Yb_2Si_2O_7$ ceramics were tested in flowing PWR water (360°C, 187 bar; 1000 ppm B, 2 ppm Li) & steam (1200°C, 1 h), and were in-situ ion-irradiated with 600 keV Kr^{++} at 350-1200°C.

3:50 PM

(ICACC-S13-039-2024) Nuclear Uses of SiC-SiC CMCs and Graphite: Design & Construction Rules in ASME BPV Code Sec, III, Div. 5 for Nonmetallics - Revisons of 2023 Edition

M. G. Jenkins*[1]; S. T. Gonczy[2]; J. W. Geringer[3]; Y. Katoh[4]

1. Bothell Engineering and Science Technologies, USA
2. Gateway Materials Technology, USA
3. Oak Ridge National Lab, Materials Science and Technology, USA
4. Oak Ridge National Laboratory, USA

US DOE is supporting 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 possible revisions of 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.

4:10 PM

(ICACC-S13-040-2024) Development strategy for SiC/SiC composite accident tolerant fuel cladding

T. Koyanagi*[1]; Y. Katoh[1]

1. Oak Ridge National Laboratory, USA

SiC fiber–reinforced SiC matrix (SiC/SiC) composite cladding technology is an engineered cladding designed to withstand the severe thermomechanical and chemical environment in light-water reactors (LWRs). Deployment of SiC/SiC in LWRs could offer enhanced performance in design and beyond design basis accidents, thereby removing economic restrictions compared with existing fuel cladding concepts. This paper outlines the SiC-based cladding development strategy. The near-term R&D goal is to advance SiC cladding concepts to a maturity level suitable to support lead test rod (LTR) insertion into a commercial LWRs. The key issues to address through successful demonstrations are (1) hermetic cladding and end plug demonstration, (2) steady-state corrosion resistance/material property issues, and (3) capability to effectively model LTR performance. Demonstration of hermetic cladding under normal operating environments has been identified as one of the most critical feasibility issues. The material property solutions will be delivered numerically and experimentally. This study was supported by the US Department Energy (DOE), Office of Nuclear Energy, for the Advanced Fuels Campaign of the Nuclear Technology R&D program under contact DEAC05-00OR22725 with ORNL, managed by UT Battelle, LLC.

4:30 PM

(ICACC-S13-041-2024) Neutron induced deformation evolution of SiC/SiC composites using X-ray computed tomography testing and digital volume correlation

J. D. Arregui-Mena*[1]; T. Koyanagi[2]; Y. Katoh[2]

1. Oak Ridge National Lab, Nuclear Materials Science & Technology Group, USA
2. Oak Ridge National Laboratory, USA

SiC/SiC composites have emerged as promising materials for fuel cladding in Light Water Reactors (LWRs) and advanced reactor core structures. The effects of thermal stresses, the extreme reactor environment, and neutron-induced dimensional changes on SiC/SiC composites must be understood for their near future deployment and qualification. This study focuses on the quantification of irradiation-induced damages in SiC/SiC composite end-plugs by conducting x-ray computed tomography (XCT) scans before and after neutron irradiation at elevated temperatures. A portion of the research focused on processing the XCT to highlight the microstructural differences caused by neutron irradiation. In addition, the XCT results were processed using digital volume correlation to assess the overall neutron-induced deformation caused by the swelling of the tube, plug, and bonding layer. By combining these results, it is possible to evaluate the compatibility of the plugs and, for the first time, estimate the deformation of a SiC composite tube via 3D characterization. These test methods are required to investigate the damage that affects the hermetic properties and microstructure changes in neutron-irradiated SiC/SiC composites.

S7 18th Intl Symp on Functional Nanomaterials & Thin Films for Sustainable Energy Harvesting

SYMPOSIUM 7: Nanotoxicity, bio-imaging, drug-delivery and tissue engineering with tailored nano-bio conjuguates

Room: Coquina B
Session Chair: Sedat Ballikaya, Istanbul University

3:20 PM

(ICACC-S7-017-2024) Flame-made Calcium Phosphate nanoaggregates with fractal-like morphology as drug nanocarriers for biologics (Invited)

G. Sotiriou*[1]

1. Karolinska Institutet, Sweden

Biologics (proteins, peptides, nucleotides) dominate the novel therapeutics market. A major obstacle in their employment, however, is their enzymatic degradation in vivo demanding high doses that result in side-effects. One way to avoid degradation of biologics is to encapsulate them in nanocarriers, such as lipid-based nanoparticles. An alternative approach is to use biocompatible inorganic nanoparticles, such as calcium phosphate (CaP). In this study, we aim to improve the drug loading capacities of biologics by utilizing inorganic CaP nanoaggregates made by flame aerosol technology, with fractal-like morphology and high specific surface area. We use flame spray pyrolysis, a technique which allows tuning of NPs properties like composition, size, crystallinity. These parameters are critical as they determine the mode of cellular uptake. We optimize the loading of CaP nanoaggreates with biologics, targeting two distinct applications: as antimicrobial peptide delivery carriers and as adjuvants for vaccination. The performance of the developed particles is benchmarked with the state-of-the-art lipid-based nanoparticles for biologic drug delivery. The flame-made CaP nanoaggrates developed on this study exhibit advantageous properties for their employment in biologics delivery with high loading values and minimal cytotoxicity.

3:50 PM

(ICACC-S7-018-2024) Recent Developments on Inorganic Nanoparticles for In-vivo X-Ray Fluorescence Bioimaging (Invited)

M. S. Toprak*[1]

1. KTH Royal Institute of Technology, Dept. of Applied Physics, Sweden

Nanoparticles (NPs) have found use in many areas from hybrid materials, to sensors, antiviral/antibacterial coatings and various biomedical applications. Each application area has its own set of NP attributes that has to be complied in order to assure their successful implementation. We have been designing different families of ceramic nanoparticles for a diverse set of biomedical applications, using specifically bottom-up solution chemical techniques. Whenever needed the individual NPs were assembled into larger micron-scale architectures by the use coupling mechanisms using their surface functionality. In this respect, it is of utmost importance to have the right surface chemistry to minimize the number of process steps, in order to reach the desired material attributes. In this talk, I will present about some of the recent activities and materials dedicatedly designed for X-Ray fluorescence bioimaging (XRF). Starting from the first generation, to the development of core-shell particles and various surface functionalization schemes. Some of the recent activities using the developed NPs for in-vitro and in-vivo studies will be presented.

S8 18th Intl Symp on APMT for Structural & Multifunctional Materials & Systems

SYMPOSIUM 8: Rapid prototyping, 3D printing, patterning, templates and self-assembly

Room: Coquina F
Session Chair: Surojit Gupta, University of North Dakota

3:20 PM

(ICACC-S8-038-2024) Design of Interparticle Photo-cross-linkable Suspension: Toward Efficient Processing of 3D Structured Ceramic Components (Invited)

M. Iijima*[1]; Y. Yamanoi[1]; J. Tatami[1]

1. Yokohama National University, Japan

3D structuring of ceramics has attracted wide attention toward fabricating components having on demand structures. Photocuring of ceramic suspensions is one of the promising routes to achieve 3D ceramic structures in fine resolution. However, most system suffers from long dewaxing/ sintering due to the large organic contents treated in the suspension to ensure the mechanical properties of photocured bodies. Here, we propose a new series of suspension which can be photo-cured using reduced amounts of monomers by interparticle photo-cross-linking reaction. The proposed suspension design involves the dispersion of reagent particles in solvents assisted by polyamine-based polymer dispersants, followed by the addition of small amounts of multifunctional acrylates and photo-radical initiators. UV light irradiation to the designed suspension results to form a nano-scaled polymer networks among the particles which enables a successful photocuring. We demonstrate that the designed suspension can be applied to various 3D structuring processes, such as vat photopolymerization, in-situ photo-molding, and their combinations with green machining. Owing to the reduced amounts of monomers in the designed suspension, the photo-cured compacts were dry-, dewax-, and sinter-able into dense components using rapid heating profiles without forming any structural collapses.

3:50 PM

(ICACC-S8-039-2024) Hierarchical structuring of ceramic and ceramic-metal hybrid materials via vat photopolymerization of preceramic polymer resins (Invited)

T. Konegger*[1]; J. Eßmeister[1]; A. Fuchsberger[1]; D. Steiner[2]; S. Schwarz[3]; T. Schachinger[3]; A. Lale[4]; M. Schwentenwein[4]; K. Föttinger[2]

1. TU Wien, Institute of Chemical Technologies and Analytics, Austria
2. TU Wien, Institute of Materials Chemistry, Austria
3. TU Wien, University Service Centre for Transmission Electron Microscopy, Austria
4. Lithoz GmbH, Austria

Photopolymerization-based structuring techniques such as vat photopolymerization (VPP) have gained significant importance in additive manufacturing of ceramics, in particular when combined with the polymer precursor route. This contribution highlights the augmentation of established 3D structuring approaches combining VPP and preceramic polymer technology by the concept of polymerization-induced phase separation, thus presenting a highly versatile approach towards simultaneous macro- and microstructuring of polymer-derived ceramics, yielding complex-shaped monoliths with porosity spanning from the sub-micrometer to the millimeter scale. This concept furthermore allows for a straightforward functionalization of the final materials by chemical modification of the preceramic polymer resins with organometallic compounds, which has been successfully demonstrated for the hybridization of SiOC-based materials with Ni, Co, La, or Mo. The resulting complex-shaped, hierarchically porous, functionalized ceramic materials are of particular interest for current and prospective future applications in diverse fields such as chemical conversion, energy

*Denotes Presenter

storage, or separation, which is highlighted by the application of hierarchically porous SiOC monoliths containing in-situ formed Ni nanoparticles as heterogeneous catalysts for CO_2 methanation.

4:20 PM

(ICACC-S8-040-2024) Laser Ablative Patterning of B_4C and MoAlB Ceramics for Hydrophobic Surfaces

B. Cui*[1]; S. Ruiz[1]; Y. Yoo[1]; L. Wadle[1]; X. Chen[1]; N. Li[1]; Y. Lu[1]; C. Wohl[2]; V. L. Wiesner[2]

1. University of Nebraska–Lincoln, USA
2. NASA Langley Research Center, USA

Through a novel laser ablative patterning (LAP) process, the present work reported the increase in wetting contact angle of two ceramic materials, boron carbide (B_4C) and molybdenum aluminum boride (MoAlB). The LAP technique employed picosecond laser pulses to form a crosshatch pattern with micropillars on ceramic surfaces. The wetting behavior of both ceramic surfaces was successfully transitioned from hydrophilic ($\theta < 90°$) to hydrophobic ($\theta > 90°$), which was related to Wenzel and Cassie-Baxter mechanisms. Parameters such as laser fluence and number of laser scans were varied to study their effects on ceramic surface characteristics. Lunar dust adhesion experiments were performed on these hydrophobic ceramic surfaces, which showed that the adhesion of lunar soil simulant dust particles was significantly reduced in the patterned B_4C and MoAlB surfaces.

S16 Geopolymers Inorganic Polymers and Sustainable Construction Materials

SYMPOSOIUM 16: Sustainable materials and novel applications

Room: Coquina C
Session Chair: Ange Therese Akono, North Carolina State University

3:20 PM

(ICACC-S16-026-2024) Investigation of the Effect of Basalt Microspheres on the Wear Resistance of Metakaolin Based Geopolymer Composites

Y. Zubko*[1]; E. Zubko[2]; W. M. Kriven[1]

1. University of Illinois at Urbana-Champaign, USA
2. Micro Basalt Innovations - Canada Corp, Canada

Often, the surfaces of concrete products are subjected to strong abrasive effects from the surrounding environment during usage. The results of studying the impact of such destructive factors on the structural components of hydraulic structures, concrete pipelines, and industrial floors are usually key criteria in material selection, where the properties must ensure the long-term and safe operation of the respective objects. We investigated the influence of microfillers in the form of a mixture of basalt microspheres with diameters ranging from 600 to 1200 microns and chamotte powder, on the abrasive resistance of a composite material based on a geopolymer matrix. The results of comparative testing are presented, involving base samples made from pure geopolymer material, geopolymer material with the addition of chamotte powder, basalt microspheres, and their combination, in accordance with the ASTM C1803-20 standards. It has been established that the addition of basalt microspheres consistently exerts a positive influence on the abrasive resistance of both pure geopolymer and geopolymer composites containing chamotte powder. Numerical results of tests conducted are provided. The data obtained can prove to be useful in the development of new, wear-resistant, composite materials based on geopolymer matrices, infused with basalt microspheres.

3:40 PM

(ICACC-S16-027-2024) Carbon Nano-particles in Concrete; Challenges and Outcome

J. Paul*[1]; A. Kumar[1]

1. University of South Florida, Mechanical Engineering, USA

Presently, the manufacturing sector is growing increasingly concerned with its carbon footprint. The global warming potential of products plays a major role in swaying consumer decisions. One such industry is cement manufacturing which is held responsible for 8% of global carbon dioxide emissions. The need for infrastructure development drives this sector and as a result, it becomes necessary in society. Concrete mix designs are overdesigned and, as a result, contain much more cement than required. Here we discuss multiple dispersion strategies for Carbon nano-particles in concrete and associated challenges in addition to realistic expected outcomes.

4:00 PM

(ICACC-S16-028-2024) Gamma Radiation Attenuating Geopolymer Composites: A Heavyweight Concrete Alternative

A. Fields*[1]; J. Zhou[2]; A. Di Fulvio[2]; W. M. Kriven[1]

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

Geopolymers are hydrated, inorganic aluminosilicate polymers that can be used to bind different dispersant phases to create structural composites. With geopolymers, a wide range of material properties can be achieved through the selection of appropriate filler materials and processing parameters. The incorporation of gamma radiation-attenuating dispersants, specifically dense dispersants to increase the probability of interaction with an electron, would allow for highly versatile radiation shielding solutions. Geopolymer resin is a thixotropic and shear-thinning fluid, meaning that the viscosity can be manipulated as desired. This combination of being able to tune properties such as radiation attenuation, structural strength, and manipulable flow makes for the ideal recipe for radiation shielding in the next generation of nuclear reactors. In this work, the shielding ability of potassium-based geopolymer composites was investigated using gamma radiation. The linear and mass attenuation coefficients for different energies of gamma radiation were measured for different amounts of tungsten powder (32 μm average particle size) dispersed in a $K_2O \bullet Al_2O_3 \bullet 4SiO_2 \bullet 11H_2O$ geopolymer. The addition of tungsten shows great promise in being able to increase the shielding ability of the geopolymer beyond that of a computationally simulated, iron-limonite, high-density concrete.

4:20 PM

(ICACC-S16-029-2024) Polyethylene-Geopolymer Composite for Neutron Radiation Shielding

A. Fields*[1]; J. Zhou[2]; A. Di Fulvio[2]; W. M. Kriven[1]

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

Geopolymers are hydrated, inorganic aluminosilicate polymers that have generated a great deal of interest as a replacement for ordinary Portland cement. As concrete, specifically heavyweight concrete, is ubiquitous in nuclear power plants, exploration of possibly more effective and environmentally friendly alternative structural and radiation shielding materials is relevant to the future production of nuclear reactors. The future of nuclear power generation will also require smaller form factors for the development of modular nuclear reactors, meaning that more compact shielding solutions will be needed. The incorporation of specific neutron-attenuating materials in a geopolymer binder would allow for more efficient neutron shielding solutions. In this work, the shielding ability of potassium-based geopolymer composites was investigated using neutron radiation. Polyethylene beads (~1-2 mm), powder (~400 μm), and/or fibers (1/4″, 32 μm diameter) were incorporated in potassium geopolymer of the composition $K_2O{\bullet}Al_2O_3{\bullet}4SiO_2{\bullet}11H_2O$. These polyethylene-geopolymer composites were used to find the neutron removal cross-section. The removal cross-section of 0.15 cm^{-1} for potassium geopolymer with 33.3 wt% PE powder was found to show a significant improvement over the 0.12 cm^{-1} cross-section found for a computationally simulated, iron-limonite, high-density concrete.

4:40 PM

(ICACC-S16-030-2024) Definition of geopolymer in terms of alkali activated material terminology

W. M. Kriven*[1]

1. University of Illinois at Urbana-Champaign, USA

The term "geopolymer" has different meanings to different scientific communities. In materials science geopolymers are made from kaolinite clay of composition $Al_2O_3{\bullet}2SiO_2{\bullet}2H_2O$ heated at ~750°C/2 hours and converted to amorphous metakaolin. When mixed under high shear with water glass solution (of composition $M_2O{\bullet}2SiO_2{\bullet}11H_2O$), it undergoes dissolution, polycondensation and precipitation to form a geopolymer where all the silicate and aluminate tetrahedral are corner shared. In civil engineering the term "geopolymers" refers to the product resulting from high shear mixing of class F fly ash mixed with ground granulated blast furnace slag waste products. The solid is amorphous or crystalline but based on calcium silicate hydrate (CSH), C(A)SH, KASH, NASH) (charge balanced with Na^+ or K^+) binder phases which form cements. In this structure, silicate or aluminate tetrahedra form layers sharing only two or sometimes three corners, and separated by layers of $Ca(OH)_2$. CSH is the main binder phase in Portland cement. One main difference between the cements versus geopolymers is that geopolymers are chemically stable up to 700-1,000°C, after which they crystallize into ceramic. Thus metakaolin-based geopolymers are made like a cement but can behave like a ceramic, while cements will always remain as low temperature-capable cements.

FS1 Bioinspiration/Green Processing & Related Technologies of Advanced Materials

Focused Session 1: Bioinspiration, Green Processing, and Related Technologies of Advanced Materials

Room: Ponce de Leon
Session Chair: Jinhui Tao, Pacific Northwest National Lab

3:30 PM

(ICACC-FS-004-2024) Design of Bamboo-Skin Inspired Composites by Machine Learning for Tunable Strength and Toughness (Invited)

Z. Qin*[1]

1. Syracuse University, Civil and Environmental Engineering, USA

Bamboo has been widely used in construction for its high strength, lightweight, and low cost. We experimentally characterize the pattern of the distribution of silica particles within the bamboo skin, build coarse-grained models to simulate its fracture in mechanical loading, and investigate the effect of the pattern on fracture through massive simulations. We find that other than the material volume ratio, rigid inclusions' distribution pattern strongly affects the composite's toughness modulus. We find that the distribution pattern in bamboo skin yields a higher toughness modulus than randomly distributed particles and validate our observation by multi-material 3D printed samples. We examined the geometric features of the pattern and found that it is neither random nor fully ordered but we can use Deep Convolutional generative adversarial network (GAN) to learn the features and use it for composite designs. We are thus inspired to apply the knowledge to the design of synthetic composites composed of metal alloys and thermoplastic materials, can be used by regular thermal-plastic printers and recyclable after printing, leading to wide green engineering applications.

4:00 PM

(ICACC-FS-005-2024) Bioinspired Ceramics Inspired by Biological Structural Design Elements (Invited)

S. E. Naleway*[1]; M. Schmitz[1]; I. Elnunu[1]; J. Gallagher[1]

1. University of Utah, Department of Mechanical Engineering, USA

Bioinspired ceramic materials that are able to mimic the structure and properties of biological materials are of interest to a wide range of scientific and engineering fields. When looking to create these bioinspired ceramic materials it can be advantageous to focus on those structural design elements that have been shown to provide significant mechanical advantages by a variety of species in nature. Here we mimic these structural design elements with a variety of advanced ceramic fabrication techniques (with a focus on the application of energized fields) such as freeze casting, aerogel synthesis, and biotemplating. Applications in biomedical materials and advanced ceramic composites will be discussed.

4:30 PM

(ICACC-FS-006-2024) Bioinspired process for making microstructured ceramic-reinforced composites (Invited)

H. Le Ferrand*[1]

1. Nanyang Technological University, Singapore

Highly mineralized biological materials are known for their complex hierarchical organization that endows them with unusual properties and functionalities. In addition, such biological materials are formed under mild and hydrated conditions which contrasts with synthetic processes. To combine the exquisite microstructural control in natural materials with the demands from the industry and market, there is the need to develop alternative processes. As one step toward this goal, we developed a one-pot process that assembles the mineral and organic matter at the same time in a controlled fashion. The process combines magnetically assisted slip casting (MASC) with the in-situ gelation of an interpenetrated polymer network (IPN) matrix whose threads act as sacrificial bonds between the ceramic, dissipating energy during compression. The composites exhibited 200 % higher toughness and with less than 25% reduction on the stiffness. Furthermore, as an ultimate method that emulate the biological growth of materials, 3D printing deposits materials layer-by-layer. By combining direct ink writing and MASC, advanced materials with local microstructure, anisotropic properties and combined mechanical and functional properties can be fabricated.

S9 Porous Ceramics Novel Developments and Applications

SYMPOSIUM 9: Structure and Properties of Porous Ceramics

Room: Coquina D
Session Chairs: Rudolph Olson, CONSOL Innovations; Manuella Cerbelaud, IRCER

3:50 PM

(ICACC-S9-015-2024) Quantitative Evaluation for the Effect of Microstructure in Porous Ceramics Properties (Invited)

S. Honda*[1]; S. Hashimoto[1]; B. Nait-Ali[2]; D. S. Smith[2]; Y. Daiko[1]; Y. Iwamoto[1]

1. Nagoya Institute of Technology, Japan
2. University of Limoges, France

The gas permeation and thermo-mechanical properties of porous alumina used as a support substrate for ceramic membranes were investigated. The porosity, pore size and apparent grain necking size of porous alumina were systematically varied, and the relationships between the porous microstructure and properties were examined. The grain necking size at alumina grain boundaries was evaluated quantitatively. The properties of porous alumina samples with extensive grain necking showed higher values even in samples with the largest pore size. It was demonstrated that a porous structure combining high gas permeability and excellent fracture resistance could be successfully achieved. Porous alumina with a highly textured microstructure was fabricated using alumina platelets. The nitrogen gas permeance and thermal conductivity were higher in the direction along the platelet length due to the higher conectivity of pores and platelets, respectively. The anisotropy of the thermal conductivity was investigated and explained by the effect of grain size of platelets as well as morphology of pores. The thermal shock strength was clearly different in the two directions and it was investigated by the measurement of properties and thermal stress analysis. This highly textured porous alumina can be achieved with both high gas permeability and excellent fracture resistance.

4:20 PM

(ICACC-S9-016-2024) Macro-porous ceramics for the Sustainable Development Goals (SDGs) and the efficiency of reduction of CO_2 emissions

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. Macro-porous ceramics are one of great solutions to contribute the SDGs, because that can cover almost all technological issues to 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. In this presentation, we will report several solutions to achieve the goal, and especially focus on the thermal insulation and their effect on CO_2 reduction.

4:40 PM

(ICACC-S9-025-2024) Factors affecting high-temperature interaction of ceramic aerogels with molten metals

N. Sobczak*[1]; J. Morgiel[1]; J. Sobczak[3]; S. Seal[2]; S. Terlicka[1]; K. Trembecka-Wójciga[1]; A. Kmita[3]; D. Lachowicz[3]; A. Jeyaranjan[2]; G. Rasua[2]

1. Polish Academy of Sciences, Institute of Metallurgy and Materials Science, Poland
2. University of Central Florida, Mat. Sci. Eng, USA
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Information on high-temperature interaction of ceramic aerogels with different molten alloys is of scientific and practical importance for liquid-assisted joining aerogels to dissimilar materials. This study summarizes the results of the real-time observation of high-temperature behavior of oxide-free liquid metals (Me = Al, Mg) in contact with two types of aerogel substrates, i.e. made of silicon oxide nonopowder (SiO_2) or of reduced graphene oxide flakes (rGO). The tests were performed at a temperature of 700°C in non-oxidizing atmosphere (vacuum or pure argon) using the pendant drop method combined with the sessile drop method and purification of the metal drop from the primary oxide film directly at the test temperature. It was found that all examined aerogels exhibit a good adhesion to selected liquid metals, which does not degrade during the solidification of a metal drop and subsequent cooling of the Me/aerogel couples. Structural characterization (SEM, TEM, HRTEM, EDS) allowed to identify different mechanisms of permanent bonding affected by the following main factors: (1) residual oxygen contained in aerogel substrates; (2) type and amount of dopants (CeO_2 or rGO) used for preparation of aerogels; (3) possible redox reactions between liquid metals and aerogels.

S8 18th Intl Symp on APMT for Structural & Multifunctional Materials & Systems

SYMPOSIUM 8: Advanced powder synthesis and processing

Room: Coquina F
Session Chair: Motoyuki Iijima, Yokohama National University

4:40 PM

(ICACC-S8-041-2024) Aion powder via dynamic thermochemical method

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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.

5:00 PM

(ICACC-S8-042-2024) Sliding wear behaviour of plasma sprayed 8YSZ/$Nd_2Zr_2O_7$ double layer thermal barrier coatings at elevated temperatures

M. V. Sudandaradoss*[1]; S. Kalasala[1]

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Zirconia has been considered to be a good candidate coating material for high-temperature applications on account of its superior properties such as low thermal conductivity, high thermal expansion coefficient, high toughness, good chemical and dimensional stability, high melting point, high wear resistance, low density, high hardness, stiffness, strength and refractoriness. Plasma spraying is versatile; practically, all materials that can be melted without decomposition can be deposited as coatings on virtually all reasonably heat resistant substrates. Hence, in this present work, an attempt has been made to study the dry sliding wear behaviour of 8 wt% Yttria Stabilized Zirconia (8YSZ) and Neodymium Zirconate ($Nd_2Zr_2O_7$) single layer and double layer thermal barrier coatings on AISI 304 Stainless Steel, using a pin-on-disc wear test apparatus. Empirical relationships were derived using Response Surface Methodology (RSM) to study the effects of applied load, sliding velocity, sliding distance and temperature on the dry sliding wear behaviour of the above mentioned coatings. The wear behaviour of the AISI 304 Stainless steel material is also compared with that of the considered ceramic coatings for a greater understanding of the dry sliding wear phenomenon.

Poster Session II- Group B presenting

Room: Ocean Center

5:00 PM

(ICACC-P056-2024) Multilayer interface for garnet solid electrolyte in Study on the Characteristics of Lithium Metal Batteries

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Lithium-ion batteries (LIB), which is a rechargeable battery, mainly used in related electronics industries. The LIB life would decrease with increasing in the number of charge and discharge cycles. Besides, the risk of LIB electrolyte is a liquid organic solvent, which is easy to burn and explode in case of fire. Recently, solid-state electrolytes have attracted attentions and show the many advantages including high safety, high energy density, and greater temperature tolerance. Among different types of solid-state electrolytes, garnet-type LLZTO electrolyte has high ionic conductivity and chemical stability towards lithium metal. However, lithium dendrites will be generated during the charge and discharge process of solid electrolyte batteries. The dendrites could lead to short circuit and failure of battery. Moreover, poor contact in the interface between solid electrolyte and lithium metal could cause the increased impedance and decreased conductivity. The aim in this study is to use different types and proportions of metal fluorides to modify the interface; meanwhile, the interface forms an electronically insulating and lithium-friendly lithium fluoride layer to promote the chemical diffusion of lithium in lithium metal alloys and reduce interface impedance. we expect that a new generation of lithium metal solid-state batteries will be developed.

(ICACC-P057-2024) Optimizing Secondary Phase Dispersion within High Entropy Rare Earth Oxides for Environmental Barrier Coatings

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High entropy rare earth oxide (HERO) coatings are under investigation as potential environmental barrier coatings (EBCs) to be utilized for Nb-base refractory metal alloys (RMAs). Nb-base RMAs are of interest due to their ability to operate at higher temperatures compared to state-of-the-art Ni-base super alloys. However, Nb-base RMAs lack the ability to form a protective oxide layer, making an EBC essential. $(Y,La,Nd,Ho,Yb)_2O_3$ compositions were synthesized with varying percentages of rare earth cations to optimize thermochemical and mechanical properties. Lab scale coupons were created using spark plasma sintering (SPS) and were characterized using scanning electron microscopy (SEM) for microstructure examination, and X-ray diffraction (XRD) hot-stage analysis for coefficient of thermal expansion (CTE) measurements. Results show that a secondary phase was present in the equimolar HERO composition $(Y,La,Nd,Ho,Yb)_2O_3$. In contrast, modifying the ratio of RE_2O_3 (RE = $La,Nd,Ho,Yb)_2O_3$ in a majority cubic Y_2O_3 (80 mol%) system led to a formation of a single phase. This presentation will examine the effects of varying the rare earth oxide amounts within the HERO composition $(Y,La,Nd,Ho,Yb)_2O_3$ on secondary phase formation and thermal expansion anisotropy.

(ICACC-P058-2024) Co-free protective coatings by electrophoretic deposition technique for reversible solid oxide cells

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2. Catalonia Institute for Energy Research, Advanced Materials for Energy Applications, Spain
3. SolydEra Spa, Italy

The metallic interconnects (ICs) in the reversible Solid Oxide Cell (SOC) stacks are exposed to high current density, thermal gradients and a corrosive atmosphere. These factors lead to high-temperature corrosion and chromium evaporation, with possible degradation of the metallic component. The deposition of a protective coating is an accepted solution to mitigate the degradation of the metallic material and to protect the IC. Currently, the scientific community's interest resides in the exploration of co-free coating compositions, while also improving the coating performances. In the framework of the project 24/7 ZEN (G.A. No 101101418, co-founded by the European Union and the Swiss State), we optimise the electrophoretic deposition (EPD) method to process co-free ceramic protective coatings based on manganese-copper spinel coatings for AISI441 stainless-steel. The EPD method allows to obtain dense and continuous coatings with reproducible properties in a few seconds and at room conditions. Moreover, we explore the role of iron-doping of the Mn-Cu spinel obtained by electrophoretic co-deposition route, to evaluate the improvement of thermal stability and protective properties of the coatings. Finally, developing multi-layered coating systems by the sputtering technique in synergy with EPD is investigated as a possible approach to enhance the coating Cr retention capability.

(ICACC-P059-2024) Protonic ceramic electrolysis cells: Sealants development for cell assembly

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3. EIFER – European Institute for Energy Research, Germany

In protonic ceramic electrolysis cells (PCECs) the electrolyte is a proton-conductive ceramic material, characterized by high conductivity. Compared to other high temperature electrolysis cells, PCECs are expected to present less degradation issues for long term applications, due to the operating temperature which is typically 400-600 °C. Additionally, a PCEC produces pure hydrogen at the oxygen electrode, reducing cost. Aiming to the gas tightness of a PCEC stack assembly, the utilization of glass-ceramic materials as sealants is a viable solution. However, only a few studies about the compatibility of glass-ceramic sealants and PCEC materials are nowadays present in literature. Here, a thermo-compatibility study among different glass-ceramic systems and a $BaCe_{0.7}Zr_{0.2}Y_{0.1}O_{3-\delta}$ (BCZY721) electrolyte is presented. The glass ceramic systems were also coupled with AISI 441, a typical ferritic stainless steel used for this application. The study was conducted in dry and humidified air (50 % air, 50% steam) and the results of postmortem characterization (FESEM and XRD) on the joined samples, are compared and discussed.

(ICACC-P060-2024) Unlocking stability: High entropy oxides as resilient oxygen electrode materials

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4. IREC, Nanoionics and Fuel Cells, Spain

Despite the immense potential of solid oxide fuel cells within this carbon-neutral paradigm, the widespread commercialization of SOCs faces significant impediments stemming from issues such as elevated degradation rates and polarization losses. To address these challenges, a promising solution has emerged in the form of high entropy oxides (HEOs). In this study, the authors synthesized the HEO by incorporating lanthanide elements into the A-site of the perovskite structure, while the B-site was occupied by multiple transition metals (TM). The synthesis of the HEO was achieved using the Pechini method, resulting in the formation of a single-phase finely powdered material. This HEO powder was integrated into anode-supported cell and tested under SOFC and SOEC conditions. At 0.7 V, a current density of 1.75 A cm^{-2} was obtained at 900 ºC, making this one of the highest recorded current densities for a cell using high entropy materials. The superior performance is coupled with remarkable stability over time, even at high current densities (1 A cm^{-2}). The remarkable stability exhibited by the synthesized material goes beyond merely showcasing the possibility of achieving high-performance materials. This achievement underscores the notion that entropy stabilization can be a game-changer in the pursuit of advanced materials with exceptional reliability and longevity, especially in SOC field.

(ICACC-P061-2024) Recovery and re-use of ceramic materials from end-of-life solid oxide cells (SOCs)

S. Saffirio*[1]; S. Anelli[1]; D. Ferrero[2]; I. Schiavi[3]; M. Santarelli[2]; S. Pylypko[4]; F. Smeacetto[1]; S. Fiorilli[1]

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3. Environmental Park, Turin, Italy
4. Elcogen, Estonia

SOCs are among the most promising technologies in view of achieving a deep decarbonization of the energy system. However, their market breakthrough is still hindered by the lack of efficient, scalable and cost-effective end-of-life (EoL) strategies enabling the recovery and re-utilization of raw materials. In this regard, the present study aims at demonstrating the optimization of an efficient single-step recycling process for Ni-YSZ components of end-of-life (EoL) SOCs by combining hydrothermal treatment and metal acidic leaching. The process, optimized at bench scale, has been adapted on a larger scale, allowing to target the amount of recycled powder necessary for new SOCs manufacturing (containing min. 30 wt% of recovered powders). The selective recovery of Ni (in the form of NiO) and YSZ from the sintered composite was achieved by treating the Ni-YSZ cell components inside a hydrothermal reactor containing low-concentrated (0.6–1.0M) HNO_3 solutions. The processing parameters, including time, temperature, acid concentration and solid/liquid ratio, were optimized considering their mutual interaction, maximizing the overall efficiency of the process. The re-manufactured cells were electrochemically and mechanically characterized for a comparison with standard virgin SOCs.

(ICACC-P062-2024) Enhanced performance of reversible solid oxide cells by densification of $Gd_{0.1}Ce_{0.9}O_{2-\delta}$ (GDC) barrier layer/oxygen-electrode interface *WITHDRAWN*

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3. Chonnam National University, Materials Science and Engineering, Republic of Korea

In reversible solid oxide cells (RSOCs) with YSZ electrolyte and LSCF6482-based oxygen electrode, a GDC barrier layer is used to prevent inter-diffusion of constituents. However, performance of such RSOC is limited to achievable density of GDC at prevailing temperatures of traditional fabrication methods. In this work, GDC barrier layer was densified via infiltration with polyvinylpyrrolidone-chelated sol and subsequent sintering at 1200 ºC. The NiO-YSZ fuel electrode-supported RSOCs with YSZ electrolyte, porous or infiltrated GDC barrier layer and LSCF6482-GDC composite oxygen electrode was fabricated. The performance evaluation by current-voltage and EIS indicated that the RSOC with infiltrated GDC barrier layer showed 32-48% increase in fuel cell performance, compared to that of porous GDC layer. Similarly, in electrolysis cell operation, infiltrated GDC barrier layer showed a higher current density of higher than 1.2 A cm^{-2} for a thermal neutral voltage of 1.23 V compared to 0.7 A cm^{-2} for porous RSOC. The distribution of relaxation time analysis of EIS data indicated that the effectiveness of infiltrated GDC barrier layer in performance enhancement may be attributed to its ability to suppress the possibility of current constriction effect at the YSZ/GDC and GDC/LSCF6482-GDC interfaces.

(ICACC-P063-2024) Protective Ceramic Coatings on SOFC Metallic Interconnects with Ni Buffer Layer

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2. Korea Institute of Energy Research, Republic of Korea

Metal interconnectors in solid oxide fuel cells (SOFCs) serve the purpose of electrically connecting single cells while separating oxygen (air) from the fuel supplied to adjacent cells. Typically, the surface of the metal interconnector is coated with a conductive oxide to enhance surface stability, mitigate chromium poisoning of the oxide anode, and ensure electrical conduction simultaneously. In this study, a Ni buffer layer was electroplated onto the base material to improve long-term durability by promoting adhesion. Subsequently, Mn and Co were applied using the chemical-assisted electrochemical deposition method in the form of metal hydroxide. Finally, a dense and thin composite spinel coating layer consisting of Ni, Mn, and Co was formed through heat treatment. The Ni, Mn, Co spinel coating effectively prevents the evaporation of Cr-containing species from the interconnect. Moreover, we confirmed the long-term durability and high conductivity of the metal interconnector after applying the (Ni, Mn, Co) composite spinel coating.

(ICACC-P065-2024) Influence of Magnetic Fields during Metal Organic Chemical Vapor Deposition

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Chemical Vapor Deposition (CVD) is a well-studied and versatile form of thin film production. Nevertheless, the decomposition of gas phase precursors and the material formation remains not fully understood and oftentimes empirical measures are necessary for obtaining high quality films. Applying a magnetic field (mf) during the deposition introduces a new parameter, over which crystallinity, grain size, phase and morphology of the deposited material can be tuned and thus can help understanding processes happening inside the reactor. Functional properties of materials can be altered and improved. The deposition of the $[\mathrm{Fe^{III}(O^tBu)_3}]_2$ precursor with applied magnetic field results in improved chemical homogeneity of the magnetite films, high crystallinity and increased grain size compared to the control experiment. The precursor $[\text{fac-Re(I)(CO)}_3\text{(L)}]$ (e.g., L= N,N-(4,4,4-trifluorobut-1-en-3-on)-dimethyl propylene diamine) was deposited under the influence of an external magnetic field and the preferred growth direction of the obtained ReN thin films was altered and materials properties like roughness and densification are tuned. The deposition of $[\mathrm{Cr(O^tBu)_4}]$ in high magnetic field yields Cr_2C_3 opposed to Cr_2O_3 without magnetic field and leaves a striking impression of the potential of an applied magnetic field during CVD experiments.

(ICACC-P066-2024) Micro and Nano Scale Applications of Nanocrystalline Diamond Films

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Many current Si-based MEMS components and devices exhibit performance limitations due to the relatively poor mechanical, chemical and tribological properties of Si. Diamond and diamond-like carbon offer advantages over conventional materials currently being explored for such applications, particularly over Si and SiC being explored as alternative MEMS materials. Fabrication of MEMS components and devices based on diamond and diamond-like films can be achieved using microfabrication techniques developed for Si-MEMS. However, fabrication of diamond-MEMS components using conventional CVD processes results in diamond coatings with coarse grains (≥ 1 μm) and rough surfaces (rms ~0.5-1mm), while vapor-deposited diamond-like coatings are not suitable for covering high aspect ratio MEMS features conformally, require high temperature post-deposition processing to relieve stresses, and exhibit lower hardness than natural diamond. Nanocrystalline diamond (NCD) coatings with small grains and smooth surface provides a unique combination of excellent mechanical, tribological, chemical, electrical and biocompatible properties. The NCD films were grown by microwave plasma enhanced chemical vapor deposition (MPECVD) method. SEM, Raman, NEXAFS, TEM, and other techniques were used to characterize the NCD films. A discussion will be presented on the applied science done on NCD processing and characterization techniques.

(ICACC-P067-2024) Development of Direct Z-Scheme g-C3N4/TiO2/CdS Heterojunction for Photocatalytic Water Splitting

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g-C3N4, a metal-free semiconductor, has attracted immense attention due to its potential application in solar energy conversion/storage, photocatalysts, and electrocatalysts. Due to its appropriate band gap energy and its capacity to absorb across the solar spectrum, g-C3N4 has the potential to demonstrate superior photocatalytic performance. However, its performance is limited by the rapid recombination of electron-hole pairs. Many strategies such as metal deposition, non-metal doping, and forming direct-Z Scheme heterojunction are being employed to overcome this limitation. Among them, the direct-Z Scheme heterojunction approach might be an effective way to maintain the separation of photogenerated carriers and reduce the recombination rate. In the current work, we investigated the synthesis, characterization, and photocatalytic hydrogen production of a direct-Z Scheme heterostructure, i.e. g-C3N4/TiO2/CdS. We performed a sequential synthesis in which TiO2 and CdS were co-deposited onto g-C3N4 nanosheets synthesized by polycondensation of urea at 550 °C for 2 hours. Photocatalytic water splitting results depicted that the hydrogen production capacity of the ternary heterostructure exceeds 570 μmol/g under UV light in an hour. ACKNOWLEDGMENTS: This works is financially supported by Izmir Natural Gas Distribution Inc. and the Republic of Turkiye Energy Market Regulatory Authority.

(ICACC-P068-2024) Is it possible to achieve ultra-rapid debinding and sintering of samples fabricated using direct ink writing in a single step?

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In the past few years, there has been a considerable development and growth of additive manufacturing involving ceramic materials. Among all the available AM technologies, Direct ink writing (DIW), has evolved to be one of the most widely used techniques as it offers the benefit of using a very low amount of organics while formulating the ink, which proves to be advantageous in mitigating issues such as cracking and excessive shrinkage during the subsequent debinding and sintering steps. Over the last decade, there has been a significant revolution in ceramic processing with the development of novel field-assisted sintering techniques which aims to save energy by drastically reducing the sintering temperature and time. In this study, using $BaTiO_3$ as a model ceramic system, we investigated the UHS (Ultra-fast high temperature sintering) and P-SPS (Pressureless spark plasma sintering) of components (porous log-pile structure) fabricated using Direct ink writing (Nozzle 0.4mm). With optimized ink rheology, it was possible to obtain crack-free sintered components in the order of seconds to minutes. This study demonstrates that UHS and P-SPS can be used to rapidly debind and sinter 3D printed components in one step, without the need for a separate debinding step, which can be further extended to a wide range of complex geometries and compositions.

(ICACC-P069-2024) Characterization of nano apatite powders synthesized via microwave heating and wet chemical precipitation method

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Some research methods in the past make use of the calcination process to produce apatite powders. It is then necessary to produce apatite powders using a shorter processing time. In this paper, hydroxyapatite powders were synthesized using microwave-assisted wet chemical precipitation method using appropriate amounts of calcium and phosphorous precursors. The structural, crystallographic and morphological properties of produced hydroxyapatite powders were then evaluated. Specifically, this study reports the lattice parameters, crystallite size, crystallinity, composition, and morphology of the synthesized hydroxyapatite powders including its purity. Scanning electron microscopy (SEM) with Energy Dispersive X-Ray Analysis (EDX), x-ray diffraction (XRD) analysis and Fourier transform infrared spectroscopy (FTIR) were done to characterize the acquired powdered samples. The formed products were stoichiometric and nano-sized which have low crystallinity. The synthesized apatite powders were comparable to the hydroxyapatite samples that is commercially available. There is a decrease in lattice parameters for microwave-heated apatite powders prepared via dropwise addition of diammonium phosphate chemicals. The surface morphologies of the powders were shown as agglomerated nano-sized spherical grains.

(ICACC-P070-2024) Effect of carbon content on electrical, thermal, and mechanical properties of pressureless sintered SiC ceramics

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The electrical, thermal, and mechanical properties of pressureless solid-state sintered SiC ceramics with B_4C-C additives were investigated as a function of carbon content. The electrical resistivity of SiC ceramics decreased from 4.74×10^2 to $9.07 \times 10^{-2}\Omega$.cm with an increase in carbon content from 2.5 wt% to 10.5 wt% due to the presence of higher electrically conductive C phase while the thermal conductivity decreased from 151.6 to 116.5 $W.m^{-1}k^{-1}$ due to higher phonon scattering at the SiC-C interfaces. The typical flexural strength of the SiC ceramics initially increased from 291.7 to 384.0 MPa with the addition of 4.0 wt% carbon due to higher densification and then gradually decreased to 159.9 MPa with increasing the carbon content. The relative density, flexural strength, and fracture toughness of the SiC ceramics with 0.8 wt% B_4C-4.0 wt% C additives are 98.8 %, 384.0 MPa, and 2.85 $MPa.m^{1/2}$, respectively. Thus, by controlling the carbon content in SiC ceramics, the electrical resistivity, thermal conductivity, and flexural strength can be tuned based on the application areas.

(ICACC-P072-2024) A Study of Tribological Behavior of Non-Oxide Ceramics

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Non-oxide ceramics have become important for different types of wear resistant applications. More detailed studies are needed to understand their tribological behavior in different environments. In this undergraduate research poster, we will review the tribological behavior of non-oxide ceramics like SiC and Si3N4. Their behavior will be compared with AlN which is fabricated by salt encapsulation method. Detailed microstructural studies will be presented.

(ICACC-P073-2024) Current Status on the Design and Development of Ice-Phobic Materials

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In the pursuit of enhancing the durability and safety of critical infrastructure in cold and icy environments, the development of ice-phobic materials has gained significant attention recently. This poster presentation delves into the advancements in ice-phobicity test stands we are making at UND, shedding light on their pivotal role in materials research. Our research addresses the need for reliable and standardized methods to evaluate the ice-phobic properties of materials. We are exploring novel designs and testing protocols that encompass a wide range of environmental conditions, ensuring a comprehensive assessment of material performance. These advancements will not only facilitate the development of new ice-phobic materials and surfaces but also enable the optimization of existing materials for diverse applications, such as aircraft components, power transmission lines, and transportation infrastructure. Additionally, we discuss the integration of state-of-the-art instrumentation and automation techniques into these test stand, enhancing precision and repeatability.

(ICACC-P074-2024) Electrical, Thermal, and Mechanical Properties of Pressureless Sintered SiC-TiB_2 Composites

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SiC-TiB_2 composites were fabricated from α-SiC and TiB_2 powders with 3.5 wt% B_4C-C additives by pressureless sintering. Electrical, thermal and mechanical properties of the SiC-TiB_2 composites were investigated as a function of initial TiB_2 content. Relative densities of ≥98.5% were achieved for all samples. The addition of a small amount of TiB_2 decreased electrical resistivity and thermal conductivity, increased flexural strength. However, further addition of TiB_2 in excess of 10wt% deteriorated flexural strength of the composites. The minimum value of electrical resistivity was $4.23 \times 10^{-3}\Omega$.cm, the maximum values of thermal conductivity and flexural strength were 145 W/(m.K) for 0 wt% TiB_2 and 333 MPa for a 10 wt% TiB_2 composite.

(ICACC-P075-2024) Progress in Multiferroic Composites: Broadening the Frontiers of Ferroelectric and magnetic Substances

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Magnetoelectric coupling represents a specialized aspect within the broader spectrum of multiferroicity. This coupling has garnered significant global research attention due to its ability to facilitate the manipulation of electric polarization through magnetic fields and the control of magnetization via electric fields. The interplay between ferroelectric and ferromagnetic properties can be realized through various mechanisms, including strain-mediated, magnetoelectric, and exchange interactions. A commonly adopted approach involves combining a material such as Pb $(Zr,Ti)O_3$,Bi(Na,K)TiO_3,(Bi,Ba)(Na,K)$(Ti,Sn)O_3$ or $(K,Na)NbO_3$ with a magnetic counterpart like cobalt ferrite ($CoFe_2$O4), $NiZnFe_2O_4$, or $CoMn_2O_4$. Overcoming challenges associated with achieving robust coupling between the ferroelectric and magnetic phases is a focus, alongside ongoing endeavors to optimize material compositions and processing techniques. Simultaneously, efforts delve into unraveling the fundamental mechanisms underpinning the coupling between ferroelectric and ferromagnetic properties. This entails a detailed examination of the roles played by defects, interfaces, and domain structures in shaping the overall behavior of multiferroic composites. In summary, this abstract underscores the promising future of multiferroic composites as advanced functional materials.

(ICACC-P076-2024) New antipathogenic metal-ceramic composites coatings

J. Wojewoda-Budka*[1]; I. Kwiecien[3]; A. Bigos[1]; M. Bugajska[1]; M. Janusz-Skuza[1]; R. Major[1]; M. Szczerba[1]; A. Wierzbicka-Miernik[1]; J. Wiecek[1]; M. Dyner[2]; A. Misztela[2]; D. Veselinov[3]; H. Skulev[3]

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3. Bulgarian Academy of Sciences, Institute of Metal Science, Bulgaria

Studies are dedicated to designing and manufacturing new copper-based coatings with enhanced functional properties that align with the global trend of disease prevention rather than treatment. The primary innovative goal of this research is to diminish pathogen viability on newly produced copper and copper-ceramic composite coatings. These coatings are obtained through a relatively simple implementation of environmentally friendly plating technology utilizing eco-friendly reagents. The antibacterial activity of copper is greatly reliant on its concentration. Hence, the economically viable approach is to employ durable and wear-resistant coatings with the highest feasible copper content. An important aspect was to validate that copper-based composite coatings, obtained by simultaneously adding the TiO_2 particles during the deposition process, would offer an even more effective defence against the spread of pathogenic organisms. The influence of TiO_2 particle size on the anti-pathogenic properties of the copper coatings was also studied, followed by a comprehensive microstructure characterization of the resulting coatings with scanning and transmission electron microscopy techniques. This research was in the frame of project MERA.NET2/2020/AntiPathCoat/4/2021 financed by The National Centre for Research and Development in Poland by the M-ERA.NET program.

(ICACC-P077-2024) Ceramic to metal joining for high temperature oxygen separation membrane applications

S. De La Pierre[1]; P. Fedeli[2]; A. Cavaliere[2]; A. Cammi[2]; F. Da Prato[1]; S. Anelli[1]; F. Smeacetto[1]; F. Drago[2]; M. Ferraris*[1]

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The integration of Oxygen Transport Membranes (OTM) in industrial processes can lead to energetic and economic advantages, but proof of concept membrane modules are highly necessary to demonstrate the feasibility of this technology. This study identifies innovative materials to be used to join ceramics to metals, with a specific focus on joining materials for the integration of OTM modules for high temperature applications. Perovskite based membranes have been selected for the oxygen diffusion, in order to improve robustness, reliability and high temperature resistance with respect to the most used polymeric ones. Glass ceramic joining materials (or sealants) are widely employed in the energy production sector, as they avoid gas mixing and consequent device failure. The sealants are characterized by high gas tightness, thermo-chemical and thermo-mechanical compatibility with metallic and ceramic materials, as well as stability in the relevant operating conditions (800-950 °C, thousand hours). The housing of the membrane component is a metallic case joined to the membrane component by means of custom developed glass-ceramic sealants, that exhibited a remarkable thermo-chemical compatibility both with metal and ceramic. In this work is presented the development and high temperature characterization carried out on glass-ceramic sealants selected to join ceramic membranes (LSCF) to the metallic part.

(ICACC-P078-2024) Development and Cost Assessment of Ceramic Membranes for CO_2 Separation from Natural Gas

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A ceramic membrane designed for the separation of CO_2 from natural gas streams was developed at the Laboratory of Ceramic Materials (LMC), Federal University of Minas Gerais (UFMG), Brazil. The membrane's structure comprises three layers: a tubular alumina support (d_p 0.7 to 1 μm), an intermediate alumina layer (d_p 4 to 100 nm), and a top selective silica layer ($d_p < 2$ nm). Gas permeation tests (CO_2, N_2, and CH_4) were conducted by varying the feed pressure from 2 to 10 bar and ΔP from 1 to 2 bar, aimed at evaluating the membrane's performance concerning gas flux and selectivity. The data derived from these tests were employed to compute the permeance and permselectivity of the membrane. Operating under conditions with a feed pressure of 10 bar and ΔP of 2.0 bar, the developed ceramic membrane exhibited a substantial CO_2 flux, reaching approximately 4000 GPU (10^{-6} mol/m^2.s.Pa), and demonstrated a CO_2/N_2 permselectivity of 7 and a CO_2/CH_4 permselectivity of 10. The obtained permeance significantly surpassed that of commercial polymeric membranes, typically around 1500 Barrer (10^{-7} mol/m^2.s.Pa), which are presently utilized in natural gas treatment facilities. In terms of cost assessment for the ceramic membrane, a comprehensive analysis was undertaken, encompassing all stages from ceramic support production to the deposition of the separation layer and a value of 1,678 US$/m^2 was obtained.

(ICACC-P079-2024) Microplastic transformation and retention mechanism in ceramic membranes for wastewater treatment

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FlowCam offers multiple measurement outputs, making it suitable for automated classification to separate polyethylene (PE) and polyamide (PA) microplastics in wastewater samples. Customized libraries for each microplastic were constructed using statistical analysis with appropriate confidence intervals, enhancing sorting accuracy. Comparing multiple FlowCam outputs before and after membrane filtration revealed limitations in dimensional outputs, particularly for irregularly shaped PA microplastics. In particular, a marginal increase in the elongation of PA microplastics after filtration suggested their ability to longitudinally penetrate pores due to their high length relative to thickness. Conversely, PE microplastics with higher circularity, resembling ideal spheres, exhibited greater susceptibility to removal by the membrane pores. These findings indicate the significant role of microplastic geometry and morphology in retention mechanism and membrane fouling. This study highlights FlowCam's potential for microplastic identification and provides valuable insights into characterizing microplastic transformations within membrane filtration systems, addressing a pressing concern in environmental science within engineered systems.

(ICACC-P080-2024) Fabrication and characterization of nanofibrous porous ceramic materials in B_2O_3-Al_2O_3-SiO_2 system

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Boron Alumina ($Al_{18}B_4O_{33}$) and Mullite ($Al_6Si_2O_{13}$) are stable ceramic materials within a B_2O_3-Al_2O_3-SiO_2 reaction system. In nanofiber form, these materials exhibit attractive thermal, mechanical, chemical, and other properties that make them appealing for many demanding applications. However, there are few reports on sizeable production of such nanofibrous ceramics. This study explores the fabrication of several types of B_2O_3-Al_2O_3-SiO_2 nanofibrous materials using a high-yield alternating field electrospinning (AFES) at several g/h (up to 14.4 g/h) final ceramic fiber productivity. In a typical process, nanofibrous ceramic sheets with thickness up to 25 mm and 99.97% porosity were produced. SEM imaging revealed that the ceramic nanofibers with all tested compositions had ~400 ±100 nm fiber diameter after the annealing up to 1200 °C. FTIR and XRD measurements revealed that the nanofibers start to crystallize above 800 °C and the degree of crystallization increases with temperature. Pure phase mullite and $Al_4B_2O_9$ nanofibers were obtained at 1000 °C, and pure phase $Al_{18}B_4O_{33}$ nanofibers were prepared at 1200°C. With the increasing content of silica in B_2O_3-Al_2O_3 nanofibers, the loss of boron is compensated by silicon atom entering the crystalline lattice and resulting in a boron-silicon "mullite" structure with different B/Si ratio.

(ICACC-P081-2024) Macroporous Alumina Foams Fabricated by Gel-casting Using Pre-expanded Microspheres

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Macroporous alumina foams were fabricated by gel-casting using pre-expanded polymeric-based microspheres with average sizes of 40 μm and cellulose-based microspheres with an average size of 50 μm as sacrificial templates. The gel-casting method, the effect of solvent on the stability of the microspheres, the volume fraction of microspheres, as well as the drying, burnout, and pre-sintering conditions, were investigated to produce mechanically stable alumina monoliths with 65 and 80 vol% porosity. The electron microscopy investigation of the pre-sintered alumina monoliths revealed that the size distribution and the shape of the pores could be tailored by controlling the particle size distribution and the shape of the wet pre-expanded microspheres. Highly porous and mechanically stable alumina foams achieved compressive strengths from 3 - 40 MPa. Given the relatively open pore structure, the pore size distribution, the pre-sintered mechanical strength, and the high porosity achieved, the produced alumina foams could potentially be used as support structures for separation, catalytic, and filtration applications. Moreover, the analysis of evolved gases showed the polymer microsphers released nitric oxides (NOx), sulfur compounds and COx components while the cellulose microspheres released COx and CH_4 gases suggest a reduction in the evolved harmful components and their low CO_2 impact.

(ICACC-P082-2024) Zirconia-yttria/lithium-sodium-potassium carbonates ceramic membranes with sodium-potassium carbonate anti-fouling layer for carbon dioxide permeation

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The interfaces of porous ZrO_2:3 mol% Y_2O_3 (3YSZ), prepared by thermally removing graphite during sintering, were modified by introducing a layer of $(Na,K)_2CO_3$ (NKC) before molten $(Li,Na,K)_2CO_3$ (LNKC) was impregnated into the pellet pores. X-ray diffraction (XRD), scanning electron microscopy (SEM-EDX), and electrochemical impedance spectroscopy (EIS) were the techniques applied to analyze structural phases, pore content, and carbon dioxide ion conductivity, respectively. EDX analyzes showed that LNKC face-to-face percolation was completed. EIS experiments were conducted at temperatures both below and above the melting point of LNKC for assessing the improvement of carbon dioxide permeation with the introduction of the NKC protective layer, apparently preventing partially 3YSZ-LNKC fouling reaction.

(ICACC-P083-2024) Application and characterization of a kerosine-fuelled High Velocity Oxy-Fuel (HVOF) Ti_2AlC coating on thermally stable P91 steel

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A class of ternary layered carbides and nitrides, known as MAX phases, combines some of the best properties of two distinct classes of materials: metals and ceramics. Specifically, MAX phases are stable at high temperatures, resist thermal shock, and some even form stable and protective oxide layers in oxidizing environments. Thus, they are excellent candidates for protective coatings in high-temperature applications. In this context, our focus is on using kerosene-fueled High-Velocity Oxy-Fuel spraying to deposit MAX phases onto P91 steel substrates for use as thermal barrier coatings. The chosen MAX phase for this study is Ti_2AlC, which offers an outstanding oxidation resistance. Our results indicate that during the spraying procedure, a portion of the MAX phase decomposes, another portion oxidizes, yet more than half maintains the initial and desired stoichiometry. Nevertheless, the resulting coating establishes a stable and robust bond with the steel substrate. In this presentation, we will discuss the detailed analysis concerning the relationship between the process, structure, and performance of the Ti_2AlC coating on the P91 steel substrate.

(ICACC-P084-2024) Continuous fiber-reinforced MAX-Phases: Investigation of a Pressure Slip Casting Route for the Production of $Al_2O_{3(f)}/Ti_2AlC$-CMCs

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MAX phase ceramics show great potential for a new, innovative generation of engineering materials for energy technology due to their excellent mechanical properties in a high-temperature atmosphere. Fiber-reinforcement of MAX phases show great potential to further increase the materials engineering capabilities. However, the mechanisms to introduce continuous fiber-reinforcement into the novel ceramic system are not understood yet. Suitable processes for the production of continuous fiber-reinforced ceramics are currently formulated only by colloidal manufacturing processes. A major challenge of colloidal processes regards the homogeneous impregnation of textile reinforcement structures. However, the formulation of highly filled, fiber-reinforced green bodies decreases with increasing complexity of the molded part design. For this purpose, the production of $Al_2O_{3(f)}$-Ti_2AlC-CMC is investigated for the pressure slip casting technology. The innovative composite material is being developed within the publicly funded project ContiMAX (German Research Foundation DFG project No.: 508093957). To this end, the research to be presented is investigating basic mechanisms of MAX phase CMC processing to contribute to the development of future sustainable material systems to replace a large number of metallic high-temperature elements and wear-intensive components.

(ICACC-P085-2024) MXene Derived Carbides As Precursors For Ultra High Temperature Ceramics

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$Ti_3C_2T_x$ MXene is a two-dimensional (2D) refractory carbide which has high solution processability, high aspect ratios, and exhibits one of the highest stiffness (~330 GPa) among solution-processable 2D materials. We have investigated a one-pot, surfactant-free, aqueous mixing method to develop homogeneous ZrB_2-Ti_3C_2 MXene green bodies. We present the phase transformation of Ti_3C_2 MXene to TiC_y at the grain boundaries upon sintering, role of cationic species on texturing, the interface interactions between the ZrB_2-TiC_y grains and their densification mechanisms. A nominal relative density of ~96% is achieved when 0.5 wt.% Ti_3C_2 MXene is added to ZrB_2 and spark plasma sintered at 1900C with 50 MPa pressure in inert atmospheres. The microstructure evolution, stability, and their effect on micromechanical properties of the resulting UHTCs will be presented and discussed. This study lays the groundwork for 2D MXenes to be used as template precursors for 2D carbides and their implementation as compatible materials for high-temperature applications.

(ICACC-P086-2024) Thermodynamically Consistent Model of Electrocaloric Effect

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The electrocaloric effect (EE) is a phenomenon in which a material shows a reversible temperature change under an applied electric field. Current state-of-art is presented briefly summarized in the WIKI paper “Electrocaloric effect”: “The underlying mechanism of the EE effect is not fully established; in particular, different textbooks give conflicting explanations”. One of the main difficulties in the phenomenological modeling of EE stems from the various gaps in the interpretation of the thermodynamic fundamentals of electricity and magnetism. That is why we pay particular attention to the thermodynamic consistency of the suggested model of EE. Potential practical applications of EE include computer cooling and the development of batteries for computer systems. In the long-term progress, the electrocaloric effect might be used in high-efficiency heat pumps. The main ideas of our approach were presented in Grinfeld M., Grinfeld P. “Thermodynamically Consistent Analysis of Magnetocaloric Effect”, Applied Mathematics and Physics. 2020, 8(1), 14-19. In this paper, in addition to the magnetocaloric effect we develop a similar approach for the case of EE.

(ICACC-P087-2024) Modulating self-biased near-UV photodetection of Gd-doped bismuth ferrite ceramics by introducing zinc oxide as electron transport layer

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ZnO semiconductor offers many advantages as an electron transport layer (ETL) in photovoltaic-based devices, including high charge carrier mobility and hole-blocking ability. In this work, ZnO thin film is introduced as an ETL between (Bi0.93Gd0.07) FeO3 (BFO7Gd) ferroelectric and ITO thin film to form ITO/ZnO/BFO7Gd/Au heterostructure. The device with the ETL exhibited a superior photoresponsivity than the one without ETL, reaching ~32% enhancement. Furthermore, a subsequent E-field poling on the ITO/ZnO/BFO7Gd/Au heterostructure resulted in an additional ~25% increase in photoresponsivity. The enhancement is mainly attributed to two factors: (1) high electron mobility and lower recombination rate resulting from the introduction of ZnO ETL, and (2) efficient charge separation facilitated by the polarization-driven internal E field that superimposes with the interfacial built-in E fields. The introduction of ZnO ETL and the utilization of the ferroelectric polarization prove to be an alternative route to further modulate the photosensing performance of BiFeO3-based near-UV photodetectors.

(ICACC-P088-2024) Development of Bi_2O_3 Nanoparticle-loaded Plastic Scintillators Based on Poly(9-vinylcarbazole)

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We developed surface-modified Bi_2O_3 nanoparticle-loaded plastic scintillators for application in scintillation detectors that have fast response to high-energy X-rays. We used poly(9-vinylcarbazole) (PVK) as the host polymer because of its low band gap energy. The scintillation properties of the plastic scintillators with different concentrations of nanoparticles and phosphor (9,10-diphenylantrazene (DPA)) were characterized to optimize concentrations of nanoparticles and DPA. Nanoparticles of 4.5 ± 0.92 nm with high dispersibility in PVK matrix were successfully synthesized by subcritical hydrothermal synthesis. Detection efficiencies and light yields significantly larer than those of EJ-256 were achieved for all samples with a nanoparticle concentration of 10 wt%. Among them, the sample with Bi_2O_3 nanoparticle concentration of 10 wt% and DPA concentration of 0.25 mol% showed the highest light yield of 10000 photons/MeV for high-energy X-rays of 67.41 keV, which is approximately twice of the light yield of EJ-256. Decay time constants of about 6-18 ns were obtained for all samples regardless of nanoparticle concentration. We achieved the fabrication of plastic scintillators with high light yield and high detection efficiency without losing the fast response derived from plastic scintillators.

(ICACC-P089-2024) Identification of numerical model based on rheological behavior of the 3D printed geopolymer paste

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Geopolymer materials have garnered increasing attention for their exceptional mechanical properties and environmental advantages, positioning them as promising candidates for 3D printing applications. This work introduces a numerical model, implemented in COMSOL Multiphysics, with the primary objective of predicting and managing dimensional variations in 3D-printed parts made from geopolymer pastes. We experimentally determine viscoelastic properties, including the storage modulus (G′) and loss modulus (G″), through rheological tests carried out with a rheometer. Our numerical model establishes connections between these properties and classical viscoelastic models, such as the Kelvin-Voigt and Burgers models, employing viscoelastic behavior equations. Through COMSOL Multiphysics simulations, we meticulously examine the behavior of various formulations of geopolymer pastes. These insights are invaluable for optimizing additive manufacturing processes, ensuring dimensional accuracy, and preventing part collapse. Furthermore, in future research, we will explore the incorporation of drying phenomena into our model, enhancing our ability to predict and manage dimensional changes. Our model also empowers the development of customized geopolymer materials, offering the capability to predict and manage dimensional changes, thereby significantly advancing 3D printing technology.

(ICACC-P090-2024) Property Tuning of Piezoelectric Ceramic Sensors

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Piezoelectric ceramic sensors exhibit high sensitivity and wide frequency range and are inexpensive to produce. However, state-of-the-art manufacturing techniques result in brittle piezoelectric ceramics susceptible to fracturing under mechanical stress. Consequently, the application of piezoelectric ceramic sensors is limited by their durability. Polymer-derived ceramics (PDCs) offer an alternative manufacturing process to address this challenge. Using PDCs improves the durability of the sensor by minimizing porosity, allows for the tuning of a sensor’s mechanical properties by adjusting the pyrolysis temperature, and may increase the temperature limit of the sensor. It also allows for additive manufacturing which enables geometric design flexibility and rapid prototyping. However, the use of PDCs to produce piezoelectric ceramics has not been studied extensively. This experiment investigated the properties of piezoelectric ceramics derived from pre-ceramic polymer solutions containing barium titanate. The mechanical and piezoelectric properties of piezoelectric ceramics created under different pyrolysis conditions, poling processes, and compositions of the pre-ceramic polymer solution were analyzed. The resulting data was used to develop a framework that allows for the creation of piezoelectric ceramics with specific mechanical and piezoelectric characteristics.

(ICACC-P091-2024) Thermal Properties of HfB_2 Produced by Boro/carbothermal Reduction

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The thermal properties of ultra high temperature ceramics are sensitive to transition metal contaminants introduced during processing. Additions of a few weight percent of Hf or Ti to ZrB_2 have been shown to affect the thermal conductivity. In this study, HfB_2 will be synthesized by boro/carbothermal reduction of high purity HfO_2. Different milling procedures will be used to determine the effect of impurities or residual oxides on the properties of the densified ceramics. The milling procedures will consist of high energy ball milling, or ball milling using either WC or ZrO_2 media. Thermal diffusivity, thermal expansion, and Vickers' hardness will be measured to determine the effect of milling parameters on the properties of the resulting HfB_2 ceramics. Scanning electron microscopy and x-ray diffraction will be used to determine the phase purity and analyze the effects of the milling procedures and impurities on the resulting microstructures. These tests will help define synthesis conditions to produce high purity HfB_2 powder for future studies.

(ICACC-P092-2024) Direct ink write additive manufacturing of carbon fiber reinforced ZrB_2

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The additive manufacturing technique of direct ink writing (DIW) allows for the near-net and complex-shaping of ceramic components using a viscous, shear thinning slurry. In this work, highly loaded (~ 50vol.% ceramic) aqueous slurries of zirconium diboride containing milled carbon fiber at various loadings have been prepared for DIW. The effects of fiber addition, sintering aid composition, and particle size on rheology, printability, and final sintered microstructure will be presented.

(ICACC-P093-2024) High entropy thin films ceramics for high hardness materials

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Transition metal high-entropy ceramic thin films are created through physical vapor deposition (PVD) using bipolar high power impulse magnetron sputtering (HiPIMS). This process stabilizes a chemically complex cation sub-lattice into a single-phase rock-salt (S.G. #225) or aluminum diboride (S.G. #191) structured ceramic film that demonstrates a high degree of configurational entropy compared to binary counterparts. Sample crystallinity and hardness are examined via X-ray diffraction (XRD), while Knoop microhardness tests determine hardness. Thin film morphology is elucidated using atomic force microscopy (AFM) and scanning electron microscopy (SEM). Manipulating the PVD process creates films high hardness and melting temperature. These properties are crucial to the development of materials in the aerospace industry such as thermal barriers for engines. This research investigates the ability to maximize the hardness of transition metal, high-entropy ceramic thin films.

(ICACC-P094-2024) Validation of Thermal Conductivity Technique for Low k/Insulating Oxides

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We present results for thermal conductivity measurements of UO_2 during laser heating in the extreme 1800K+ temperature regime using a newly developed technique. Utilizing both analytical and numerical solutions we measure a thermal conductivity by comparison of two steady-state distributions. Results suggest a strong dependence on spatial and temporal properties of the radiative heat transfer distribution and align closely with previously published literature.

(ICACC-P095-2024) Low-Temperature Fabrication of Si_3N_4 and AlN via the Salt Encapsulation Method

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With an increased focus on hypersonic platforms comes an increased need for ultra-high temperature ceramic (UHTC) materials. UHTCs are difficult to manufacture because of their high melting points, which also means higher sintering temperatures. This is especially true for non-oxide ceramics like Si_3N_4 and AlN, where oxidation below the nitrides' sintering temperatures (around 1800 °C) requires fabrication in an inert atmosphere. Ultra-high temperature ($T > 1200$ °C) vacuum furnaces and alternative sintering furnaces, e.g., spark plasma sintering furnaces, are expensive to purchase and maintain. As an alternative, the salt encapsulation method has been used to fabricate UHTC powders at temperatures as low as 800 °C. Few of these studies; however, have looked at sintering of bulk UHTCs. Here, we report our attempts at sintering Si_3N_4 and AlN ceramics with the goal of achieving at least 95% theoretical density. Through the salt encapsulation method, 80% theoretical density was achieved for Si_3N_4 ceramics while 85% was obtained with AlN. These results were achieved with powders compacted at pressures no higher than 40 MPa (uniaxial) and 60 MPa (hydrostatic) and heated, in a bed of NaCl, to 1000 °C for three hours. Further results will be presented for sintering temperatures up to 1200 °C in both NaCl and KBr.

(ICACC-P096-2024) Effect of hot forging condition on mechanical properties of modified tricalcium phosphate / poly(lactic acid) composite screw with stearic acid

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Composite of tricalcium phosphate (TCP) and poly(lactic acid) (PLA) was attracted attention as a material of bone fixation device which was used fracture treatment, because of bioabsorbable and osteoconductivity. However, the mechanical properties of this TCP/PLA composite are degraded due to stress concentration at the interface between TCP and PLA. In this study, a hybrid reinforcement combining interfacial treatment on TCP surfaces and hot forging was proposed as a molding method of TCP/PLA composite bone fixation device. Then, effect of forging condition on mechanical properties of hybrid reinforced TCP/PLA screw was investigated. At first, the composite of PLA and modified TCP with stearic acid was drawn in tensile direction. As a result, the tensile strength and elastic modulus of drawn TCP/PLA increased with interfacial treatment with stearic acid. And, the orientation function of PLA chain also increased with this treatment. Next, TCP/PLA screw was molded hot forging. As a result, shear strength and orientation function of this screw increased with extrusion ratio.

(ICACC-P097-2024) Analytical model for viscoplasticity behavior in CFRP angle-ply laminates

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In this study, viscoplastic mechanical behavior in CFRP angle-ply laminates is investigated both experimentally and analytically. Monotonic tensile tests at different strain rates, loading-unloading tests and stress relaxation tests are performed to evaluate the viscoplastic behavior experimentally. Based on the experimental results, we decided to employ a viscoplasticity model with overstress. In this model, total stress is additively decomposed into equilibrium stress and overstress. In equilibrium stress, an orthotropic plasticity model (Hoffman model) is used. For overstress, Lion's model is employed. The model is incorporated in a finite element analysis to compare with experimental results.

(ICACC-P098-2024) Splitting Progress in Unidirectional Carbon Fiber Reinforced Plastics under Fatigue Loading

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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-P099-2024) Effect of Preheating Conditions and Laminate Configuration on Dome-Shaped Deep Drawability of Carbon Fiber Reinforced Thermoplastics

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The deep drawability of carbon fiber reinforced thermoplastic laminates was evaluated. Deep drawing was performed on the twill weave laminates by changing the mold temperature. When the mold temperature was 160°C, large wrinkles were observed on the specimens and fiber breakage occurred. In the case of 210°C, deep drawability was good and no fiber breakage was observed. From the load-displacement diagram during molding, the load required for molding decreased with increasing temperature during molding. Deep drawing was also performed on laminates with various laminate configurations. In the woven laminate, the flange area deformed significantly. As a result, large wrinkles occurred. The generated wrinkles spread to the dome section and affected the formability of the dome section. In the quasi-isotropic laminate, wrinkles were dispersed throughout the laminate and the generation of large wrinkles was suppressed. The shape of the dome section was better molded in the quasi-isotropic laminate.

(ICACC-P100-2024) Cemamic phase addition in inorganic polymer matrices for carbor fiber rinforced composites: The FENICE project

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Electric vehicles (EVs), and battery boxes in particular, require safe materials for safety and fire prevention reasons. The "FENICE – Fire rEsistant eNvironmental frIendly CompositEs" project aims to produce innovative fire-resistant composite materials, consisting of carbon fibers and inorganic polymeric matrices, to reduce the risk of fire in vehicles and increase service temperatures above 750°C. The addition of the carbide and oxide ceramic phases is proposed to improve the performance of an alkali-aluminosilicate based inorganic polymer with a Si:Al ratio above 20. It has also been found that storage at +3°C allows the matrix to be kept in a semi-fluid/plastic state offering a perfectly mouldable impregnated fabric for easy industrial exploitation. Some results are presented demonstrating that inorganic polymer matrices doped with ceramic phases are the key feature to ensure a safe protective environment for electric vehicle batteries.

(ICACC-P101-2024) Improvement of powder injection molding process with cellulose nanofibers

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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-P102-2024) Injection molding of Cu/CF composites

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Metal Injection Molding (MIM) process is suitable process for fabricating small and complex shaped metal parts with high production volume. Carbon Fiber (CF) has high thermal conductivity, electrical conductivity and mechanical properties, thus, Copper/CF composites would show great properties. Especially using MIM process to fabricate the composites, fiber orientation would be controlled during injection molding. Also short fiber is easily and homogeneously dispersed in the metal powder during mixing process. In this study, Cu/CF composites were fabricated thorough MIM process. Effects of fiber contents and fiber orientation on the mechanical and thermal properties were investigated.

(ICACC-P103-2024) Improvement of thermal conductivity and mechanical properties in composites of PA11 using Sn and Al_2O_3

M. Ijiri*[1]; T. Osada[1]; S. Kobayashi[2]

1. Tokyo Metropolitan University, Japan
2. Tokyo Metropolitan University, Mechanical Engineering, Japan

In recent years, with the spread of electric vehicles, there has been a demand for smaller motors. However, as motors become smaller, the heat dissipation space decreases, so materials with high thermal efficiency are required. In this study, in order to produce polyamide-11 (PA11) with excellent thermal conductivity, PA11 containing Al_2O_3 and Sn was prepared, and the effect of the combination of fillers on thermal conductivity was investigated. The thermal conductivity of PA11 was 1.29 W/m K, and improved to 2.02 W/m·K when the Al_2O_3 content was 60 vol%. Moreover, when the total filler content was fixed at 60 vol%, the thermal conductivity of PA11 containing Al2O3 (55 vol%) and Sn (5 vol%) improved to 2.55 W/m K.

(ICACC-P104-2024) Effect of plasma treatment condition in the atmosphere on mechanical properties of plain weave carbon fiber reinforced polypropylene

M. Sakaguchi*[1]; Y. Nishi[1]

1. Salesian polytechnic, Mechanical and Electronic Engineering, Japan

Carbon fiber reinforced thermoplastics (CFRTP) has higher toughness and recyclability and lower molding cost than that of conventional carbon fiber reinforced thermoset (CFRTS). However, adhesiveness and/or wettability between carbon fiber and thermoplastics are low. Then, mechanical property of carbon fiber is not fully expressed in CFRTP. In this study, atmospheric pressure plasma treatment was focused to improve the adhesiveness and the wettability. The purpose of this study is to clarify the effect of atmospheric pressure plasma treatment conditions on mechanical property of CFRTP. The plain weave carbon fibers were treated by plasma generated in the atmosphere. Then, the composites plate of the treated carbon fibers and polypropylene (PP) was molded. The mechanical properties of this CF/PP composite were investigated bending test. As a result, bending strength of CF/PP composite increased with plasma treated time, from about 20.7 MPa at 0 s to 27.2 MPa at 40 s. And, elastic modulus also increased from about 4.1 GPa at 0 s to 6.1 GPa at 40 s. This may be due to the formation of hydrophilic groups such as hydroxyl groups and carboxyl groups on the surface of carbon fibers by atmospheric pressure plasma. These hydrophilic groups may have improved the adhesion between carbon fiber to matrix by bonding with the matrix chains.

(ICACC-P105-2024) Effect of Aluminum Content on Microstructure of Magnesium Alloys treated with Cavitation Treatment Containing Phosphoric Acid

M. Ijiri*[1]; S. Matsuoka[2]; S. Kikuchi[2]; T. Yoshimura[3]

1. Tokyo Metropolitan University, Japan
2. Shizuoka University, Japan
3. Sanyo-Onoda City University, Japan

Bioabsorbable vascular stents have been developed as bio-friendly medical devices that do not leave foreign substances in the body. The material used for the stent is a bioabsorbable magnesium. However, magnesium alone has poor strength, so alloys with high strength have been developed by adding small amounts of aluminum. Surface modification technology is attracting attention in order to further increase the strength of these alloys. In this study, we used AZ61 and AZ91, which have a high aluminum content, and investigated the corrosion resistance and fatigue properties of the treated surfaces by multifunction cavitation treatment (MC-MFC) with the addition of phosphoric acid. Cracks in the film and network-like products were observed on the surface after MC-MFC treatment. Based on elemental mapping, this product was thought to be formed by a compound derived from Mg-Zn-Al.

(ICACC-P106-2024) Experimental characterization of R curve during transverse crack growth in unidirectional CFRP laminates using size-effect law

S. Oshima*[1]; T. Noma[2]; K. Fukayama[2]; H. Hirai[2]; C. Gao[2]; T. Kusaka[2]

1. Tokyo Metropolitan University, Department of Aeronautics and Astronautics, Japan
2. Ritsumeikan Univrsity, Department of Mechanical Engineering, Japan

A crack growth resistance curve (R curve) in unidirectional carbon fiber reinforces polymer (CFRP) during transverse crack growth was evaluated using Bazant's size effect law with different sizes of specimens. In addition, a microscopic damage process in the vicinity of a crack tip was observed using a microscope. The damage process observed with a microscope was correlated with an R curve to understand the failure mechanisms. An R curve was successfully obtained using the size-effect methodology. The size of the fracture process zone and fracture toughness during steady-state crack propagation were determined from the R curve. The size of the fracture process zone determined by the size-effect law and that observed by microscopy were almost identical. Thus, this result suggests that the damage zone mainly developed in the crack growth direction.

(ICACC-P107-2024) Aerosol Deposition of Alumina Ceramic Coatings for High-Voltage Insulation

B. Xie*[1]

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

In new-generation more electric aircrafts, electrical machines are required to operate at higher voltages and be mounted in a hotter part of the engine to meet the increased power requirement. The use of high-quality insulation materials serves a crucial role in protecting the machine from electrical hazards. Compared to organic polymers, inorganic insulation materials offer superior resistance to high temperatures and chemical degradation. However, ceramic densification, requiring high sintering temperatures, can lead to thermal damages due to interdiffusion or oxidation. Aerosol deposition (AD) is a dry, high-velocity spray method for manufacture of dense ceramic coatings at room temperature directly from ceramic powders, capable of retaining the properties of the constituent materials. In the present work, alumina coatings were deposited on poly ether ketone substrates by AD. Polished stainless steel electrodes were employed for electrical endurance test until sample breakdown occurred after successive partial discharges. The coated samples showed significantly improved electrical endurance under a 1 kV, 1 kHz voltage source, surviving approximately 70 times longer than the uncoated reference material in an accelerated lifetime test. Further microstructural characterization by SEM and TEM performed after breakdown allowed for the study of high-voltage failure mechanisms in the ceramic coatings.

(ICACC-P108-2024) Exploring the Dielectric and Conduction Characteristics of $Sr_7Mn_4O_{15}$

R. Baranwal*[1]

1. Indian Institute of Technology(BHU), PHYSICS, India

$Sr_7Mn_4O_{15}$ has a complex layered structure that is susceptive to temperature. Its structure is a double octahedra version of the K_2NiF_4 structure. The electrical properties of this compound have been investigated at high temperatures for application as a Negative Temperature Coefficient (NTC) Thermistor. A detailed study of its electrical and dielectric properties has never been reported before. Therefore, in this work, we have attempted to synthesize $Sr_7Mn_4O_{15}$ and study electrical and dielectric properties in depth. Single phase $Sr_7Mn_4O_{15}$ has been prepared by the conventional ceramic route at 1400C for 12 h. Rietveld refinement of x-ray diffraction data confirmed monoclinic structure with a space group $P12_1/C$ 1 and lattice constants a=b= 5.22639 Å, and c= 11.43164 Å. The UV-Vis-IR spectrum has been recorded in the wavelength range 200-1200 nm, optical band gap is found to be 1.57eV. The A sintered sample's AC electrical conductivity and dielectric properties have been studied in the frequency range 20Hz–2MHz for temperatures ranging from 40C to 500C. A dielectric relaxation phenomenon has been observed. Dielectric loss in the sample loss mainly due to DC conduction. AC conductivity of the sample follows Johnscher's Power law which might be due to the hopping conduction mechanism.

(ICACC-P109-2024) Processing and Testing of Lightweight Composite Conductors

C. Hernandez*[1]; A. S. Almansour[2]; M. Lizcano[2]; D. Santiago[2]

1. University of Puerto Rico-Mayaguez, Engineering Sciences and Materials, Puerto Rico
2. NASA Glenn Research Center, USA

The development of sustainable electrified aircraft propulsion (EAP) adds more wiring, which increases aircraft's overall weight. Moreover, the electrical conductivity of the wires is inversely proportional to electrical systems' power losses and directly proportional to electric motors efficiency. Therefore, designing high-conductivity lightweight conductor materials for electrical wires can address the above challenges, leading to more sustainable

EAP. This work is focused on fabricating high conductivity, lightweight copper-coated carbon nanotubes (CNT) composite wires via electroplating. Seven batches of copper electroplated carbon nanotube (Cu/CNT) yarns were reproduced by varying electrode wiring, power source, and plating solution. Four-probe electrical resistance (ER) monitoring was used to measure the electrical conductivity of the unannealed composite wires. The variations in the deposited copper thicknesses and electrical conductivities were correlated with the samples' positions in the electroplating experiment and electrode connections. Samples fabricated exhibited electrical conductivities as high as 61.8 MS/m and 6.55% percent change compared to the International Annealed Copper Standard (IACS). The results from processing, characterization, and analysis give insights into possible optimization paths.

Thursday, February 1, 2024

FS1 Bioinspiration/Green Processing & Related Technologies of Advanced Materials

Focused Session 1: Bioinspiration, Green Processing, and Related Technologies of Advanced Materials

Room: Ponce de Leon
Session Chair: Zhao Qin, Syracuse University

8:30 AM

(ICACC-FS-007-2024) On Nano- and Mesoscale Structure and Composition of Dental Enamel (Invited)

D. Joester*[1]

1. Northwestern University, Materials Science and Engineering, USA

Dental enamel is composed of hydroxylapatite crystallites, thousands of which are bundled into rods that are organized in a three-dimensional weave. This architecture provides great fracture resistance and a much-enhanced fatigue life. The susceptibility of enamel to acid corrosion is dependent on the presence of Mg^{2+}, Fe(III), CO_3^{2-}, and F^-. However, imaging the distribution of these minor components in enamel has remained challenging. Using UV-laser pulsed atom probe tomography (APT), in combination with correlative imaging and spectroscopy techniques, we recently discovered that they are enriched in an amorphous intergranular phase that cements together rodent enamel crystallites. Here, I will report on our recent discovery that unlike in rodent enamel, there are highly characteristic gradients of these ions across individual crystallites in human enamel as well. I will discuss implications for the mechanical properties of enamel, its dissolution behavior, and tooth development. I will further describe a sensitive approach for mapping crystallite properties and composition across enamel rods and the interrod enamel that separates them, using X-ray diffraction with a sub-micrometer probe size. Our measurements indicate that there are systematic differences in composition between rod and interrod enamel, but also hint at other factors that control inter-individual variation.

9:00 AM

(ICACC-FS-008-2024) The role of additives on the thermodynamics and kinetics of nucleation, crystal growth and dissolution (Invited)

J. Tao*[1]

1. Pacific Northwest National Lab, USA

The mechanism of the interplay between additives and crystals is a long-term mystery in the field of crystallization. Monitoring crystallization processes in real time is challenging due to the sub-nanometer scale and highly dynamic nature of the incorporation/detachment events. As a result, to date material synthesis has been advanced mainly by empirical optimization of different synthesis parameters (concentration, temperature or solvent) rather than by rational manipulation based on thermodynamic or kinetic controls. As a result, there is a significant knowledge gap in understanding 1) how additive interact with growth units resulting in changes in the thermodynamics and kinetics of crystallization, and 2) how additives affect elementary processes such as transport, adsorption, and desolvation of growth unit at interface. From the energetic viewpoint, incorporation of growth units at the additive-crystal interfaces depends on the balance between the kinetic factor—the desolvation or reorganization energy at interfaces—and the thermodynamic factor—the structural changes created by incorporation on the crystal surfaces.

9:30 AM

(ICACC-FS-009-2024) From nanoscale assembly processes to toughening textures in biomimetic and biogenic hybrid ceramics (Invited)

S. E. Wolf*[1]

1. Friedrich-Alexander-University Erlangen-Neurnberg, Department of Materials Science and Engineering, Chair of Glass and Ceramics, Germany

Biominerals are biogenic hybrid ceramics evolutionary honed to high-performance materials with high functional density and damage tolerance. Their unique traits render them a source of inspiration for the novel design of materials and syntheses, motivating an in-depth analysis of their process-structure-properties relationships. In the biosynthesis processes of these "bioceramics", transient amorphous phases play a decisive role that subsequently crystallizes in a shape-preserving manner. Biominerals are praised for their exquisite control over this process, reminiscent of glass-ceramics formation but occurring at ambient conditions. While earlier studies highlighted near-to single-crystalline biominerals, complex polycrystalline biominerals dominate but still are challenging to appreciate. Here, we propose that (bio)ceramics with varying crystal textures are functionally graded materials with enhanced endurance against shard and blunt contact damage. We show that the formation of such complex crystallographic textures is inherent to their nonclassical formation via nanoparticle accretion processes and can be mimicked by biomimetic mineralization processes. This unforeseen process-structure-properties relationship highlights the untapped potential of (bio)synthetic approaches that employ nanoparticle-based and nonclassical routes to generate functional materials.

FS2 Advanced Materials for Thermoelectric and Thermionic Energy Conversion

Focused Session 2: Advanced Materials for Thermoelectric and Thermionic Energy Conversion

Room: Coquina C
Session Chair: Michitaka Ohtaki, Kyushu Daigaku

8:30 AM

(ICACC-FS2-001-2024) Interface and Grain Boundary Effects on Thermal and Electrical Transport (Invited)

J. Snyder*[1]

1. Northwestern University, Department of Materials Science and Engineering, USA

Grain boundaries have a remarkable effect on the thermal and electrical transport properties of polycrystalline materials but are often ignored by prevailing physical theories. Grain boundaries and interfaces can adversely alter or benefit the properties of Power Electronics, Solar Cells, Batteries and Thermoelectrics via interfacial electrical and thermal resistance and even an interfacial Seebeck effect. New scanning thermal reflectance techniques can image the thermal resistance of interfaces and boundaries directly. The thermal conductivity suppression at grain boundaries can even be imaged showing that different grain boundaries can have very different thermal resistances with high energy grain boundaries having more resistance and low energy boundaries having lower thermal resistance. Electrical grain boundary resistance can be so high in some thermoelectric materials; it is the dominant property that limits zT. While small grains are usually considered beneficial for thermoelectric performance due to reduced thermal conductivity, Mg_3Sb_2 based thermoelectric materials, so far at least, contradict that trend. Indeed, atomic segregation has been recently observed at the nanometer scale in grain boundaries in many materials suggesting interfacial or complexion phases should be specifically considered when understanding thermoelectric materials.

9:00 AM

(ICACC-FS2-002-2024) Reconsidering the Physics of High-temperature Energy Conversion (Invited)

A. Nojeh*[1]

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

The conversion of high-temperature heat to electricity is the backbone of modern society. This is likely to continue with both traditional sources of heat and new ones such as contentrated sunlight and thermal energy storage systems. Given the cost, complexity, and lack of scalability of turbine-based generators, static heat engines may eventually be needed. Thermionic energy converters are the ultimate such devices, where electrons directly move from a hot emitter to a cold collector. However, their performance to date has been far below what thermodynamics allows. Efficient conversion requires tight control of the energy exchange channels: to promote electron transfer while minimizing the heat transfer via various mechanisms. Historically, at high temperatures, these transport phenomena have been treated semi-classically, without a direct link to the specific materials physics of the emitter and collector. Underlying this approach has been the understanding that quntum effects are washed out at high temperatures. However, this may not always be the case. Using examples from theory and experiment, I will make the case that to advance thermionic energy conversion will require an integrated approach to treating materials and device physics which taps into our knowledge of quantum phenomena and the use of advanced theoretical and experimental techniques developed in various areas of condensed matter physics.

9:30 AM

(ICACC-FS2-003-2024) Weighted Mobility Ratio Engineering for High-Performance Bi-Te-based Thermoelectric Materials (Invited)

S. Kim[1]; S. Kim[3]; H. Kim*[1]; K. Lee[2]

1. University of Seoul, Republic of Korea
2. Yonsei University, Republic of Korea
3. SungKyunKwan University, Republic of Korea

Thermoelectrics, which can generate electricity from a temperature difference, or vice versa, is the 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 by a figure of merit of materials, zT, which is utterly related to the electronic and thermal transport characteristics, this review aims to elucidate physical parameters that should be considered to understand transport phenomena in semiconducting materials. It is found that a weighted mobility ratio of majority and minority carrier bands is an important parameter that determines zT. For nanograined Bi-Sb-Te alloy, we demonstrated the unremarked role of this parameter on temperature-dependent electronic transport properties. Our analysis shows that the control of the weighted mobility ratio is a promising way to enhance the zT of narrow band gap thermoelectric materials.

10:20 AM

(ICACC-FS2-004-2024) First Principles Assessment of Materials for Direct Energy Conversion (Invited)

J. C. Goldsby*[1]

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

Solid-state energy conversion devices offer the possibility for highly efficient and reliable operations. In a thermionic material, heat-to-electric potential conversion results from the thermal gradient and the work functions of the electrode collector relative to the emitter electrode. Combined with a thermoelectric, high efficiencies are possible. Computational methods offer an efficient and systematic manner to design new materials and guide their development. Density functional theory allows us to determine the suitability of various materials as thermoelectric and thermionic converters. To this end, low-work function materials are highly sought as thermionic materials, while high values of ZT work best for thermoelectrics. In this presentation, we studied mixed oxide-pyrochlore ceramics for their potential as a thermoelectric material. We also evaluate several oxides and intermetallic emitter candidate materials as thermionic converters through their respective work functions. These calculations used a projector-augmented wave (PAW) method. A commercial code (Materials Design Inc.) MedeA incorporated the Vienna Ab-initio Simulation Package (VASP) as the computational engine. This study makes predictions and comparisons between experimental and theoretical properties. In addition, the calculated phonon dispersion curves and elastic constants allowed a mechanical stability prediction.

10:50 AM

(ICACC-FS2-005-2024) The thermoelectric properties and the chemical bonding of cubic-GeTe material from first principles

M. Zebarjadi*[1]; S. Das[1]

1. University of Virginia, Electrical and Computer Eng., USA

A subgroup of chalcogenides has shown excellent thermoelectric (TE) properties as a result of their unique electronic and phononic properties including high valley band degeneracy, large dielectric function, and strong lattice anharmonicity attributed to their meta-valent bonding. GeTe having a maximum ZT of 2.7 ($Ge_{0.87}Y_{0.02}Sb_{0.10}Ag_{0.01}Te$) is a lead-free, eco-friendly low band gap TE material. Pure GeTe goes through a phase transition from low-temperature rhombohedral to high-temperature cubic phase at 700K owing to the Peierls distortion in the metavalent bonding mechanism. This anomalous bonding nature in GeTe enables us to optimize the TE properties by adjusting the atomic interactions by substitutional alloying. Exceptional TE performance was obtained in GeTe at mid-high temperature regions through charge transfer engineering on chemical bonding by means of alloying GeTe with Pb, Sb, Bi, Mn, In, etc. Although this material has been studied extensively, theoretical studies on transport properties based on the first principle approach to map the nature of the bonds to alloying and TE properties are missing which is the focus of this talk.

11:10 AM

(ICACC-FS2-006-2024) Interfacial Engineering Using Additive Manufacturing to Decouple Electrical and Thermal Conductivity for Next-Generation Thermoelectrics (Invited)

D. Madan*[1]; J. Huang[1]; J. Lombardo[1]; R. Ambade[2]; P. Banerjee[1]; S. Kulkarni[1]; B. Brooks[1]

1. University of Maryland Baltimore County, Mechanical Engineering, USA
2. Hanyang University, Seoul, Republic of Korea, Republic of Korea

Additive manufacturing has been investigated as a more time, energy, and cost-efficient method for fabricating thermoelectric generators (TEGs). This work investigates the synergistic effect of four factors – a small amount of chitosan binder (0.05wt%), a heterogeneous particle size distribution, the application of mechanical pressure, and thickness variation – on the performance of p-$Bi_{0.5}Sb_{1.5}Te_3$ (p-BST) and n-$Bi_2Te_{2.7}Se_{0.3}$ (n-BTS) TE composite films. The combination of these four factors controls the micro and nanostructure of the films to decouple their electrical and thermal conductivity effectively. This resulted in figures of merit (ZTs) (0.89 and 0.5 for p-BST and n-BTS, respectively) comparable to other additive manufacturing methods despite eliminating the high-temperature, long-duration curing process. The process was also used to fabricate a 6-couple TEG device which could generate 357.6 μW with a power density of 5.0 mW/cm^2 at a temperature difference of 40 K. The device demonstrated air stability and flexibility for 1000 cycles of bending. Finally, the device was integrated with a voltage step-up converter to power a LED and charge and discharge capacitor, demonstrating its applicability as a self-sufficient power source.

11:40 AM

(ICACC-FS2-007-2024) Design and Synthesis of Multi-anion Chalcogenides for Thermoelectric Applications (Invited)

J. Hodges*[1]

1. Pennsylvania State University, Chemistry, USA

Metal chalcogenides underpin a wide variety of energy-related applications and are ideal systems for probing lattice dynamics and fundamental transport phenomena. In this talk I will discuss chemical approaches to optimizing narrow-gap chalcogenides for thermoelectric applications. First, I will show how the thermoelectric performance of lead chalcogenides can be enhanced using a combination of aliovalent and isovalent substitutions. These are powerful techniques that can be used as a handle for tuning the Fermi level and band structure of IV-VI semiconductors. The second part of the talk will focus on the importance of exploratory synthesis in the field of thermoelectrics. Here, I describe a series of fully ordered multi-anion chalcogenides that exhibit unusual transport properties, including suppressed phonon transport and p-type metallic conductivity. These quaternary systems are used as a platform for demonstrating a unique method for tuning electronic transport, while also providing synthetic guidelines for targeting other multi-anion systems.

FS3 Nanostructures and Low-Dimensional Materials for Chemical Sensors

Focused Session 3: Chemical sensors using na

Room: Flagler A
Session Chairs: Vincenzo Guidi, University of Ferrara;
Kengo Shimanoe, Kyushu University

8:30 AM

(ICACC-FS3-007-2024) VOC-Sensing Properties of YSZ-Based Gas Sensors Attached with Au-Based Electrodes (Invited)

T. Ueda*[1]; T. Hyodo[1]

1. Nagasaki University, Graduate School of Engineering, Japan

We have studied yttria-stabilized zirconia (YSZ)-based solid-electrolyte gas sensors and reported that the addition of CeO_2 to the Au sensing electrodes (SEs) improved the toluene responses. The sensor response to toluene is generally determined by mixed potential arising from the balance of the electrochemical activity between the toluene oxidation and the oxygen reduction at triple-phase boundaries (TPBs) consisting of target gases, solid electrolytes, and electron conductors at the interface between SE and YSZ. In addition, a certain amount of toluene is catalytically oxidized during gas diffusion in SE. In this study, we fabricated thin-film CeO_2-added Au SEs (thickness: 30-150 nm) by using spin-coating method to decrease the amount of toluene, which catalytically oxidized during gas diffusion in SE. The magnitude of response (ΔE) to 50 ppm toluene of the sensors attached with 8 wt% CeO_2-added Au SE increased with a decrease in the SE thickness. On the other hand, ΔE of the sensors using 24 wt% CeO_2-added Au SE increased with an increase in the SE thickness, and the sensor attached with the thickest 24 wt% CeO_2-added Au SE showed the largest response (ca. 212 mV, at 450°C). The sensing mechanisms were discussed on the basis of the I-E characteristics and the electrochemical impedance properties.

9:00 AM

(ICACC-FS3-008-2024) New Developments in Microfabrication of Solid Electrolyte Gas Sensors (Invited)

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., Japan

Oxygen sensors for air-fuel ratio control using stabilized zirconia are widely employed in various combustion systems. These oxygen sensors require mechanical strength and resistance to thermal shocks since they come into direct contact with high-temperature combustion gases. Therefore, it is uncommon to find solid electrolyte sensors configured as thin films. However, for gas measurements in the atmosphere, such as O_2, CO_2, NO_2 and CO, there is a need to reduce the power consumption of the sensors, which has led to considerations for miniaturizing solid electrolyte gas sensors. Solid electrolyte gas sensors employ various detection methods, including electromotive force, limiting current, mixed potential, and short-circuit current, which are selected based on the type of gas to be detected and its concentration range. When solid electrolytes are made into thin films, the resistance of the film decreases due to ion conduction, enabling operation at lower temperatures, which was previously considered challenging. One critical aspect of producing thin films is achieving uniform composition. Without uniform composition, sufficient ion conductivity cannot be ensured, making sensor operation difficult. In this presentation, we report "highly c-axis-oriented apatite-type lanthanum borosilicate (c-LSBO) thin films" with uniform composition and their application in micro solid-state electrolyte gas sensors.

9:30 AM

(ICACC-FS3-009-2024) Chip-less wireless wearable chemical sensor using epitaxial freestanding piezoelectric nanomembrane (Invited)

Y. Kim*[1]

1. University of Cincinnati, Department of Electrical and Computer Engineering, USA

Recent advances in flexible and stretchable electronics have led to a surge of electronic skin (e-skin)–based health monitoring platforms. Conventional wireless e-skins rely on rigid integrated circuit chips that compromise the overall flexibility and consume considerable power. Chip-less wireless e-skins based on inductor-capacitor resonators are limited to mechanical sensors with low sensitivities. I will talk about a chip-less wireless e-skin based on surface acoustic wave sensors made of freestanding ultrathin single-crystalline piezoelectric gallium nitride membranes. Surface acoustic wave–based e-skin offers highly sensitive, low-power, and long-term sensing of strain, ultraviolet light, and ion concentrations in sweat. We demonstrated weeklong monitoring of pulse. These results present routes to inexpensive and versatile low-power, high-sensitivity platforms for wireless health monitoring devices.

10:20 AM

(ICACC-FS3-010-2024) Wide Dynamic Range CO_2 Sensor through Chemoresistive Semiconductors (Invited)

A. Rossi[1]; B. Fabbri[1]; E. Spagnoli[1]; A. Gaiardo[2]; M. Valt[2]; V. Guidi*[1]

1. University of Ferrara, Physics and Earth Sciences, Italy
2. Bruno Kessler Foundation, Italy

Carbon capture and storage is critical to climate change policies and strategies aimed at reducing global warming under the Paris Agreement. Thereby, the development of miniaturized devices is pivotal to map CO_2 in IoT applications, such as smart home/buildings, smart agriculture, smart cities and industrial IoT. Nowadays, most widely used devices for monitoring CO_2 are based on nondispersive infrared sensors. However, such devices are power-consuming and are somewhat expensive. Solid-state gas sensors are a viable alternative, although materials proposed for chemoresistive devices have yet to demonstrate appealing CO_2 detection capabilities. In this work, an innovative nanostructured semiconducting powder based on indium oxide doped with Na is proposed as functional material for chemoresistive CO_2 sensors [1]. A complete material characterization was performed from a morphological, structural, chemical, and optical point of view, to highlight the impact of Na on the In_2O_3 properties. Then, a deepened electrical investigation was carried out to study the sensing performance towards CO_2 (sensitivity, selectivity, stability, humidity effect). The film reversibly detected CO_2 within 250-5000 ppm at 200 °C. Humidity exhibited negligible impact on the sensing capabilities from 17 to 65 RH%, even at low CO_2 concentrations. All of these characteristics make $Na:In_2O_3$ based sensors suitable for applications.

10:50 AM

(ICACC-FS3-011-2024) Molybdenum Disulfide-Conducting Polymer Composite Structures for Electrochemical Biosensor Applications

A. Kumar*[1]

1. University of South Florida, Mechanical Engineering, USA

Lactic acid is widely existing in human bodies, animals and microorganisms. Nanocomposites is one of the best materials used for biosensor because their wonderful conductivity, optical and electrochemical properties. In the study, MoS_2 and polypyrrole (PPY) are used for the composite material electrode. To determine whether lactate oxidase (LOD) was helpful for the biosensor's detective properties, both PPY-MoS_2 film with LOD and PPY-MoS_2 film without LOD are being tested. The fourier transform infrared spectroscopy (FTIR) and Raman spectroscopic techniques have been used to understand the chemical bonds in the nanocomposite film. The X-ray diffraction (XRD) technique has been performed to understand the crystallographic structure of the MoS_2-PPY film. The morphologies were confirmed by scanning electron microscopy (SEM). The UV-vis spectroscopy has been used to determine the band structure of composite film. Cyclic voltammetry (CV), chronoamperometry (CA), and electrochemical impedance spectroscopy (EIS) were used to analyze in different concentration of solution, under different scan rate to obtain stability and work efficiency. These results were compared with PPY-MoS_2 film with and without lactate oxidase conditions. The chronoamperometric technique has been used to detect the concentration of lactic acid.

11:10 AM

(ICACC-FS3-014-2024) Synthetic receptors in (bio-)sensing architectures: The dusk of antibodies? (Invited)

F. Polo*[1]; G. Moro[1]; L. Masutti[1]; E. Sossich[1]; S. Tartaggia[2]; Y. Mazzocato[1]; A. Angelini[1]

1. Ca' Foscari University of Venice, Molecular Sciences and Nanosystems, Italy
2. National Research Council of Italy, Istitute of Biomolecular Chemistry, Italy

The interest in synthetic bioreceptors to develop (bio-)sensing platforms has increased quite rapidly in recent years. Ligand binding assay, which employs a receptor to capture the analyte, relies on the affinity of the receptor toward the binding ligand. In this respect, antibodies (Abs) are still playing a fundamental role. However, they suffer of high production costs to meet the requirements of specificity and selectivity for each analyte of interest. Immunoassays, in fact, rely on a couple of Abs that selectively bind two different epitopes of the same protein cancer biomarker. This aspect is crucial to develop biosensing platforms. Moreover, it is usually very difficult for researchers worldwide to find commercially available Ab couple that meet such requirements. Furthermore, Abs are not particularly useful to detect small molecules, such as chemotherapeutic drugs, as the detection system may imply the use of highly sophisticated equipment. However, there are innovative and valuable solutions represented by aptamers and peptides. In this presentation, we will show few interesting and recent applications of aptamers and of a new class of bicyclic peptides. The former ones have been employed to monitor a chemotherapeutic drug in plasma in an SPR-based sensing platform. Whereas the latter ones have been devised in an electrochemical sensing platform to detect a cancer protein biomarker.

FS5 High Voltage Materials for Advanced Electrical Applications

Focused Session 5: High Voltage Materials for Advanced High Power Electrical Applications

Room: Ballroom 4

Session Chairs: Marina Gandini, Prysmian Group; Maricela Lizcano, NASA Glenn Research Center

8:30 AM

(ICACC-FS5-001-2024) Electric Field Neutralization: Rethinking Insulation for WBG Power Electronics (Invited)

C. Park*[1]

1. University of Wisconsin-Milwaukee, USA

Decarbonizing our society through electrification requires high-voltage (HV) power electronics reliably operating at high temperatures. As power semiconductor technology outpaces packaging and insulation materials, ensuring the dielectric integrity of power electronic devices and systems is becoming increasingly challenging. Studies have shown that recurring, steep voltage pulses induced by WBG devices operating at high temperatures promote more significant and frequent partial discharge (PD), degrade electrical insulators at a higher pace, and pose a greater risk to the modernizing grid and electrifying transportation systems. While most dielectric materials and electrical insulation techniques have managed electric fields through electric field grading, the electret-based mitigation approach proposed neutralizes electric fields to mitigate PD. As the electrical equivalent of permanent magnets, electrets generate electric fields without a voltage source. We proposed to use electrets to counter PD-causing electric fields and have reduced PD magnitude and rate under recurring, steep voltage transients induced by power electronic switches. Though proven effective in mitigating PD, the successful adoption of electrets into power modules as a packaging component depends on their high-temperature survivability. This talk introduces an electret-based technology that mitigates PD at high temperatures.

9:00 AM

(ICACC-FS5-002-2024) Research in High Voltage Power Transmission Materials for Aerospace Systems (Invited)

M. Lizcano*[1]

1. NASA Glenn Research Center, USA

This presentation gives an overview of recent materials research activities carried out at NASA Glenn Research Center. The background and motivation for the development of thermally conductive electrical insulation for a high voltage (HV) turboelectric aircraft concept is briefly discussed. A summary of the challenges and technology gap associated with HV aerospace electrical systems is also covered as well as the development of electrical insulation, engineered ceramic fillers and HV test capabilities. The research has expanded to include the development of a lightweight copper/carbon nanotube composite conductor. Applications for these materials include hybrid electric aircraft, all electric aircraft, spacecraft, electric propulsion systems, and future power distribution and transmission systems for Lunar and Martian destinations.

9:30 AM

(ICACC-FS5-003-2024) Electrical Endurance to Extrinsic Aging of Polyphenylsulfone-Boron Nitride Composite Insulation for Aeronautical Power Systems (Invited)

M. Shafiq*[1]; S. Myneni[1]; G. Montanari[1]; M. Lizcano[2]; T. Williams[2]

1. Florida State University, USA
2. NASA Glenn Research Center, USA

Various insulating materials which can be potentially used in energy application, especially in electrified transportation, are investigated in this paper. They are based on Polyphenylsulfone (PPSU) matrix filled by micro and nano hexagonal Boron-nitride particles. Our study focuses on electrical properties, particularly endurance to partial discharges. Upon developing an original testing procedure and adequate partial discharge inception modeling, it is shown that the h-BN micron and nano fillers can significantly improve the dielectric strength and resistance of PPSU to extrinsic aging, even in comparison with the insulating material typically used for electrified transportation, i.e., corona resistant Kapton. Also, a new diagnostic marker, extracted from partial discharge monitoring and analytics obtained by an innovative software, i.e., the likelihood surface discharge identification, seems to be very promising for the dynamic residual life prediction for an insulation system subjected to surface discharges.

10:20 AM

(ICACC-FS5-004-2024) Electric Field Tailoring in Power Modules By Field Grading Materials

O. Faruqe*[1]; P. C. Saha[1]; A. Juberi[1]; C. Park[1]

1. University of Wisconsin-Milwaukee, USA

In this study, we conduct a comparative analysis of the electric stress reduction capabilities between nonlinear field grading materials (NLRFG) and materials with field-dependent permittivity (FDP). We performed experiments to characterize both the nonlinear conductivity and permittivity of the fabricated materials. Subsequently, we created finite element analysis (FEA) models of the power module substrate and integrated the material properties obtained from the experiments. Electric field magnitudes near the triple points of an power module with and without the incorporation of field grading materials are compared under both DC and AC waveforms, with a frequency range spanning from 0 Hz to 5000 Hz. Based on our findings, the inclusion of NLRFG results in a reduction of the electric field near the triple point by approximately 98%, 63%, 45%, 38%, and 21% for frequencies of 0 Hz, 60 Hz, 500 Hz, 1000 Hz, and 5000 Hz, respectively. In contrast, the incorporation of FDP leads to a reduction in the electric field at the same point by approximately 22%, 32.2%, 32.1%, 32%, and 32% for the same frequencies. These results suggest that FDP materials are less effective in DC compared to NLRFG materials. Furthermore, it is noteworthy that the field-reducing performance of FDP is not affected by the increase in frequency, while the effectiveness of NLRFG materials decreases as the frequency rises.

10:40 AM

(ICACC-FS5-015-2024) Lightweight, Multifunctional Nanocomposites for High-Voltage Insulation on the Moon

L. Alexis[1]; J. Lee[1]; G. Alvarez[1]; D. Santiago[2]; Z. Tian*[1]

1. Cornell University, USA
2. NASA Glenn Research Center, Materials and Structure Division, USA

Sustained human presence on the Moon requires advances in mission-enabling cables and wires to realize a lunar electrical grid. The lunar environment presents challenges such as exposure to cosmic and UV radiation, extreme temperature deltas, and the electrostatic lunar exosphere and regolith. The electrical performance of high-voltage power transmission (HVPT) cables is closely associated with the properties of the cable insulation. The objective of our project is to develop lightweight, multifunctional nanocomposites that can simultaneously act as an insulation and shielding layer for high-voltage lunar cables. The resulting lightweight material could improve thermal conductivity, dielectric strength, and mechanical durability while providing electromagnetic interference shielding. In this talk, I will share our recent developments on boron nitride (BN) based polymer nanocomposites, with a particular focus on its thermal conductivity.

11:00 AM

(ICACC-FS5-006-2024) Mitigating Space Charge Induced Breakdown With Electrets Under Square Voltage Pulse

P. C. Saha*[1]; O. Faruqe[2]; A. Juberi[2]; C. Park[1]

1. University of Wisconsin-Milwaukee, USA
2. University of Wisconsin-Milwaukee, Electrical Engineering, USA

In high voltage direct current (HVDC) systems, charge carriers are introduced into solid insulators, leading to their accumulation and the generation of space charge. This space charge distorts electric fields and diminishes the effective thickness of dielectric materials. In this research, we empirically validate the effectiveness of incorporating electrets to mitigate space charge-induced breakdown when subjected to square voltage pulses. A square voltage of 5kV was applied to the conventional dielectric material, polyvinylidene fluoride (PVDF), to induce space charge injection and accumulation, which, in turn, reduced the effective thickness and consequently lowered the breakdown voltage of the dielectric. In contrast, electrets prevent space charge injection and accumulation within the dielectric, thereby averting space charge-induced breakdown. The comparative analysis of breakdown voltage reveals that electrets successfully alleviate space charge-induced breakdown when exposed to square voltage pulses.

11:20 AM

(ICACC-FS5-007-2024) Boron Nitride Fibers as a Potential Solution for High Voltage Insulation

K. Vailonis*[1]; D. Santiago[1]; M. Lizcano[1]; T. Sabo[2]

1. NASA Glenn Research Center, USA
2. Case Western Reserve University, USA

The aerospace industry is quickly evolving, creating a need for novel multifunctional materials. The push for electric aviation will require optimization of each component to ensure the success and sustainability of the aircraft. One aspect in need of optimization, is low mass, flexible insulation for the high voltage power transmission. Insulation materials will need to have high thermal conductivity, high electrical resistivity, and high chemical stability. Composite materials are currently studied to solve this technology challenge. Boron nitride nanomaterials have shown promise when utilized as a filler in novel composite materials since they meet the criteria for high voltage insulation. The goal of this project is to apply the Forcespinning^{TM} technique to form polymeric precursor fibers with additional boron loadings and then convert to boron nitride fibers via a controlled heat treatment process. These fibers will then be incorporated into polymer composites to study their success as insulation materials. Chemical analysis of the fibers and composites will be studied and reported here.

11:40 AM

(ICACC-FS5-008-2024) Engineered Hexagonal Boron Nitride:Titanium Dioxide Composites for High Voltage Insulation

S. P. McDarby*[1]; D. Santiago[1]; M. Lizcano[1]

1. NASA Glenn Research Center, USA

Hexagonal boron nitride (hBN) and Titanium dioxide are both ceramic materials with widespread use in commercial and consumer applications in everything from cosmetics and paint to deep space satellite components. As a dielectric material, hBN is electrically insulating, thermally conductive, and stable to most temperatures and environments. Titanium dioxide is also a good electrical insulator with a wide bandgap and large dielectric constant, as well as high temperature tolerance and chemical stability. This presentation covers our recent efforts to combine the best properties of hBN and titanium dioxide to produce an intercalated hBN:titanium dioxide composite which was then further processed and incorporated into polymer composites, as well as a standalone ceramic material for testing as a novel insulation material.

S10 Modeling and Design of Ceramics and Composites

SYMPOSIUM 10: Modeling and design of ceramics and composites

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

8:30 AM

(ICACC-S10-008-2024) Correlation between the shape of the particles and elastic properties of the particle reinforced composite (Invited)

P. Happ[1]; R. Piat*[1]

1. Darmstadt University of Applied Science, Germany

Numerical evaluation of the elastic properties of particle reinforced composite was provided. For understanding the effects of the particle shapes onto the overall linear elastic properties of the two-phase composites the particles with polyhedral, periodic undulated and other shapes were modelled using analytical functions. Creation of some shapes, like polyhedral, is known from literature but Laplace's spherical harmonics as also Goursat's surface and some other one were used first time to create new particle shapes. Elastic properties of the composites with different particle shapes were calculated using finite element analysis. Good agreement of the present and obtained with Mori-Tanaka and Lielens as also known from literature results is achieved. The mesh independence of the modeling was demonstrated. The dependence of the effective Young's moduli of the composite from shape and corresponding surface-to-volume ratio (for constant volume) of the particles was studied. It was observed the increasing of effective Young's moduli with increasing of the surface-to-volume ratio of the particles. It was also shown that parameters like surface curvature and sharpness of edges describing the particles geometry have also essential influence on effective elastic properties of the composite.

9:00 AM

(ICACC-S10-009-2024) Transition-Metal Diboride Thin Films Investigated by X-ray Spectroscopy and Ab-Initio Electronic-Structure Calculations (Invited)

M. Magnuson*[1]

1. Linköping University, Department of Physics, Chemistry and Biology (IFM), Sweden

Transition metal-borides are known to exhibit interesting materials properties from superhardness to superconductivity with high thermal and chemical stability. These materials have potential for the next generation of hard, wear- and corrosion-resistant coatings. In particular, this is the case for borides with an AlB_2 structure (C32), where the B atoms form honeycombed, graphite-like sheets that are interleaved between hexagonal close packed TM atoms giving unique properties including high melting point, hardness, and corrosion resistance, yet limited oxidation resistance, combined with high electrical conductivity. Industrial applications are growing rapidly as TMB_2 begin competing with conventional refractory ceramics like carbides and nitrides, including pseudo-binaries such as $Ti_{1-x}Al_xN$. From a combination of analysis with XPS, XRD, XANES, and EXAFS compared to ab-initio electronic structure calculations, the local chemical bonding and structural properties were investigated. The underlying chemical bonding, orbital overlap, and electronic structure are correlated to the mechanical properties, resistivity, and high-temperature properties unique to this class of materials. TMB_2 films have a multitude of applications not solely as bulk materials, but also as protective coatings and electrically conducting high-temperature stable thin films.

9:30 AM

(ICACC-S10-010-2024) Hierarchical thermal transport and dual-phonon theory in complex-structure ceramics (Invited)

Y. Luo*[1]

1. Institute of Metal Research, Chinese Academy of Sciences, China

The mechanism of thermal transport for complex-structure ceramics is a long-standing challenge in the community of thermal management. Some complex-structure materials exhibit glass-like temperature (T) dependence of lattice thermal conductivity (κ_L), particularly at high-temperature ranges, which contradicts the κ_L~T dependency of crystallines. This intriguing puzzle calls for theoretical and computational advances beyond the traditional Peierls-Boltzmann transport theory. In this study for some prototype complex-structure ceramics, such as rare-earth silicates and pyrochlores, and perovskites, etc., we find that there coexist well-defined phonon modes together with large amounts of strongly perturbated modes whose mean free path is smaller than or comparable to the interatomic spacing. A dual-phonon thermal-transport theory is proposed to account for combined contributions from both propagation and diffusion thermal-transport channels, which yields better description of the κ_L~T dependency that agrees well with experiments. It is found that the diffusion channel is important especially at high-temperature ranges. These results are expected to advance the theoretical understanding and computational paradigm for the complex-structure ceramics, and cast light on the materials discovery and thermal-transport control in the fields such as thermoelectrics, high-temperature protective coatings, etc.

10:20 AM

(ICACC-S10-011-2024) A DIC-Based Study of Thermal Stress-Strain Behavior of Cu on Metallized Si_3N_4 Substrate under Thermal Cycling

M. Ngo*[1]; H. Miyazaki[1]; K. Hirao[1]; T. Ohji[1]; S. Ozaki[2]; M. Fukushima[1]

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

Cu-Si_3N_4 metallized substrate has been evaluated with excellent reliability, durable up to 1000 cycles, in the cycling test from -40 to 250 °C. While finite element methods estimated thermal stress-strain during cycling, experimental data for stress-strain of the metallized substrate remained limited. In this study, a Digital Image Correlation (DIC) method was employed to measure the thermal strain of a fully Cu-coated Si_3N_4 substrate in various consecutive thermal cycles from 1~2 to 199~200. The thermal strains exhibited in hysteresis curves, which expanded slightly with cycles. By incorporating the coefficients of thermal expansion (CTE) of plain Cu and Si_3N_4, both the thermal stress and strain in the Cu layer atop the substrate was computed. The stress-strain curves of the Cu layer exhibited that the yield stress of Cu increased with the number of cycles, which was attributed to the cyclic hardening of the Cu layer. These findings contribute to a deeper understanding of the thermal behavior of metallized substrates under varying conditions. This presentation is based on results obtained from a project “Development of a Technology Base and Applied Technologies for the Manufacturing Processes of Next-Generation Advanced Ceramics” (JPNP22005) commissioned by the New Energy and Industrial Technology Development Organization (NEDO).

10:40 AM

(ICACC-S10-013-2024) Numerical prediction of strength scatter of ceramics: Forward analysis of strength distribution based on microstructural information

S. Ozaki*[1]; T. Maeda[2]; R. Higashi[2]; T. Osada[3]

1. Yokohama National University, Faculty of Engineering, Japan
2. Yokohama National University, Graduate School of Engineering Science, Japan
3. National Institute for Materials Science (NIMS), Research Center for Structural Materials, Japan

The prediction of the strength scatter caused by internal defects is necessary in a reliable design of ceramics. In this study, we proposed a finite element analysis (FEA) method to predict the stochastic fracture behavior of ceramics based on the microstructural information obtained by SEM and X-ray CT. Specifically, the spatial distribution of microstructure data, such as pore and grain sizes, are approximated by appropriate probability density functions and are reflected in the dispersion of parameters of the damage model via a fracture mechanics model. We then analyzed the three-point bending test using the proposed method. Note that, in this method, the stochastic distribution of the microstructure in multiple FEA models can be naturally reproduced using random numbers, and the fracture strength for each analysis model is evaluated. The prediction performance of the strength scatter was examined for alumina fine ceramics sintered at three different temperatures to demonstrate the effectiveness of the proposed methodology. The results confirm that the proposed method reasonably predicts the strength scatter in ceramics. This presentation is based on results obtained from a project, JPNP22005, commissioned by the New Energy and Industrial Technology Development Organization (NEDO).

11:00 AM

(ICACC-S10-012-2024) Numerical prediction of strength scatter of ceramics: Inverse estimation of microstructural distribution based on strength test data

T. Maeda*[1]; T. Osada[2]; S. Ozaki[3]

1. Yokohama National University, Graduate School of Engineering Science, Japan
2. National Institute for Materials Science (NIMS), Research Center for Structural Materials, Japan
3. Yokohama National University, Faculty of Engineering, Japan

Strength of ceramic members shows scatter due to their brittleness and the inhomogeneity of microstructural features. Thus, the understanding of microstructural information, such as pores and grain boundaries, in the bulk body would contribute to the design of highly reliable ceramic members. Furthermore, from a practical point of view, it is valuable to make it possible to estimate microstructural information based on results obtained from standardized strength tests. In this study, we proposed a numerical analysis method that can estimate microstructural distribution based on strength test data, i.e., Weibull plots. Specifically, the particle swarm optimization (PSO) was applied to the prediction method for the strength scatter proposed by authors, estimating the microstructural distribution that can reproduce the experimental strength data. The estimation of grain size distribution was examined for alumina fine ceramics sintered at three different temperatures to demonstrate the effectiveness of the proposed inverse estimation method. Each estimated grain size distribution was in reasonably good agreement with that obtained by the electron back scattered diffraction (EBSD) method. This presentation is based on results obtained from a project, JPNP22005, commissioned by the New Energy and Industrial Technology Development Organization (NEDO).

11:20 AM

(ICACC-S10-014-2024) Disordered Solids by Design: A global optimization method in materials physics

M. Belhadj Larbi*[1]

1. University of Missouri, Kansas City, Physics and Astronomy, USA

The relationship between the composition, the fabrication process, and the properties of non-crystalline materials is of great interest because the final material properties can often be modulated by changes in their composition and growth conditions, e.g., amorphous molecular solids grown via plasma enhanced chemical vapor deposition from volatile molecular precursors. However, creating models of such solids is a challenging task due to the lack of appropriate interatomic potentials and the complexity of the structure. We demonstrate progress toward establishing a new method for modeling disordered solids without the need for an accurate interatomic potential. The method consists of explicitly parameterizing the bond creation/breaking processes within a classical molecular dynamics scheme (LAMMPS), coupled with an evolutionary algorithm (EA). The role of the EA is to determine the appropriate set of LAMMPS parameters (among a large pool of possibilities) that yield a target structure. We will illustrate our process for modeling amorphous molecular solids from molecular precursors with an example molecular precursor: hexamethyldisilazane (HMDS). We will show that when the EA is fed with the initial conditions for the gas phase structure of HMDS, it will progressively determine a structure that is consistent with available characterization data or desired constraints while minimizing the total energy.

S12 Design and Applications of Nanolaminated Ternary Transition Metal Carbides/Nitrides and Borides, Their solid solutions and 2D Counterparts

SYMPOSIUM 12: Design and Applications of Nanolaminated Ternary Transition Metal Carbides/Nitrides (MAX Phases) and Borides (MAB Phases), their Solid Solutions and 2D Counterparts (MXenes, MBenes)

Room: Ballroom 3
Session Chair: Sylvain Dubois, PPRIME Institute

8:30 AM

(ICACC-S12-007-2024) Expanding the family of MAX phases: Synthesis of exotic layered solids (Invited)

C. Birkel*[1]

1. Arizona State University, USA

Ternary carbides, nitrides and carbonitrides that belong to the family of MAX phases not only possess a unique set of properties (combining metallic and ceramic characteristics), they are also the precursors for their two-dimensional siblings, MXenes. According to the general chemical formula $M_{n+1}AX_n$ (n = 1, 2, 3), "211", "312" and "413" MAX phases possess n (1, 2, 3) M_6X layers (2, 3 and 4 M-layers) between the A-layers, respectively. A variant of the "211" MAX phase structure is the highly related "221" structure that crystallizes in the same hexagonal space group, $P6_3/mmc$, but features 2 A-element layers with the A-element stacked on top of one another, between the octahedral layers of M and X. "221" phases are structurally very interesting, but they also serve as a precursor of 2D MXenes. For example, MXene Mo_2CT_x can only be synthesized from "221" Mo_2Ga_2C (through HF-etching) and not from the "211" MAX phase Mo_2GaC. In this talk, I will focus on the transition between "221" Mo_2Ga_2C and the fully exfoliated MXene version Mo_2CT_x using Lewis acids as the etchant. Besides the chemistry of these systems and transitions, I will discuss the structure of the compounds with varying Ga-content. I will further highlight additional "more exotic" MAX phases and MXenes that we investigate in our group, such as P-containing MAX phases and "higher" MXenes, such as $(Mo/V)_5C_4T_x$.

9:00 AM

(ICACC-S12-008-2024) Extending the concept of crystal structure nanolamination beyond the early transition metal carbides/nitrides known as the MAX phases – the ZIA phases (Invited)

K. Lambrinou*[1]; M. A. Tunes[4]; N. Goossens[2]; S. Huang[2]; J. Vleugels[2]; S. A. Maloy[3]

1. University of Huddersfield, School of Computing and Engineering, United Kingdom
2. KULeuven, Materials Engineering, Belgium
3. Pacific Northwest National Lab, Reactor Materials and Mechanical Design Group, USA
4. Los Alamos National Lab, Materials Science and Technology Division, USA

This work introduces the concept of the ZIA (zigzag intermetallic) phases, a new potential class of nanolaminated and structurally complex intermetallic compounds (IMCs). The ZIA phases are characterized by an fcc close-packed lattice (Fd3m, space group 227) of remarkable structural complexity, while obeying the stoichiometric rule of the 312 MAX phases, i.e., $M_{n+1}AX_n$ with n = 2. The first candidate ZIA phase, i.e., the Nb_3SiNi_2 IMC, is identified as the H-phase of the ternary Nb-Si-Ni system; in this work, the Nb_3SiNi_2 ZIA phase was detected in bulk samples synthesized by arc melting (low Nb_3SiNi_2 content) and reactive hot pressing (almost phase-pure Nb_3SiNi_2). The transmission electron microscopy investigation of Nb_3SiNi_2 revealed an exceptionally large unit cell made of 96 atoms and characterized by both crystal structure nanolamination and 'zigzag' atomic arrangement. The ZIA phases extend the concept of crystal structure nanolamination beyond the early transition metal carbides/nitrides known as the MAX phases, broadening the spectrum of achievable properties into domains typically not covered by the MAX phases. Both classes of nanolaminated crystalline solids, i.e., the fcc ZIA phases and all variants of the hcp MAX phases, obey the same overarching stoichiometric rule $P_{x+y}A_xN_y$, where x and y are integers in the 1-6 range.

9:30 AM

(ICACC-S12-009-2024) Chemical Expanding of Chalcogenide MAX phase at X site (Invited)

K. Chen*[1]

1. Ningbo Institute of Materials Technology and Engineering, Chinese Academy of Sciences, Engineering Laboratory of Advanced Energy Materials, China

The realization of chemical diversity of MAX phases is distinctive and important to screen out their unique physiochemical properties for prospective applications. Compared with well-known 25 M-site elements and 23 A-site elements, the option of X-site element is under-explored and restricted in few non-metal elements such as C, N and B. Herein, a chalcogen element selenium was found to be a new member to occupy X site in MAX phases which were stabilized through boron alloying. A series of $Zr_2Se(B_{1-x}Se_x)$ phases were successfully prepared with occupancy rate x up to 0.97. The variation of M_6X octahedral distortion and the difference in binding energy between Zr_6Se and Zr_6B octahedra were found to contribute the phase transformation from boride MAX phase to chalcogenide MAX phase. The effect of occupancy of Se at X site on electrical conductivity of MAX phase indicates that the deterioration of carrier motility induced by the strong binding of Zr_6Se octahedron in MX sublayers leads to the reduction of electrical conductivity. This work also provides a new route to tune the physiochemical properties of MAX phases through the regulation of their basic M_6X octahedron units.

10:20 AM

(ICACC-S12-010-2024) Influence of Al_2O_3 continuous fiber-reinforcement on Ti_2AlC MAX-Phases (Invited)

F. Jung*[1]; L. Aretz[2]; T. Gries[1]; J. Gonzalez-Julian[2]

1. RWTH Aachen University, Institut für Textiltechnik, Germany
2. Institute of Mineral Engineering of RWTH Aachen University, Germany

MAX phase ceramics show great potential for a new, innovative generation of engineering materials for energy technology due to their excellent mechanical properties in a high-temperature atmosphere. Fiber-reinforcement of MAX phases show great potential to further increase the materials engineering capabilities. However, the mechanisms to introduce continuous fiber-reinforcement into the novel ceramic system are not understood yet. Suitable processes for the production of continuous fiber-reinforced ceramics are currently formulated only by colloidal manufacturing processes. A major challenge of colloidal processes regards the homogeneous impregnation of textile reinforcement structures. The production of continuous fiber-reinforced Ti_2AlC composites is carried out via a colloidal production route. For this purpose, suitable binder regimes for a bimodal casting slip are investigated and used for the impregnation of uncoated 2- and 3-dimensional braided alumina fiber reinforcements of the type Nextel 610 (3M Corp.). The reinforcements architecture's effect on the composite properties is carried out by using short-term mechanical tests and will be described in more detail within this presentation.

10:50 AM

(ICACC-S12-011-2024) A-site alloying-guided universal design of noble metal-based MAX phases (Invited)

Y. Li*[1]

1. Soochow University, China

MAX phases have attracted significant attention due to their structural diversity and prospects for applications. The design of new MAX phases with single-atom-thick A layers featuring 4d or 5d electronic elements is an interesting and challenging work. Here, we present a comprehensive report on noble metal-based $M_2(A_{1-x}A'_x)C$ (M= V, Ti, Nb, A= Al, Sn, In ,Ga, Ge, A' = Ru, Rh, Pd, Ir, Pt, Au and combinations thereof, $0<x\leq 1/3$) phases featuring A sublayers of 4d and 5d electronic elements through A-site alloying strategy. The chemical composition of these noble metal-based MAX phases can be fine-tuned by selecting different combinations of M and A-site elements, and the morphology can also be tailored by using distinct carbon sources. Furthermore, in 1 M KOH, it was found that the catalytic performance of the $V_2(Sn_{0.8}Pt_{0.2})C$ (15.7 wt.% Pt) for the hydrogen evolution reaction was superior to that of the commercial Pt/C (20 wt.% Pt) electrode, which was supported by density functional theory calculations. This study highlights the prospects of the A-site alloying for designing MAX phases with unique properties and new chemical compositions, with promise for applications in electrocatalysis and beyond.

11:20 AM

(ICACC-S12-013-2024) Radiation response of ultra-pure chemically complex MAX phase ceramics in the (Ti,Zr,Hf,V,Nb)-(Al,Sn)-C & (Zr,Ti)-(Al,Sn,Pb,Bi)-C systems

K. Lambrinou*[1]; N. Goossens[2]; S. Huang[2]; J. A. Hinks[1]; J. Vleugels[2]

1. University of Huddersfield, School of Computing and Engineering, United Kingdom
2. KULeuven, Materials Engineering, Belgium

The MAX phases are characterized by dynamic recovery of radiation-induced defects at >600°C, making them promising fuel cladding coating materials for nominal operation temperatures ≥600°C. The poor radiation tolerance of the MAX phases at lower operation temperatures require sophisticated materials engineering (i.e., high phase purity, strong texture, fine grains). In an effort to produce ceramics with good radiation tolerance, 211 & 312 double solid solutions with 5 elements (Ti,Zr,Hf,V,Nb) on the M-site and 2 elements (Al,Sn) on the A-site were synthesised. Due to an enhanced configurational entropy, these nanolaminated, chemically complex MAX phases might demonstrate radiation tolerance similar to that of high-entropy alloys (HEAs). The 1st generation of MAX phases in the (Ti,Zr,Hf,V,Nb)-(Al,Sn)-C system had high phase purity (4-5 vol% parasitic phases) and were free from pseudo-binary carbides, but showed local segregation of V-Al & Nb-Al intermetallics. Using fine metal hydride powders produced phase-pure MAX phase ceramics in the (Ti,Zr,Hf,V,Nb)-(Al,Sn)-C & (Zr,Ti)-(Al,Sn,Pb,Bi)-C systems. These chemically complex MAX phases were irradiated using 6 keV He^+ at 350°C and 600°C, and their radiation response was compared with that of ternary compounds (e.g., Zr_2AlC, Ti_2AlC) and less complex double solid solutions (e.g., $(Ti,Zr)_2(Al,Sn)C$).

S14 Crystalline Materials for Electrical Optical and Medical Applications

SYMPOSIUM 14: Optical transparent ceramics

Room: Coquina H
Session Chairs: Luisa Bausa, Universidad Autonoma de Madrid; Hiroaki Furuse, National Institute for Materials Science (NIMS)

8:30 AM

(ICACC-S14-001-2024) Fabrication of transparent polycrystalline ceramics by colloidal processing and SPS (Invited)

T. S. Suzuki*[1]

1. National Institute for Materials Science (NIMS), Optical Ceramics Group, Japan

Transparent ceramics can maintain their transparency and structural integrity at elevated temperatures, making them valuable for high-temperature environments and applications. Extremely low porosities are indispensable for high transparency. In ceramics with anisotropic crystal structure, in-line transmittance deteriorates due to birefringence due to the difference in refractive index at grain boundaries, but in the Rayleigh scattering region, birefringence can be suppressed by reducing the grain size. Birefringence can also be suppressed by aligning the crystal orientation. Colloidal processing is a very effective technique for controlling the pore size distribution in green compacts before densification by sintering. The green compacts having small residual pores is expected to enhance the densification. Furthermore, spark plasma sintering (SPS) is also effective way for densification in low sintering temperature. In this presentation, this processing was applied to fabrication of transparent AlN and lanthanum silicate (LSO). The c-axis orientation in AlN and LSO could be controlled by the rotating and static magnetic field, respectively and then transparency was achieved by densification using SPS. In the case of LSO, the transmittance increased with the decreasing of grain size in the sintered body and with the increasing the degree of orientation.

9:00 AM

(ICACC-S14-002-2024) Carbon Contamination in Yttria Sintered via SPS

J. Gild*[1]; A. R. Floyd[1]; B. Sadowski[3]; T. Zhou[2]; W. Kim[1]; S. Bayya[1]; J. Sanghera[1]

1. US Naval Research Laboratory, USA
2. University Research Foundation, USA
3. Jacobs, USA

There are numerous applications operating in very harsh environments that require rugged windows. Polycrystalline Y_2O_3 ceramic is a prime candidate for these applications due to the potentially high mechanical strength, wide transmission range of 0.2 – 9 μm, high thermal conductivity of 14 W/mK, and isotropic structure. To fabricate a high transparency window, phase and chemically pure powder must be obtained and great care taken in sintering to minimize contamination. Spark plasma sintering (SPS) allows for rapid densification of yttrium oxide while maintaining a fine grain size for optimal strength. Due to reducing atmosphere and graphite tooling during SPS densification, carbon contamination is frequently observed in oxides sintered by this method. In this presentation, the relationship between load application temperature and carbon contamination is discussed as well as other optical and physical properties.

9:20 AM

(ICACC-S14-003-2024) Ternary Zn-Sb Oxide Secondary Phases in Zinc Oxide Ceramics: Phase Formation and Stability as a Function of Reaction Control and Dopant Concentration

D. Schrake[1]; U. Betke*[1]

1. Otto-von-Guericke-University, Institute for Materials and Joining Technology - Non-metallic Materials and Composites, Germany

Zinc oxide is a widely applied ceramic for electronics, for example as varistors which possess a non-ohmic electrical conductivity over several orders of magnitude. For the manufacturing of ZnO varistors usually additives, e. g. Sb_2O_3, amongst others, are used as sintering aids. Control over microstructure and properties of these ceramics is finally obtained by doping with transition metal (TM) oxides, e. g. of Cr, Mn, Co, and others. Although it is understood which dopants are neccessary to achieve the desired conductivity characteristics, little is known about the mechanisms being involved in the doping process. This includes the effect of dopants on the stability and formation kinetics of the secondary phases within the varistor ceramic's microstructure. This work deals with the principal investigation of ternary Zn-Sb oxides, e. g. $Zn_7Sb_2O_{12}$, which is typically found as a secondary phase in ZnO ceramics made with Sb_2O_3 as sintering aid. The phase formation in ZnO-Sb_2O_3 mixtures is studied as a function of stochiometry, reaction temperature and the presence of additional TM oxide dopants.

9:40 AM

(ICACC-S14-004-2024) Tuning the properties of Cobalt Ferrite: Magnetoelectric material engineering for user-defined control of neural stimulation

A. Milojkovic*[1]; K. Kozielski[1]

1. Technical University of Munich, Germany

Neural devices have been largely used to treat and diagnose neurological disorders. However, they are bulky and impose a risk of infections. To overcome this, injectable nanoparticles are a potential approach to neurostimulation, but nanoscale powering is a challenge. In this research, we are working on the magnetoelectric nanoparticles (MENPs) that are minimally invasive and wirelessly powered. MENPs consist of a magnetostrictive core and piezoelectric shell which are coupled by a common interface to induce magnetic to electric coupling. Our goal is to obtain a spinel cobalt ferrite (CFO) with high coercivity, hence better magnetostriction. We expect that by tuning the properties of the core, we will get a better control on the neural activated area. CFO was synthesized with a coprecipitation method, and results were assessed via XRD, TEM and VSM. Coercivity and magnetic saturation were most strongly impacted by the temperature and the concentration of the agent. Controlling coercivity and magnetic saturation enables us to engineer the magnetoelectric properties and therefore tune the MENP's function as a neural stimulator. We demonstrate that using basic chemistry, it is possible to tune the signal-response of these nanoparticles and therefore improve the powering. Here we report a promising tool for a less invasive treatment of neurological disorders.

10:20 AM

(ICACC-S14-006-2024) Mechanical, Thermal and Optical Properties of Pyrochlores: Modeling and Experiment

L. Kotrbova*[1]; V. Nečina[1]; S. Hribalova[1]; T. Unger Uhlirova[1]; W. Pabst[1]

1. University of Chemistry and Technology, Prague, Department of Glass and Ceramics, Czechia

Pyrochlore ceramics are promising candidates for thermal barrier coatings but also for photoluminescent applications. For both, knowledge of mechanical and thermal properties and their dependence on grain size and porosity is crucial. Also, the effect of porosity on optical properties is enormous. All these properties are influenced by doping by rare-earth elements. The aim of this work is to quantify these effects. For this purpose, Eu^{3+}-doped $La_2Zr_2O_7$ ceramics are prepared with different Eu^{3+} contents via spark plasma sintering. The measured grain size is used as an input information for computer-based modeling of elastic properties, thermal conductivity and transmittance. Modeling of elastic properties and thermal conductivity is performed via phase mixture modeling and sharp interface model, while the optical transmittance is modeled via the van de Hulst approximation based on a realistic pore size estimate. The porosity is estimated based on a comparison of the model predictions with actual measurements, i.e., dynamic measurements of elastic properties, measurements of thermal diffusivity and spectrometric measurements of the in-line transmittance. Acknowledgement: This work is part of the project GA22-14200S "Low-phonon energy transparent ceramic luminophores emitting in the short- and mid-infrared region", funded by the Czech Science Foundation (Grantová agentura České republiky / GAČR).

10:40 AM

(ICACC-S14-007-2024) Experimental evidence of cubic γ- calcium lanthanum oxysulfide crystalline phase

A. Kostogiannes*[1]; B. Butkus[1]; A. Howe[3]; P. Banerjee[1]; C. R. Baleine[2]; K. Richardson[3]; R. M. Gaume[3]

1. University of Central Florida, Materials Science and Engineering, USA
2. Lockheed Martin, USA
3. University of Central Florida, CREOL, USA

Current ternary phase diagrams related to the calcium lanthanum sulfide ceramic system exclude the possibility for the incorporation of oxygen to produce a cubic phase. Typical oxysulfide crystal phases have lower symmetry crystal structures such as hexagonal or tetragonal. Further, discussion of the incorporation of oxygen into the cubic crystalline structure remains incomplete. Understanding the role of this impurity is one of the most important topics of research for this material. The work presented here provides evidence for the occurrence of a stable cubic calcium lanthanum oxysulfide system. X-ray diffraction, infrared spectroscopy, and x-ray photoelectron spectroscopy were used to understand the structural role of oxygen.

11:00 AM

(ICACC-S14-008-2024) Prism Coupling Refractometry: Characterizing Novel Optical Ceramics during Development

A. Howe*[1]; K. Richardson[2]; R. M. Gaume[2]; A. Kostogiannes[3]; M. Kang[4]

1. University of Central Florida, USA
2. University of Central Florida, CREOL, USA
3. University of Central Florida, Materials Science and Engineering, USA
4. University of Central Florida, CREOL, College of Optics & Photonics, USA

Prism coupling refractometry is a rapid means to determine the refractive index of both films and bulk materials, offering an alternative to ellipsometry. This approach is typically based on the assumption of achieving an accuracy and precision level of 0.001. A notable commercial system for conducting such measurements is the Metricon2010M, which covers a wavelength range from 500 to 1350 nm. In this work, we outline a modified Metricon 2010M measurement setup for characterizing infrared (IR) materials. Our work suggests that by reevaluating this technique, carefully considering the electromagnetic fields involved, and implementing updated procedures and equipment, we can significantly enhance its accuracy and precision. This reinvigorated prism coupling approach, when coupled with our novel methodology, has the potential to rival other established techniques such as ellipsometry in terms of measurement accuracy and precision.

11:20 AM

(ICACC-S14-005-2024) The Optically Stimulated Luminescence Dosimetry of $MgAl_2O_4$ (Invited)

L. Pan[1]; S. Sholom[2]; S. McKeever[2]; L. G. Jacobsohn*[1]

1. Clemson University, Materials Science and Engineering, USA
2. Oklahoma State University, USA

Presently, there are only two commercially available optically stimulated luminescence (OSL) dosimeters, BeO and Al_2O_3:C, fabricated by Dosimetrics and Landauer, respectively. New materials are sought towards better efficiency and performance. In magnesium aluminate solid solutions, Mg^{2+} replacing for Al^{3+} and Al^{3+} replacing for Mg^{2+} are commonly observed with the Al^{3+} ion located in a tetrahedral site becoming an electron trap and the Mg^{2+} ion in an octahedral site becoming a hole trap. This work built on this structural knowledge and investigated for the first time how solid solution disorder affects the OSL behavior of Al-rich, Mg-rich and stoichiometric $MgAl_2O_4$. $MgAl_2O_4$ spinel solid solutions were synthesized by the coprecipitation method with different Mg/Al ratios: 0.33 (Al-rich), 0.5 (stoichiometric) and 0.75 (Mg-rich). Powders were calcined at 1000ºC for 2h and their structure characterized by X-ray diffraction and Raman spectroscopy measurements. OSL was characterized as a function of the irradiation dose and in terms of its signal linearity with the irradiation dose and reproducibility. The minimum detectable dose (MDD) was determined, the OSL decay curves were analyzed in terms exponential functions, and fading was evaluated. A critical evaluation of $MgAl_2O_4$ as an OSL dosimeter is presented. This material is based upon work supported by the National Science Foundation under Grant No. 1653016.

S1 Mechanical Behavior and Performance of Ceramics & Composites

SYMPOSIUM 1: Ceramics for concentrated solar–thermal power and industrial process heat I

Room: Coquina E
Session Chairs: Dileep Singh, Argonne National Lab; Kamala Raghavan, Department of Energy; Anteneh Kebbede, GE Aerospace Research

8:30 AM

(ICACC-S1-051-2024) Ceramic Materials for High Temperature Concentrating Solar-Thermal Technologies (Invited)

K. C. Raghavan*[1]

1. Department of Energy, USA

Concentrating solar-thermal (CST) plants capture the sun's energy and convert it to thermal energy which can be stored and dispatched on-demand. The Department of Energy (DOE) has set a target cost of electricity of \$0.05/kWh by 2030 with more than 12 hours of thermal energy storage (TES). To reach this target, DOE is adopting the technical strategy that focuses on high efficiency supercritical sCO_2 Brayton cycle, operating at temperatures higher than 700°C. Additionally, the thermal energy can be a carbon-free heat source to several high temperature industrial processes such as cement, steel, chemical and fuels production. Required temperatures for many of these hard-to-decarbonize industrial process are between 600°C to 1400°C. For both power and industrial process applications, the critical & ancillary components – including receivers, reactors, heat exchangers, valves, pipes, tubes and TES materials must operate at extreme and harsh service conditions. These conditions demand innovation in new material & fabrication technologies. To this end the Solar Energy Technologies Office (SETO) supports research in high temperature ceramics & composites along with fabrication and processing technologies. This presentation will discuss the ongoing ceramic material and fabrication research work supported by SETO and will present a longer term ceramic material strategy for high-temperature CST components.

9:00 AM

(ICACC-S1-052-2024) Scalable, Infiltration-Free Ceramic Matrix Composite Manufacturing

A. Thukral[1]; C. Cordeiro[1]; R. Pandey[1]; K. Bhattacharyya[1]; B. Karki[1]; G. Iftime[1]; J. Wei*[1]

1. PARC, USA

Manufacturing of ceramic matrix composites (CMC) made with carbon fiber and carbon matrix includes a time and labor-intensive ceramic infiltration step which is responsible for more than half of the total manufacturing cost. To make CMC cost-competitive in price-sensitive markets, like concentrated solar power, it is essential to develop a CMC manufacturing process that skips ceramic infiltration. In this presentation, we will show how integrating a preceramic resin with a high char yield can eliminate the infiltration step while maintaining material density and evaluate the technoeconomic impact of our CMC manufacturing process. We monitor both morphology and porosity in each step to determine the quality of the CMC made with different preceramic resins and evaluate the impact of polymer infiltration and pyrolyzing cycles. We will further discuss how the CMC made by this scalable and infiltration-free method is suitable for the demanding operation conditions of the next generation of concentrated solar power plants.

9:20 AM

(ICACC-S1-053-2024) Performance of Multilayer Silicon Carbide Fiber Composites for Concentrated Solar Power

F. Mohammadi*[1]; J. Halfinger[1]; A. Sherburt[1]; K. Armijo[2]; N. Schroeder[2]; T. Daspit[3]; M. Anderson[4]

1. Ceramic Tubular Products, LLC, USA
2. Sandia National Laboratories, USA
3. University of Virginia, USA
4. University of Wisconsin, USA

In the past several years, silicon carbide (SiC) fiber-reinforced composites have been extensively investigated for high temperature applications especially in the areas of solar-thermal energy. Its excellent mechanical, thermal, and optical properties at high temperatures make SiC very attractive for the use in "Gen 3" concentrated solar power (CSP) plants, as a solar receiver or as a heat exchanger coupled to an sCO2 Brayton cycle power conversion system. In this work, the performance of multilayer SiC fiber composites made by a polymer infiltration and pyrolysis (PIP) process was investigated. The composites were manufactured using Hi-Nicalon and Nicalon-CG silicon carbide fibers infiltrated with a silicon carbide matrix. The multilayer SiC tubes showed stable solar absorptance and emittance of 0.97 and 0.91, respectively, at 800 °C. The hoop tensile strength of the composites revealed no loss of mechanical strength due to the thermal shock up to 1100 °C. In addition, the joined SiC components showed significant corrosion resistance to molten chloride salt at 800 °C. This work is focused on addressing the properties of multilayer SiC fiber composites manufactured by PIP process relevant to high temperature requirements of Gen 3 CSP plants. The manufacturing and properties of multilayer SiC tubes such as mechanical, thermal, and solar optical performance will be discussed in this paper.

9:40 AM

(ICACC-S1-054-2024) Current Activated Reactive Ultrafast Joining (CARUJ): An approach for rapid fabrication and evaluation of ceramic-ceramic joint assemblies

S. Shivakumar*[2]; B. Barua[1]; M. C. Messner[1]; P. S. Chaugule[1]; D. Singh[1]; J. Luo[2]

1. Argonne National Lab, USA
2. University of California, San Diego, USA

We propose and demonstrate a novel approach for rapid fabrication and evaluation of ceramic-ceramic joint assemblies, using Silicon Carbide (SiC) as an initial example. Current Activated Reactive Ultrafast Joining (CARUJ) utilizes carbon-based materials to resistively heat materials to be joined in addition to any interface material, in an ultrafast manner. Heat application can be through the interface, or around the joint zone. The former approach requires careful thought and selection of the resistive heater, and associated reaction kinetics. Combined with a reactive interfacial precursor design based on elemental silicon and carbon, we synthesize silicon carbide in-situ within the joint interface while simultaneously performing reactive joining. This approach allows the fabrication of high strength SiC-SiC joints with a Si/SiC interface, at time scales which are an order of magnitude shorter (several minutes opposed to hours/days) than conventional approaches for similar systems. CARUJ joined samples can exhibit measured joint strengths close to 100 MPa in compression shear testing. We further demonstrate SiC-SiC joining using a reactive braze interlayer, in addition to joining different geometries. The localized and rapid heat application realizes a versatile material joining technique that could be extended to the field.

10:20 AM

(ICACC-S1-055-2024) Robust Ceramic/Metal Composites for High-Temperature Heat Exchangers for Concentrated Solar Power (Invited)

C. McCormack[1]; Y. Wang[1]; M. Bidabadi[1]; S. Hwang[1]; A. Lapotin[2]; M. Adams[3]; S. Yee[3]; A. Henry[2]; S. Kakooei[1]; K. Trumble[1]; K. Sandhage*[1]

1. Purdue University, Materials Engineering, USA
2. Massachusetts Institute of Technology, Mechanical Engineering, USA
3. Georgia Institute of Technology, Mechanical Engineering, USA

Concentrated solar power (CSP) plants utilize focused sunlight to heat a high-pressure working fluid that is used to drive turbine engines to create electricity. The solar thermal-to-electrical conversion efficiency may be significantly increased, with a corresponding reduction in the cost of CSP-derived electricity, by increasing the temperature of the working fluid at the turbine inlet (e.g., by using supercritical carbon dioxide, sCO_2, at ≥750°C, instead of subcritical steam at ≤550°C). However, effective heat transfer requires compact heat exchangers (HEXs) capable of operating under such extreme conditions. The use of stainless steels and nickel-based alloys in such HEXs is inhibited by the significant decrease in mechanical performance of these alloys at such temperatures and pressures. Alumina/chromium (Al_2O_3/Cr) composites possess attractive thermal, mechanical, and chemical properties for such high-temperature, high-pressure HEXs. In this work, reaction forming has yielded dense Al_2O_3/Cr composites comprised of fine (<5 μm) Cr domains uniformly dispersed in an Al_2O_3 matrix. The thermal expansion/cyclability, thermal conductivity, creep resistance, fracture strength, and oxidation resistance of such dense Al_2O_3/Cr composites have been evaluated. The fabrication of Al_2O_3/Cr HEX plates with tailorable channel patterns, and the bonding of such plates will be discussed.

10:50 AM

(ICACC-S1-056-2024) Role of Ceramics and Composites in Gen3 Concentrated Solar Power Applications* (Invited)

D. Singh*[1]

1. Argonne National Lab, USA

Extreme environments and operating conditions of power generation from renewable energy sources is necessitating development of components and systems using advanced materials. This presentation will discuss the opportunities for ceramics and composites specific to concentrated solar-thermal power. Attributes of ceramics and their composites that make them the materials of choice are their high-temperature mechanical properties, corrosion and creep resistance, etc. As part of the presentation, specific example of a ceramic thermal component, such as heat exchangers, will be presented. Design, fabrication, and testing of a SiC based heat exchanger will be discussed. Finally, on-going efforts in the process scale-up of the ceramic heat exchanger will be highlighted. *This work was supported by the U.S. Department of Energy's (DOE) Office of Energy Efficiency and Renewable Energy (EERE) under the Solar Energy Technologies Office (SETO) Award Number 38479 at Argonne National Laboratory operated under Contract no. DE-AC02-06CH11357 by the UChicago Argonne, LLC.

11:20 AM

(ICACC-S1-057-2024) Joining of Ceramic/Ceramic and Ceramic/Metal Using Induction Heating

T. H. Lee*[1]; M. Du[1]; S. Sivakumar[2]; J. Luo[2]; D. Singh[1]

1. Argonne National Laboratory, Applied Materials Division, USA
2. University of California, San Diego, USA

Silicon carbide (SiC) based ceramics are considered as candidate component materials in several fields of engineering such as nuclear, aeronautics, electronics, and transportation. Even though SiC based ceramics possess several attributes that are deemed essential in the various fields of engineering, they are required to be used in combination with other material components. We have explored the using of induction heating technology to join metal-ceramic as well as ceramic-ceramic. We investigated both brazing and reactive boding methods to demonstrate self-joining of ceramics and metals, such as SiC, MAX phase, and nickel super alloy (Haynes 230). The joining experiments were performed in an induction furnace under a controlled atmosphere. The influence of joining parameters, temperature and time, on the quality of the joint was invested with a typical joining area of ~10 mm^2. The microstructure of joint interfaces was characterized by electron microscopy. The mechanical property of joined specimen was characterized by measuring the compression shear strength.

11:40 AM

(ICACC-S1-058-2024) UHTC-TPMS Heat Exchangers for Concentrated Solar Power Applications with Thermal Energy Storage in Molten Chlorides

J. Kelly*[1]; B. Pint[2]; S. S. Raiman[3]; J. Haslam[1]

1. Lawrence Livermore National Laboratory, USA
2. Oak Ridge National Lab, USA
3. University of Michigan, USA

This project aims to develop a UHTC composite material that is compatible with molten chloride salt even when a minor amount of water impurity is present, is compatible with oxidizing environments, and significantly advance the manufacturability of the material. Binder-jet printing and post-processing parameters will be developed for the material. Derisking materials and manufacturing will enable the development and testing of future UHTC-TPMS heat exchanger prototypes.

S3 21th Intl Symp on Solid Oxide Cells Materials Science & Technology

SYMPOSIUM 3: Fuel Electrode & Electrolytes / System modelling and validation

Room: Ballroom 1-2
Session Chairs: Julie Mougin, CEA; Mihails Kusnezoff, Fraunhofer IKTS

8:30 AM

(ICACC-S3-042-2024) Enhancement of the intrinsic Ni/GDC activity under rSOC operation by means of Fe-Au doping: An electro-kinetic study (Invited)

F. Zaravelis[1]; D. K. Niakolas*[1]

1. FORTH/ICE-HT, Greece

The investigation dealt with the effect of Fe and Au wt.% contents on the enhanced performance & stability of Ni/GDC under rSOC operation. Moreover, it focused on the comparison of the intrinsic electro-kinetics between the best performing Fe-Au-modified cell and Ni/GDC under SOE operation. All Fe-Au-Ni/GDC electrodes exhibited enhanced activity vs Ni/GDC, with variations in their stability after three rSOC cycles. Specifically, the fuel electrode with the lowest wt.% concentration in Fe and Au exhibited the highest activity and stability under rSOC operation. By increasing the wt.% loadings to 2 wt.% Fe and 3 wt.% Au, there was rapid degradation of the initially high performance, mainly due to increase of Rohm. SEM analysis showed higher macro-porosity on the modified electrodes, which in the case of the best performing was the least affected after rSOC operation. Interestingly, the SOE performance of the examined Fe-Au-modified electrodes was not similarly inhibited by the decrease of temperature, compared to Ni/GDC. This was further clarified through electro-kinetic measurements, aiming to verify the effect from the Fe-Au doping on the intrinsic electrochemical activity of Ni/GDC for H_2O electrolysis. Comparison between the SoA and the best performing 0.5Fe-1Au-Ni/GDC showed an enhancing effect on the intrinsic electro-kinetics of the modified electrode.

9:00 AM

(ICACC-S3-043-2024) Fused zirconia powders for use in advanced ceramics applications (SOFC/EC)

T. Rey Wójcik[2]; J. Szymanska[2]; A. Villalba Weinberg[2]; S. Griesser[2]; A. Börger[2]; A. Priese*[1]

1. Imerys Murg GmbH, Science & Technology, Germany
2. Imerys Villach GmbH, ITC Center, Austria

The electric arc furnace method represents a viable industrial approach for producing high-purity metal oxides with diverse applications, including advanced refractories, thermal barrier coatings, and sensors. This paper outlines the procedure for manufacturing high-purity stabilized zirconia powders, incorporating stabilizers like yttria, ytterbia, scandia-ceria, and explores their potential as oxide ion conductors in Solid Oxide Fuel Cells (SOFC) and Electrolysis Cells (EC). To showcase the distinct qualities of fusion-derived products, particularly their uniform chemical and phase compositions, we present examples. Furthermore, we compare the tape casting process for these powders with that of co-precipitated powders and find that tape casting is more easily attainable with powders produced via the fusion process.

9:20 AM

(ICACC-S3-044-2024) Bending strength and yielding behavior of 8YSZ single crystals and ceramics measured using microcantilever beam specimens

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

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

Bending strength and yielding behavior of 8YSZ single crystals and ceramics were evaluated using microcantilever beam specimens with various crystal orientations. Nonlinearity due to plastic deformation in the single crystals and grains in the ceramics was observed in the stress-strain curves, and the yield stress was found to depend on the crystal orientation. TEM observation revealed many areas of disordered lattice in the region where the maximum tensile stress occurred. Resolved shear stress (RSS) was analyzed for the primary {001}<110> and the secondary {110} <1 -1 0> and {111} <1 -1 0> slip systems as a function of the angle between the specimen's longitudinal direction and the [001] orientation, indicating that the RSS for the secondary ones was the critical RSS in the angle at 10° or lower while that for the primary one did so at 20° or higher. The measured bending strength of the single crystals, which depended on the crystal orientation, was lower than, but on the same order of magnitude as, the ideal strength. The bending strength of the grains in the 8YSZ ceramics was good agreement with that of the single crystals in consideration of the stress concentration of a pore. The strength of the solid electrolyte in a SOFC was also evaluated by the microcantilever bending test.

9:40 AM

(ICACC-S3-045-2024) High temperature tensile strength of ultra-thin 3YSZ ceramic foils for SOEC

I. Bombarda[1]; C. Sitzmann[1]; N. Langhof[1]; S. Schafföner*[1]

1. University of Bayreuth, Chair of Ceramic Materials Engineering, Germany

Due to the energy transition, hydrogen generation is playing an increasingly important role. High temperature solid oxide electrolyzer cells (SOEC) in particular support this development thanks to their high efficiency at temperatures of more than 800 °C. In addition to the electrochemical properties, the mechanical stability is decisive in the further development of SOEC and the scale-up of the stack production. In this presentation, the authors show tensile tests at room and high temperature of ultra-thin 3YSZ electrolytes for SOEC applications. Based on the results at room temperature, the specimen design as well as the test setup were adapted so that a tensile test at 850 °C could be successfully performed. The tensile test has significant advantages due to the larger material volume and the uniaxiality compared with the biaxial ring-on-ring or ball-on-three-ball bending tests. Nevertheless, the obtained results are compared with the results of the ring-on-ring bending test at room temperature and 850 °C. It will be shown for both test methods that the high temperature strength is still about 50 % of the room temperature strength.

10:20 AM

(ICACC-S3-046-2024) 3000-hour High-temperature Test of an Anode Recycle Blower for a 5 kW SOFC Power System (Invited)

M. Choi*[1]; C. Song[1]

1. InGineers Inc., Republic of Korea

An anode recycling blower (ARB) is a component of a solid oxide fuel cell (SOFC) setup that recycles unreacted combustion gases to improve efficiency. This approach contributes significantly to the overall performance of the SOFC system by increasing fuel utilization. From an engineering perspective, the ARB we designed utilizes foil bearings to support the high-temperature operating shaft and air cooling to ensure reliable motor function. Several tests were conducted to prove the flow performance and durability of the ARB. Extensive validation, including a 5000-hour endurance test at room temperature, has confirmed the robustness of bearings and accessories. The operating environment of SOFC was simulated using the Hotbox capable of simulating a working fluid at a temperature of about 500-600°C. the ARB's durability was evaluated through 3,000 hours of continuous operation while fully integrated into the Hotbox. These tests demonstrated the ARB's ability to withstand the SOFC thermal environment.

10:50 AM

(ICACC-S3-047-2024) Measurements and Modeling of Pressure Effects on Solid Oxide Cell Performance (Invited)

S. Barnett*[1]; J. Grimes[1]

1. Northwestern Univ, USA

Solid oxide cell (SOC) performance is generally expected to improve with pressurization, based both on electrode models and full cell measurements. The present talk will describe recent studies of different oxygen and fuel electrodes that have been carried out on symmetric cells under pressurization and a range of conditions; this data is then modeled to understand their various power-law variations of polarization resistance with pressure. The models for any pair of electrodes can be combined and used to predict the overall performance of the SOC over a wide range of operating conditions. The electrochemical characteristics of Ni-YSZ and GDC-infiltrated Ni-YSZ fuel electrodes in Ni-YSZ-supported cells were measured as a function of total pressure P from 1 to 5 atm in H_2/H_2O fuel mixtures with humidification of 25%, 50%, and 75% and temperatures of 600C and 700C. The electrochemical response of various oxygen electrode materials up to 8 bar. The polarization resistance decreased with increasing pressure in all cases; however, the nickelate and PrO_x-infiltrated STFC electrodes decreased more rapidly than the perovskite oxygen electrodes, and hence are favorable for pressurized applications.

11:20 AM

(ICACC-S3-048-2024) System-level integration of solid oxide electrolysis for reduced degradation in ironmaking plants (Invited)

L. Mastropasqua*[1]

1. University of Wisconsin-Madison, Mechanical Engineering, USA

Renewable hydrogen from high temperature electrolysis may be able to provide an economic pathway to completely decarbonize the ironmaking sector. The concept involves the use of Solid Oxide Electrolysis Cells (SOEC) to produce hydrogen that can be used to convert raw iron ore (Fe_2O_3) into iron (Fe) in a process called hydrogen direct reduced iron (DRI). The use of high temperature SOEC systems unlocks the potential of not only using renewable electricity to produce hydrogen, but to recover high temperature heat from the DRI process and recycle it into the SOEC unit. This process maximizes the exergetic performance of the integrated system and performs better than other low-temperature electrolysis technologies. However, one potential pitfall is that under electrolysis operation, the cathode material is subjected to degradation processes affecting the active sites for electrochemical steam reduction (e.g., Ni coarsening, Ni migration), which increase the exothermicity of the process and generally reduce the efficiency of the cell. This presentation explores how system level strategies may nevertheless limit or reduce the degradation issues identified at the cell level to maintain an almost-constant efficiency throughout the lifetime of the SOEC system. These solutions include integration of external heat sources, control of reaction rates, and operational flexibility.

S6 Advanced Materials and Technologies for Rechargeable Energy Storage

SYMPOSIUM 6: All-solid-state batteries VII

Room: Ballroom 5
Session Chairs: Pieremanuele Canepa, University of Houston; Marm Dixit, Oak Ridge National Laboratory

9:00 AM

(ICACC-S6-040-2024) Materials Design and Integration for High Energy and Long Cycle Life Solid-State Batteries (Invited)

H. Kim*[1]

1. Lawrence Berkeley National Laboratory, Materials Sciences Division, USA

Solid-state Li metal batteries hold great promise as a safe and energy-dense rechargeable battery due to the use of non-flammabile solid-state electrolytes instead of organic liquid electrolytes. However, several challenges remain to be addressed before any solid-state Li metal batteries can be competitive and outperform the existing Li-ion batteries in terms of specific energy, energy density, and cycle life. At Lawrence Berkeley National Laboratory (LBNL), we develop novel materials and integrate them into the solid-state Li metal batteries under the support by the U.S. Department of Energy's Vehicle Technologies Office (VTO). In this presentation, I will discuss the important challenges of the solid-state Li metal batteries, provide an overview of our solid-state battery project, and share our recent updates in the materials development, focusing on the composite cathode and polymer-inorganic solid electrolyte composite membrane.

9:30 AM

(ICACC-S6-041-2024) Next Generation Batteries for Electric Aviation and Space (Invited)

D. Dornbusch*[1]; Y. Lin[2]; W. H. Huddleston[1]; V. Yamakov[3]; R. P. Viggiano[1]

1. NASA Glenn Research Center, USA
2. NASA Langley Research Center, USA
3. Analytical Mechanics Associates, USA

Energy storage plays a critical part in the success of future NASA missions that desire batteries with higher energy density, higher power, and most critically improved safety. These performance requirements stretch beyond that of electric automobile markets and are required for enabling widespread adoption of electric aviation. One approach to improve the safety and energy is the transition to non-volatile solid-state electrolytes (SSE) which promise many advantages over traditional flammable liquid electrolytes and may also be an enabling technology for next generation chemistries such as lithium-sulfur (Li-S). However, significant manufacturing challenges must be overcome before the adoption of such technology. This study will discuss the development of solid electrolytes specifically designed to meet future NASA electric aviation targets with densified thicknesses between 20-30 microns derived using solvent processing techniques, 10-15 times thinner than bulk electrolytes and comparable to commercial polyolefin separators (25 micron) used in commercial liquid lithium-ion cells. Design and development of novel sulfur cathode composites integrating experiment and a particle dynamics model will be discussed. Beyond aeronautics, the harsh enviroment and vast distance of space drive the need for unique cell chemistries and alternative materials will also be briefly discussed.

S7 18th Intl Symp on Functional Nanomaterials & Thin Films for Sustainable Energy Harvesting

SYMPOSIUM 7: Nanomaterials for energy conversion, storage and catalysis

Room: Coquina B
Session Chair: Andreu Cabot, Catalonia Institute for Energy Research

8:30 AM

(ICACC-S7-020-2024) Towards sustainable and scalable synthetic methods for high-performance thermoelectric materials (Invited)

M. Ibáñez*[1]

1. Institute of Science and Technology Austria (ISTA), Austria

Thermoelectricity is the phenomenon of converting heat directly into electricity and vice versa. To maximize the efficiency of this process, precise defect control is required. Herein, we present a synthetic approach in which dense inorganic thermoelectric materials are produced by the consolidation of nanoparticle powders produced in solution. In particular, we will focus on Ag_2Se, a material for the use of thermoelectricity near room temperature, where the library of high-performing materials is minimal. Despite Ag_2Se being a promising candidate, the main problems are the large discrepancy in the reported properties and the struggles to reproduce them. Such divergence appears to arise from the difficulty of controlling the defects present in the material, such as vacancies, interstitial atoms, dislocations, grain boundaries, precipitates, etc. We will show that our solution synthesis allows for precise control of such defects, especially avoiding fluctuations in stoichiometry. Furthermore, we show how we can tune microstructural defects, such as strain, dislocations, and grain boundary density, utilizing the characteristic phase transition of Ag_2Se during the sintering process. Overall, our results will highlight that besides stoichiometry, the microstructure is crucial for tuning Ag_2Se transport properties and how this control can be provided by our novel synthetic route.

9:00 AM

(ICACC-S7-021-2024) Green Chemical Syntheses, Electrophoretic Deposition and Characterization of Nanostructured Bi_2Te_3 (Invited)

H. Batili[1]; B. Hamawandi[1]; A. B. Ergül[1]; M. S. Toprak*[1]

1. KTH Royal Institute of Technology, Dept. of Applied Physics, Sweden

There is an increasing interest to develop thermoelectric (TE) films from pre-made nanoparticles to enable conformally deposited films for waste-heat harvesting from any surface that irradiates heat. 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 has a strong impact on the transport properties. We report on the nanostructured n-type bismuth telluride (Bi_2Te_3) using two bottom-up synthesis methods through microwave-assisted hydrothermal and thermolysis routes. A comparison of the techniques, and the resultant materials' characteristics in terms of microstructure and transport properties are undertaken. Electrophoretic deposition technique has been used for the fabrication of thick films from these nanoparticles on specially designed glass substrates. Electronic transport characteristics were evaluated under a temperature gradient. The presented methods enable large scale production of TE materials. The drastic effect of the surface chemistry on the charge transport is demonstrated with the help of established films. Recent achievement in this area, and the special behavior of "activation" of these films with the use of small molecules are summarized in this talk.

9:30 AM

(ICACC-S7-023-2024) Low-cost nontoxic scalable paint thermoelectric generator for energy harvesting (Invited)

S. Ballikaya*[1]

1. Istanbul University-Cerrahpasa, Engineering Science, Turkey

Most of the thermoelectric (TE) materials used to convert heat energy into electrical energy are expensive and to a certain degree toxic. Moreover, due to the chemical complexity in the synthesis process, some of the TE materials are not reproducible. Similarly, the scarcity of TE materials hampers their scalability. In order to address the above issues, this study presents an inexpensive, nontoxic, scalable, and highly reproducible paint-based TE module for the conversion of heat energy into electrical energy. The results indicate that the as-developed TE module can withstand an operating temperature of up to 160 °C. At a temperature of 57 °C, the highest power factors of the as-synthesised n- and p-type TE paints are 1.34 $\mu W/(cm\ K^2)$ and 1.42 $\mu W/(cm\ K^2)$, respectively. It is also found that the TE module can have higher output voltage when the cold side of the TEG is allowed to float in the air in comparison to when it is in contact with the human body. The performance of the paint-based TE module is measured on five parts of the body namely the chest, palm, leg, wrist, and neck; the wrist has the highest open circuit voltage of 1.9 mV, indicating its suitability for wearable applications. Finally, at a temperature gradient of 30 °C, the maximum output power of 6.8 μW is attained.

10:20 AM

(ICACC-S7-024-2024) Efficient and Stable Pb-free Tin Perovskite Solar Cells with Graphene-Based Functional Composites (Invited)

Y. Hahn*[1]

1. Jeonbuk National University, School of Chemical Engineering, Republic of Korea

Tin-based perovskite (Sn-PS) is one of the most promising candidates in lead-free perovskite solar cells (PSCs), but its poor stability and low power conversion efficiency (PCE) have been main bottleneck towards further development. Here, to develop a stable and efficient Sn-based PSC, nitrogen-doped graphene oxide (N_xGO) has been, for the first time, incorporated in active, hole-transport and interfacial layers. The inclusion of N_xGO slowed the crystallization of Sn-PS and suppressed the Sn^{2+}/Sn^{4+} oxidation, leading to pinhole-free dense films having large grains, reduction of recombination loss, well-matched energy level with Sn-PS, and thus significantly improving the photovoltaic parameters. Compared to the pristine Sn-PS cells, the champion devices with N_xGO-based composites in active, hole-transport and interfacial layers showed dramatic enhancement of photovoltaic parameters. Furthermore, the N_xGO-based cells without encapsulation showed remarkable improvement of long-term stability with sustaining 91% of the initial PCE over 60 days, photo-stability, and reproducibility.

10:50 AM

(ICACC-S7-025-2024) Super Stable Quadruple-cation Bromide Perovskite Solar Cells- From Fundamental Research to Final Application

N. Heshmati*[1]; A. Kulkarni[3]; T. Kirchartz[3]; S. Mathur[2]

1. University of Cologne, Department of Chemistry, Institute of Inorganic Chemistry, Germany
2. University of Cologne, Institute of Inorganic Chemistry, Germany
3. Iek5-Photovoltaics, Forschungszentrum Julich, Germany

Hybrid organic-inorganic lead halide perovskite (LHPs) possess a combination of high-quality average structural order and defect tolerance. Degradation of the perovskite material or the phase transition to the photo inactive crystal structure due to environmental exposure hinders their commercialization. In this research, we designed a novel quadruple-cation LHP mentioned as $(4cat)PbBr_3$. A single crystal of (4cat)PbBr3 was made and investigated utilizing XRD and Raman spectroscopy from 300K to 80K. The analysis indicated a photoactive cubic phase maintains up to 80K, which corresponds to a new range of perovskite stability at lower temperatures. Afterward, thin films of $(4cat)PbBr_3$ were optimized and fabricated with perfect surface coverage and a homogeneous morphology. Ultimately, a fundamental understanding of the optoelectronic properties of thin films approved the superior performance of $(4cat)PbBr_3$, namely a high PL emission and charge carrier mobility. Therefore, the p-i-n structured perovskite solar cells were fabricated yielding a power conversion efficiency close to the Shockley–queisser limit with remarkably high open circuit voltage. With this study, A-site cation engineering improved phase stability and device performance. In conclusion, wide-bandgap bromide perovskites are promising candidates for tandem solar cell and light-emitting diode applications.

11:10 AM

(ICACC-S7-026-2024) Green Solvent Perovskites- One Step Closer To Commercialization Of Perovskite Solar Cells

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1. University of Cologne, Department of Chemistry, Institute of Inorganic Chemistry, Germany
2. Department Of Solution-Processed Materials And Devices, Hysprint Innovation Lab, Helmholtz-Zentrum Berlin For Materialen Und Energie Gmbh, Germany
3. University of Cologne, Institute of Inorganic Chemistry, Germany

Perovskite solar cell technology is moving closer to commercialization, while the environmental effects of solvents involved in perovskite processing are still a concern. A variety of polar aprotic solvents are commonly used for perovskite film fabrication while the majority of them are dangerous solvents according to EHS concerns. Hence, solvent selection is crucial to achieving high performance with the final aim of preventing environmental issues. In this research, we utilized new Cyrene blend solvents, which are bio-based and fully green alternatives to hazardous DMF. As a reference solvent DMF: DMSO, 4:1 Vol% is the most common solvent composition for making a solution and deposition of the thin-film $MAPbI_3$ perovskite. We tried to gradually reduce the amount of DMF in solutions, optimize the thin-film deposition properties, and evaluate the film properties. Ultimately, we could achieve DMF:DMSO: Cyrene-blend 10:20:70 Vol%. In fact, we reduced the amount of DMF from 80% of whole solvents to 10% while the thin-film properties are comparable with the reference sample. Besides, some properties like thin-film absorbance and PL emission were improved by new solvents. Therefore, in this work, we could come one step closer to scalable manufacturing of perovskite thin film.

S9 Porous Ceramics Novel Developments and Applications

SYMPOSIUM 9: Structure and Properties of Porous Ceramics

Room: Coquina D
Session Chairs: Manabu Fukushima, National Institute of Advanced Industrial Science and Technology (AIST); Sawao Honda, Nagoya Institute of Technology

8:30 AM

(ICACC-S9-019-2024) Crystallization of nanofibrous $Zr_xTi_{1-x}O_2$ (x = 0.9–0.2) ceramic materials from electrospun precursors

R. A. Yager*[1]; S. Nealy[1]; A. Stanishevsky[1]

1. University of Alabama at Birmingham, Physics, USA

Nanofibrous $Zr_xTi_{1-x}O_2$(x = 0.9–0.2) ceramics were produced using a high-yield, free surface alternating field electrospinning (AFES) from metal alkoxide precursors. AFES is an uncommon method of electrospinning that can significantly increase production rates when compared with other electrospinning techniques. For $Zr_xTi_{1-x}O_2$ nanofibers, production rates of 4.8–6.4 g/h in terms of the resulting ceramic nanofibers were demonstrated with a single spinneret. The average diameter of $Zr_xTi_{1-x}O_2$ fibers varied between of 190–435 nm, depending on the precursor composition and annealing temperature. Crystallization and phase compositions after thermal processing between 600 and 1200°C of $Zr_xTi_{1-x}O_2$ nanofibers were found to be dependent on the Zi/Ti molar ratios. When the molar fraction of zirconia is > 0.7, the nanofibers are composed primarily of a nanocrystalline titania-rich TiO_2–ZrO_2 solid solution without a separate TiO_2 phase formed. At zirconia molar fraction of 0.5, stable orthorhombic $Zr_{0.5}Ti_{0.5}O_2$ zirconium titanate structure forms above 680 °C. TiO_2 in either the rutile or anatase phase forms as a separate phase in addition to $Zr_xTi_{1-x}O_2$ phases when Zr

molar fraction is 0.2. The results of this investigation demonstrated that ZrO_2-TiO_2 ceramic nanofibrous materials can be prepared with precisely tailored compositions depending on desired applications using free surface AFES.

8:50 AM

(ICACC-S9-020-2024) The role of microstructural defects in grain growth behavior

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1. Carnegie Mellon University, Materials Science and Engineering, USA

Abnormal grain growth and porosity can lead to uncontrolled microstructural heterogeneity and reduced performance in ceramics. Abnormal grain growth in alumina is generally attributed to impurities. The interaction of impurities with porosity must be studied to thoroughly understand grain growth behavior and to accurately predict abnormal grain growth behavior. This work investigates the role of intentionally induced, large, isolated pores on the behavior of abnormal grains in calcia doped alumina. The effects of pores on abnormal grain growth are presented. Changes in the matrix grain size distributions are also reported. Relative grain boundary energies are compared for systems with and without large pores as an indirect indicator of solute segregation. The results highlight the importance of considering free surfaces, such as pores, in the study of abnormal grain growth.

9:10 AM

(ICACC-S9-021-2024) Superhigh porosity ultra-high temperature ceramics with high strength and low thermal conductivity

Z. Wu*[1]; J. Wang[1]

1. Institute of Metal Research, Chinese Academy of Sciences, Shenyang National Laboratory for Materials Science, China

Due to the fast development of aerospace industry, ultra-high temperature thermal insulation materials are urgently requested to resist the drastic aerodynamic heating. At present, ultra-high temperature ceramics (UHTCs) are the best choice due to their excellent capability at ultra-high temperatures. Herein highly porous UHTCs were prepared by foam-gelcasting-freeze drying method. The strategies of multi-component modification and realizing superhigh porosity are employed to develop the eminent thermal insulators. The result shows that the multi-component modification and high porosity greatly reduce the thermal conductivity of UHTCs. Moreover, realizing superhigh porosity (95.9%-86.4%) obtains the lightweight UHTCs with low density of 0.30-1.27 g/cm^3. Besides, porous UHTCs exhibit high strength and improved oxidation resistances. This work indicates highly porous multi-component UHTCs are ideal candidates for ultra-high temperature insulation in extreme environments.

9:30 AM

(ICACC-S9-022-2024) Structure and properties of freeze-tape cast phyllosilicate ceramics

P. Letang[1]; G. Lecomte-Nana*[1]; S. Abdelouhab[3]; B. Nait-Ali[1]; M. Gonon[2]; L. Jouego Doho[1]; E. Juste[3]; C. Peyratout[1]

1. University of Limoges, IRCER (UMR CNRS 7315), ENSIL-ENSCI, Industrial Ceramics, France
2. University of Mons, Belgium
3. BCRC, Research & Technological Support Department, Belgium

Kaolinite- and halloysite-rich slurries were optimized to manufacture porous ceramics shaped by freeze tape casting (FTC). Two kaolins were used as raw materials, a commercial one provided by Imerys named KCR and the other one originating from Cameroon named KMY. The halloysite-rich clay was collected in West-Cameroon and labeled HCR. The obtained FTC samples were freeze-dried prior to their sintering at 1200, 1300 and 1400°C. Structural characterizations were performed to understand the effective changes in main phases, in line with mechanical resistance and thermal conductivity. The porosity of dried samples was very similar. The stress to rupture values for KCR and KMY were greater than for HCR. The thermal conductivity values were low, due to the high porosity combined with the presence of organics within dry tapes. Sntering at 1200°C leads to an increase in porosity. Kaolinite-based samples exhibited improved stress to rupture values in comparison to HCR samples. The latter trend resulted from the pore size distribution and, to the type and amount of mullite within these samples. Despite the high porosity of sintered samples, their mechanical resistance was less affected, while the thermal conductivity increased. In the case of HCR samples, a sintering at 1300°C allowed improving their stress to rupture values. This behavior suggested a profitable effect of the porosity architecture, thanks to FTC.

10:10 AM

(ICACC-S9-023-2024) Exploring Shape Memory and Superelastic Transformations in Bulk-Scale, Porous Zirconia

L. Quinn*[1]; R. Esteves[3]; P. C. Latorre-Suarez[2]; G. R. Rossman[1]; S. Raghavan[2]; K. Faber[1]

1. California Institute of Technology, USA
2. Emby-Riddle Aeronautical University, Aerospace Engineering, USA
3. University of Central Florida, USA

Shape memory materials can undergo reversible, diffusionless martensitic transformations that enable them to exhibit shape memory or superelastic behavior. Although shape memory alloys are more commonly commercialized, shape memory ceramics such as ceria-stabilized zirconia can also take advantage of this martensitic transformation. However, the volume expansion that accompanies the martensitic transformation in zirconia limits the exploitation of superelastic and shape memory behaviors in polycrystalline systems, as the change in volume causes stress mismatch at grain boundaries that leads to cracking and failure. To prevent this premature failure, this work utilizes freeze casting to create a network of honeycomb-like pores. By increasing the ratio of surface area to volume in this manner, the volume expansion of the martensitic transformation is accommodated in a polycrystalline system. Shape memory and superelastic behavior can then be explored over several cycles, using X-ray diffraction and Raman spectroscopy to track the extent of the transformation over millimeter- and micrometer-scale volumes. Additionally, the use of Raman spectroscopy to track the martensitic transformation presents opportunities both to monitor shape memory and superelastic behaviors in-situ, and to more closely examine the effect of pore wall thickness on the shape memory and superelastic behaviors.

S2 Advanced Ceramic Coatings for Structural/Environmental & Functional Applications

SYMPOSIUM 2: Coatings for wear, corrosion protection

Room: Flagler C
Session Chair: Peter Mechnich, DLR - German Aerospace Center

8:40 AM

(ICACC-S2-040-2024) Polymer-Derived Ceramic Coatings for Corrosion Protection

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1. Indian Institute of Technology Ropar, Metallurgical and Materials Engineering, India

In this work, polymer-derived silicon oxycarbide (SiOC) layer was coated on stainless steel to enhance the corrosion resistance in seawater environment. SS304 was dip-coated with vinyl-functionalized polysiloxane solution and then pyrolyzed under argon environment at 800°C. SiOC layer with a thickness of ~1 mm was obtained after two times coating/pyrolysis steps. Structural characterization of the samples were performed by NMR, FTIR, XRD, Raman spectroscopy, optical microscopy, SEM, and XPS. Electrochemical characterization of SiOC-coated SS304 samples in a 0.6 M NaCl solution showed protection efficiency of about 99 % indicating significantly improved corrosion behavior of SiOC-coated SS304 compared to that of uncoated SS304.

9:00 AM

(ICACC-S2-041-2024) Microstructural Analysis of High entropy alloy coating

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2. RRCAT, India
3. SVNIT, India

Surface coating improves functional properties of substrate. Coating composed of multi component high entropy alloy(HEA) consisting of at least five principal elements, each of which has an atomic concentration between 5% and 35% are widely researched globally due to their excellent properties like wear resistance, corrosion resistance and high hot hardness. In the present investigation HEA coating consisting of equiatomic Co, Cr, Ni, Mo, Fe were deposited on mild steel using laser cladding technique. Further to modify surface properties of HEA metal composite coatings (reinforced using carbides, oxides or ceramics) are commonly added into the metal matrix to enhance the performance of the composites. In present investigation HEA coating is reinforced with 5%TiB_2,10%TiB_2 and 15%TiB_2. The microstructural characterization was carried out using optical microscopy, XRD,FESEM and EDS. Mechanical characterization was carried out using micro hardness testing. Micro hardness testing indicates hardness increasing as the percentage of TiB_2 reinforcement increases and maximum hardness is achieved at the 15 weight%. Similar behaviour is observed for erosion test.

9:20 AM

(ICACC-S2-042-2024) Microstructural evolution and in-situ stress recording in enamel coating for automotive applications

A. Lejeune*[1]; J. Chevalier[1]; L. Gremillard[2]; S. Meille[2]; P. Steyer[2]; J. Jamart[3]

1. INSA Lyon, Materials Science, France
2. INSA, Materials, Engineering and Science, France
3. Saint-Gobain, France

In automotive applications, black enamels are mostly used for their opacifying property, especially on SLS glass substrates for products such as windshields. Enamel undergoes several microstructural transformations: sintering, glass transition, crystallization, leading to a weakening of the substrate where enamel is deposited. The resulting difference in thermomechanical properties between the substrate and the enamel layer is of great importance in the induced weakening mechanisms, but also in the generation of optical defects in areas of glazing products, where high optical quality is required. In the present work, a standard enamel used in the automotive field was studied and characterized via in-situ monitoring methods (XRD, SEM, curvature measurement) to establish correlations between the chemical composition, microstructural evolutions, and thermomechanical properties. Crystallization induced during thermal treatments of the enamel layer between 25°C and 700°C seems to strongly influence both the biaxial Young's modulus of the layer and its coefficient of thermal expansion. This also seems to impact the apparent viscosity of this material, and thus the phenomena of stress relaxation by viscous flow. It was observed that the chemical composition of enamel (glass frit, pigments) strongly influences its rheological behavior at high temperature and mechanical properties.

9:40 AM

(ICACC-S2-043-2024) Thermochemical interactions of yttria-stabilized zirconia and molten lunar regolith simulants

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1. Caltech, Materials Science, USA
2. California Institute of Technology, USA
3. NASA Glenn Research Center, Environmental Effects and Coatings Branch, USA
4. NASA Marshall Space Flight Center, USA

Oxygen produced from lunar resources through in-situ resource utilization (ISRU) is critical to maintaining a permanent human presence on the lunar surface. Molten regolith electrolysis and carbothermal reduction are two promising ISRU techniques for generating oxygen directly from lunar regolith, which is primarily a mixture of oxide minerals; however, both processes require operating temperatures of 1600°C to melt lunar regolith and dissociate the molten oxides. These conditions limit the use of many oxide refractory materials due to rapid degradation resulting from reactions between the refractory materials and molten lunar regolith. Yttria-stabilized zirconia (YSZ) is a promising refractory oxide to provide containment of molten regolith. This work focuses on corrosion studies of YSZ powders and dense YSZ crucibles in contact with molten lunar mare and highlands regolith simulants at 1600°C. The interactions between YSZ and molten regolith are characterized using SEM/EDS, XRD, and EBSD with an emphasis on elemental and microstructural analysis to assess reactivity and degradation of YSZ. Due to lunar regolith's similar composition to calcium-magnesium-aluminosilicates (CMAS) and YSZ's usage as a thermal barrier coating, these interactions can serve to inform YSZ/CMAS behavior by simulating cases of elevated CMAS/YSZ ratios and for higher than intended gas turbine temperatures.

FS1 Bioinspiration/Green Processing & Related Technologies of Advanced Materials

Focused Session 1: Bioinspiration, Green Processing, and Related Technologies of Advanced Materials

Room: Ponce de Leon
Session Chair: Derk Joester, Northwestern University

10:20 AM

(ICACC-FS-010-2024) Bioprocess inspired fabrication: From biominerals formation to materials fabrication (Invited)

Z. Zou*[1]

1. Wuhan University of Technology, China

Living organisms are able to produce various types of materials to fulfill their biological functions. For example, biomineralization is the process by which organisms produce hard biominerals for supporting/protecting their soft body or eating, usually with excellent mechanical properties, such as shells, bones and teeth, etc. Understanding the formation process of these biological materials, from biological control and chemical reaction to the structure-function relationship, could provide inspirations for fabrication of functional materials. Here, I will present our recent studies on investigating the formation process of shark tooth, the crystallization mechanisms of amorphous materials, and the fabrication of dense calcium carbonate ceramic at room temperature.

10:50 AM

(ICACC-FS-011-2024) Bioinspired Tendon-like Strong and Tough Conductive Organo-Hydrogels via Freeze-Casting Assisted Solution Substitution

W. Zhai*[1]; X. Dong[1]; X. Guo[1]; G. Zou[2]; H. Gao[3]

1. National University of Singapore, Mechanical Engineering, Singapore
2. A*STAR, Institute of High Performance Computing, Singapore
3. Nanyang Technological University, School of Mechanical and Aerospace Engineering, Singapore

Biological soft tissues, such as tendons and cartilage, exhibit an exceptional combination of strength, flexibility, and message-passing ability owing to their hierarchical structures. This could be an inspiration for strong and tough conductive hydrogels, which are among the most sought-after materials for flexible electronics. A bioinspired strategy for tough conductive organo-hydrogels via freeze-casting assisted solution substitution (FASS) will be presented. This FASS strategy enables the formation of organo-hydrogels in one step with tendon-like hierarchical anisotropic structures coupled with synergistic strengthening and toughening effects across multiple length scales. As an exemplary material, polyvinyl alcohol organo-hydrogel with solvent content up to 87 wt.% exhibits a combination of high strength, high stretchability, ultra-high toughness, as well as high ionic conductivity with excellent strain sensitivity. Via finite element simulation and molecular dynamics simulations, we will present the underlying multiscale multimechanisms come from the gel's hierarchical structures, which include microscale anisotropic honeycomb–structured fiber walls and matrix, with a modulus of 8.96 and 0.73 MPa, respectively; hydrogen bond–enhanced fibers with nanocrystalline domains; and cross-linked strong polyvinyl alcohol chains with chain-connecting ionic bonds.

S6 Advanced Materials and Technologies for Rechargeable Energy Storage

SYMPOSIUM 6: Solid electrolytes for batteries VIII

Room: Ballroom 5
Session Chairs: Chunmei Ban, University of Colorado, Boulder; Huiwen Ji, University of Utah

10:20 AM

(ICACC-S6-042-2024) Moving Beyond Seeing is Believing: Advanced Synchrotron Characterization Studies for Solid-State Batteries (Invited)

M. Dixit*[1]

1. Oak Ridge National Laboratory, USA

X-ray tomography has been widely used to probe the failure mechanisms of solid-state batteries. While powerful, this technique has fundamental limitations in probing the origin mechanisms of failure. Dynamic electrochemical and chemical processes at the interfaces of solid-state batteries cannot be effectively captured unless they manifest as microstructural changes spanning several microns in ideal cases. In this talk, I will present diffraction- and spectroscopy- based imaging approaches to probe electro-chemo-mechanical dynamics in solid-state battery systems. Specifically, I will discuss the utilization of both high-energy X-ray diffraction, far-field high-energy diffraction microscopy and tomography measurements to uncover the existence of phase heterogeneity within the solid electrolyte's bulk that can perturb the local chemical and mechanical properties. I will also discuss a study employing redox-sensitive mapping of Li | LAGP solid electrolyte interfaces using confocal x-ray absorption microscopy. I will also discuss evolution of phase distribution in composite cathodes for solid-state batteries evaluated using a absorption microscopy approach. These innovative techniques provide novel approaches to investigate solid electrolyte interfaces, offering insights into the temporal and spatial aspects of solid electrolyte failure mechanisms.

10:50 AM

(ICACC-S6-043-2024) Disorder but more Ionic Conductive – Data Driven Design of Materials for High-Rate Solid-State Batteries with Disordered Metal Oxides (Invited)

B. Ouyang*[1]

1. Florida State University, Chemistry and Biochemistry, USA

Early theory for disordered matters usually suggests that disorder can be detrimental to transport of ion and charge transport. The underlying physical intuition is that the randomness of lattice can trap carriers (ions, electrons, or charge) and kill the long-range diffusion. More recently, several emerging works have indicated that in disordered metal oxides, even if carrier trapping can still exist, there could be a highly ionic conductive pathway formed with careful control of microstructures at different scale. To better illustrate this idea, several case studies that design highly disordered metal oxides into high-rate battery electrodes and superior solid-state electrolytes will be presented. The close interplay between multiple data-drive approaches and advanced spectroscopy microscopy will also be demonstrated.

11:20 AM

(ICACC-S6-044-2024) Characterization of Electrochemical Performance of Reaction-Sintered LATP ($Li_{1.3}Al_{0.3}Ti_{1.7}(PO_4)_3$) Solid Electrolytes

D. Kim*[1]; Y. Cho[1]; D. Kim[1]

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

All-solid-state batteries (ASSBs) offer superior safety and volumetric energy density compared to conventional liquid electrolyte-based batteries. NASICON-structured phosphate electrolytes have gained attention due to their favorable high ionic conductivity and chemical stability. LATP ($Li_{1.3}Al_{0.3}Ti_{1.7}(PO_4)_3$) solid electrolyte exhibits high ionic conductivity and phase stability but suffers from low sintered density. Herein, we introduced a single step pressureless reaction-sintering of the ceramic electrolyte with LATP seed grains. Through the utilization of the reaction-sintering process, we successfully sintered LATP ceramics with a density exceeding 96.4% and a conductivity of 1.6×10^{-4} S cm^{-1} at room temperature. The LATP electrolytes of 1 mm thickness were assembled into symmetrical battery cells with Li-metal anode. The critical current density and long-term cyclability were evaluated. The results showed that the LATP-symmetric cells exhibited stable cycling performance for over 70 hours at a current density of 0.2 mA cm^{-2}. The relationship between the microstructural parameters and electrochemical performance of the LATP will be discussed.

11:40 AM

(ICACC-S6-045-2024) A comparative study on the oxide doping of promising NASICON-structured glass-ceramic electrolytes for solid-state lithium batteries

S. Saffirio*[1]; F. Smeacetto[1]; S. Fiorilli[1]; A. Tarancón[3]; A. Sabato[2]; C. Gerbaldi[1]

1. Politecnico di Torino, Applied Science and Technology, Italy
2. IREC, Nanoionics and Fuel Cells, Spain
3. IREC / ICREA, Spain

New glass-ceramic $Li_{1.4}Al_{0.4}Ti_{1.4}Ge_{0.4}(PO_4)_3$ (LAGTP) and $Li_{1.5}Al_{0.3}Mg_{0.1}Ge_{1.6}(PO_4)_3$ (LAMGP) NASICON-type solid-state electrolytes were developed through a melt-casting method. The effect of adding different sintering aids on grain cohesion and, thus, on the overall ionic conductivity, was investigated for the two systems. To this purpose, their thermal, crystalline and microstructural properties were evaluated through DTA, HSM, XRD and SEM analyses. The corresponding ionic conductivities were assessed through electrochemical impedance spectroscopy (EIS) between -20 and 80 °C, along with the electrochemical stability window (ESW) of the most conductive samples. Heat-treated bulk materials showed improved performance compared to their powder-sintered counterparts, and the addition of sintering aids - particularly B_2O_3 - demonstrated to be effective on grain cohesion for both systems. Ionic conductivity values of 0.4 and 0.2 mS cm^{-1} were recorded at 20 °C for bulk B_2O_3-added LAGTP and LAMGP, respectively. The two systems were found to be electrochemically stable up to about 4.8 V vs. Li^+/Li, making them appealing for high-V operation. LAMGP powders were also sintered and crystallized by ultra-fast high-temperature sintering (UHS) method, providing new insights into the impact of UHS technique on the physical and electrochemical properties of LAMGP.

S2 Advanced Ceramic Coatings for Structural/Environmental & Functional Applications

SYMPOSIUM 2: High- and ultrahigh temperature coatings

Room: Flagler C

Session Chair: Ravisankar Naraparaju, DLR - German Aerospace Center

10:20 AM

(ICACC-S2-044-2024) Experimental and Analytical Studies on Stand-off Thermal Protection System for Reentry Space Vehicles Applying Low Thermal Emissivity Ceramics

Y. Sudo*[1]; T. Ogasawara[2]; S. Akamine[4]; T. Aoki[3]

1. Tokyo University of Agriculture and Technology, Graduate School of Engineering, Japan
2. Tokyo University of Agriculture and Technology, Japan
3. Japan Aerospace Exploration Agency, Advanced Composite Research Center, Institute of Aeronautical Technology, Japan
4. Okinawa College, National Institute of Technology, Japan

For upper-stage reusable rockets and hypersonic aircrafts, the airframe temperature reaches 800-1000 degrees Celsius or higher due to aerodynamic heating during reentry and cruising. Therefore, it is necessary to apply a thermal protection system (TPS) to the outer surface of the main structure of the airframe. While lightweight ceramic tiles have been widely applied as TPS materials for re-entry space planes, standoff TPS, in which heat-resistant material plates are fixed to the main structure via posts, is also expected from the viewpoint of maintainability and robustness. When thermal radiation is used in addition to thermal conduction as the internal heat transfer of the standoff TPS, the application of low thermal emissivity materials is advantageous. In this study, we propose a standoff TPS structure utilizing a novel magnesium aluminate spinel based low thermal radiation ceramics (THERMOSCATT, Coors Tech.) and its coating. Quantitative verification of the adiabatic properties of the application of the low thermal emissivity ceramics was performed by IR lamp heating tests under vacuum on structural element models. In addition, parametric calculations of the standoff TPS structures using finite element analyses were performed to verify its thermo-mechanical properties, adiabatic properties, weight, etc.

10:40 AM

(ICACC-S2-045-2024) Combinatorial and high throughput screening on $Ta_xHf_{1-x}C$ ceramic: Exploration of composition with optimized hardness and oxidation resistance

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

1. Institute of Metal Research, Chinese Academy of Sciences, Advanced Ceramics and Composites Division, China

Due to high melting point, high hardness, and good oxidation resistance, $Ta_xHf_{1-x}C$ ternary ceramics are considered as advanced ultra-high temperature coating materials to protect C_f/C composites from oxidation and ablation. Their properties are significantly tuned by Ta/Hf ratio, while continuous solid solution over the entire compositional space makes it time-consuming and insufficient to explore optimal Ta/Hf ratio by conventional trial-and-error method. In the present work, hundreds of $Ta_xHf_{1-x}C$ samples with continuous gradient covering the compositional range of $Ta_{0.8}Hf_{0.2}C$ to $Ta_{0.2}Hf_{0.8}C$ are parallel prepared by efficient synthesis of combinatorial material chip, and high throughput screening are conducted. $Ta_{0.4}Hf_{0.6}C$-$Ta_{0.45}Hf_{0.55}C$ are identified as potential hardest $Ta_xHf_{1-x}C$ ceramics through mapping hardness and elastic modulus values to various compositions. Moreover, phase constituents and oxide scale structures after oxidation in static air at 1200-1400 °C are systematically investigated, revealing that optimal compositional range for the formation of dense $Hf_6Ta_2O_{17}$ protective layer is $Ta_{0.4}Hf_{0.6}C$-$Ta._{0.55}Hf_{0.45}C$. High throughput experimentation provides a broad composition library, and the obtained results pave the way for compositional design of $Ta_xHf_{1-x}C$ ceramics in ultra-high temperature applications.

11:00 AM

(ICACC-S2-046-2024) Enhancing the oxidation resistance of transition metal carbide thin films through Al and Si alloying

S. Richter*[1]; E. Ntemou[2]; D. Primetzhofer[2]; T. Wojcik[1]; O. Hunold[4]; S. Kolozsvári[3]; P. Polcik[3]; J. Ramm[4]; H. Riedl[5]

1. Christian Doppler Laboratory for Surface Engineering of high-performance Components, TU Wien, Austria, Austria
2. Uppsala University, Department of Physics and Astronomy, Sweden
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4. Oerlikon Surface Solutions AG, Oerlikon Balzers, Liechtenstein
5. TU Wien, Institute of Materials Science and Technology, Austria

Transition metal carbides (TMCs) are well-known for their exceptional mechanical properties, combined with high-temperature phase stability and melting points above 3000 °C. However, these extraordinary high-temperature characteristics are degraded by the inherent oxidation resistance of the materials at hand. Therefore, this work focuses on an alloying strategy, comprising Al and Si as strong oxide-forming elements, to position TMCs as promising materials for novel applications in demanding environments. In more detail, group IV to VI transition metal carbides are screened via a combinatorial physical vapor deposition (PVD) based approach, co-sputtering the respective ceramics with Al/Si. The broad range in structural and chemical compositions is characterized by XRD, nanoindentation, and ERDA-calibrated XRF to overcome the difficulties in accurate chemical quantification. In a second step, a limited number of compositions has been selected based on structure-mechanical criteria for oxidation treatments. The scale formation is studied using in-situ XRD, performed in synthetic air up to 1200 °C, as well as oxidative treatments in conventional box furnaces. The outcome of this study does not only focus on the fundamental mechanisms of the oxidation behavior of these TMCs, but also reveals insights on the formation of ternary fcc-structured TM-Al/Si-C solid solutions via PVD.

11:20 AM

(ICACC-S2-047-2024) Revisiting the structure-property relationship of superhard TiB_{2+z} coatings

A. Hirle*[1]; C. Fuger[1]; R. Hahn[1]; T. Wojcik[1]; P. Kutrowatz[1]; M. Podsednik[2]; O. Hunold[3]; P. Polcik[4]; S. Kolozsvári[4]; H. Riedl[5]

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As one representative of ultra-high temperature ceramics (UHTCs), titanium diboride (TiB_2) is well-known for its excellent properties, such as super hardness, good thermal conductivity, or chemical stability. Based on these unique features, it is used for diverse bulk and thin film purposes. Although TiB_2 thin films have already been thoroughly investigated, there are still unresolved questions regarding stoichiometry's impact and structural imperfections (B-rich tissue phases) on the mechanical properties. For a detailed investigation, we synthesized a stoichiometric variation from $TiB_{2.04}$ to $TiB_{4.42}$ by DC magnetron sputtering. We characterized the coatings using a broad set of techniques including SEM, TEM, nanoindentation, and in-situ microcantilever bending experiments to assess the fracture behavior. For achieving super hardness (> 40 GPa), a (0001) orientation is essential, whereas for (10-11) and (1000) oriented coatings, we observed a linear decrease in hardness down to ≈ 30 GPa. The fracture toughness (K_{IC}) features a tissue phase-dependent behavior with increasing values from 2.51 ± 0.14 MPa√m for $TiB_{4.42}$ to 3.55 ± 0.16 MPa√m for $TiB_{2.22}$ due to a decreasing tissue phase width, showing the impact of grain boundary constitution on the fracture properties. In summary, our results highlight the effects of texture and morphological design on PVD grown TiB_{2+z} coatings.

11:40 AM

(ICACC-S2-048-2024) Synthesis of Ultrahigh Temperature Ceramic Coatings on Carbon-Carbon Composites using Selective Laser Reaction Synthesis

J. B. Spicer*[1]; A. J. Yuan[2]; D. Zhang[2]; A. B. Peters[1]; E. Berry[1]; Y. Rhim[2]

1. Johns Hopkins University, Materials Science and Engineering, USA
2. Johns Hopkins University Applied Physics Laboratory, USA

Ultrahigh temperature ceramics (UHTCs) are often considered for coating applications that require materials to perform under conditions that might include high temperatures, reactive species, surface wear, and mechanical loading. Various non-oxide UHTCs have favorable properties for these applications, but processing them using laser-based methods to create coatings has received little attention. This is particularly the case when these coatings have special geometries that might only be produced using additive manufacturing methods. When applied to UHTCs, these methods have not been as successful as they have been for metal-based materials systems. In this work, selective laser reaction sintering (SLRS) methods were used for production of near net-shape UHTC parts and for coatings of HfC on carbon-carbon composites. To mitigate conversion-induced stresses associated with SLRS, composite metal/metal oxide precursors were formulated to compensate for the volume changes of the metal (which expands) and the metal oxide precursor (which contracts). These formulations reduce conversion stresses by minimizing volumetric changes, but interparticle adhesion must occur as well in order to produce robust UHTC coatings. SLRS may be a viable option for producing complicated coatings of UHTC materials that are not readily produced using current processing methods.

S9 Porous Ceramics Novel Developments and Applications

SYMPOSIUM 9: Porous Ceramics for Environmental, Energy, Biological and Functional Applications I

Room: Coquina D
Session Chairs: Gisele Laure Lecomte-Nana, ENSCI;
Arianna Bertero, Politecnico di Torino

10:30 AM

(ICACC-S9-024-2024) HT characterization of the oxygen transport in CeO_2 ceramics for the radiative design of 3D porous architectures for H_2 production high solar flux

L. Gaillard*[1]; B. Rousseau[2]; P. Geffroy[3]

1. LTEN, France
2. LTN UMR CNRS 6607, France
3. Laboratoire SPCTS, France

Solar thermochemistry is a green way to produce H_2 at high temperature (HT). This technology is based on a 2-step redox thermochemical cycle, heated by a concentrated solar power device. The most commonly used material is cerium oxide (CeO_2). The oxidation step is done at 1500°C with the introduction of water in the reactor. This water vapor is reduced at 900°C by the oxide, forming then hydrogen. The main challenge of this method is to rise the efficiency up from 5% to 20%. One of the main locks of this improvement is the lack of understanding of the radiative and oxygen transport properties within CeO_2 porous architectures. Nowadays, thermal radiative properties of CeO_2 are known only at 20°C and the oxygen transport properties are not quite evaluated at HT. This study aims to apprehend the relations between these properties, depending on the temperature and the partial pressure of oxygen. This is done by measuring the ionic and electronic conductivities of CeO_2 pellets at the conditions of interest, and the reaction kinetics linked to it. This presentation will show the characterization of ionic and electronic conductivities of CeO2 ceramics, demonstrating it's mainly electronic feature. The measured values can be used to compute the radiative properties of 3D architectures with improved volumic absorption of solar rays.

10:50 AM

(ICACC-S9-026-2024) A polymeric aerogel for oil-water separation

H. Hayes*[1]; P. Kroll[1]

1. University of Texas, Arlington, USA

Polymeric siloxane- based aerogels were prepared through the hydrosilylation of polymethylhydrosiloxane (PMHS) with vinyl-terminated methoxysilanes (VMS). A superhydrophobic and mechanically flexible material was produced which can be used for oil-water separation. After modification, the polymer was then condensed to a wet gel, washed, and dried under ambient conditions over two days. The first step of the synthesis was characterized through FTIR, H^1 NMR, and TG analysis. The dry gel was also characterized with these techniques as well as others. Thermogravimetric analysis indicates thermal stability up to 300 °C which is well above the typical operating temperature for oil spill sorbents. Different properties are attained by changing the ratio of PMHS to VMS and by choice of VMS. Mechanically stable low-density aerogels with superhydrophobic characteristics were obtained. Their usage to separate oil and water mixtures was demonstrated indicating the potential for an environmental remediation technique.

11:10 PM

(ICACC-S9-017-2024) Application of the Japanese wood joining technique for ceramic interlocking structures

P. Hoffmann*[1]; T. Fey[1]

1. Friedrich-Alexander University Erlangen-Nürnberg, Department Material Science and Engineering, Germany

Modular miniaturized materials based on ceramic building blocks offer great potential due to their volume-dependent improved mechanical properties. They are often connected by a secondary bonding phase and are therefore fixed, unchangeable in terms of their properties. We are investigating self-supporting interlocking constructs with inherent stability consisting of detachable and tunable ceramic building blocks. The prism blocks are truncated cuboids with tilted angles and a coupling element based on the Japanese wood joining technique. Due to the individually replaceable blocks variable material properties for specific application fields and local control is provided. The Young's modulus and energy absorption was determined using 4-point bending and the influence of different joinings with various interlocking parameters as well as different materials and porosities of each block were investigated. In particular, the adhesive-free joint was analyzed microstructurally using μCT evaluation and FE simulations. Thus, this advanced multimaterial approach is promising for structured cellular ceramics with tailored and exchangeable properties.

FS1 Bioinspiration/Green Processing & Related Technologies of Advanced Materials

Focused Session 1: Bioinspiration, Green Processing, and Related Technologies of Advanced Materials

Room: Ponce de Leon
Session Chair: Stephan Wolf, Friedrich-Alexander-University Erlangen-Neurnberg

1:30 PM

(ICACC-FS-012-2024) Matrix proteins in biomineralization of molluscan shells (Invited)

M. Suzuki*[1]

1. The University of Tokyo, Department of Applied Biological Chemistry, Japan

Biominerals that are composed of inorganic minerals and organic matrices. Molluskan shells are typical biominerals containing calcium carbonates and few organic molecules. Pinctada fucata that is the pearl oyster used for pearl aquaculture has been investigated for the shell formation process. The shell of P. fucata has layered structure of the inner part of the nacreous layer, outer part of the prismatic layer and hinge ligament. In the nacreous layer that is a brick-and-mortar microstructure, we identified a novel matrix protein of Pif consisting of two proteins, Pif 97 and Pif 80. Pif 80 is high acidic protein and has calcium carbonate binding activity. The hinge of bivalve is composed of the ligament that contains the nanofiber of calcium carbonates surrounded by dense organic frameworks. In the ligament, we identified a novel acidic peptide of LICP that consists of 10 amino acids with N-terminal pyroglutamate amino acid. We also discovered a novel protein of prismalin-14 and chitinolytic enzymes from the prismatic layer. These matrix proteins play important roles to form the fine microstructure of calcium carbonate. These new insights of organic molecules in molluskan shells will develop the new technologies for material and environmental sciences.

2:00 PM

(ICACC-FS-013-2024) Self-assembled silica colloids as lightweight and tough bioinspired composites (Invited)

F. Bouville*[1]; V. Vilchez[1]; S. Zhou[1]

1. Imperial College London, Department of Materials, United Kingdom

Highly mineralised biological materials present unique microstructure leading to toughness well in excess of their constituents. Nacre, enamel, the dactyl club of the mantis shrimp present enhanced fracture resistance by leveraging near-perfect stacking of building blocks and anisotropic microstructures. To date, numerous research efforts have focused on manufacturing bioinspired structures and achieved excellent mechanical response. However, synthetic highly mineralized composites have not yet exhibited highly delocalized damage during fracture propagation. This toughening mechanism is responsible for the high damage resistance found in natural counterparts. In this study, we decided to test whether this delocalized damage can only be triggered in more ordered structure. We synthesised mono-dispersed colloidal silica rods through a sol-gel process and controlled their assembly into tens-of-millimetre-sized highly packed colloidal crystals. Upon resin infiltration, these crystals formed highly textured crystalline-like composites. Our results demonstrate that highly packed and ordered microstructure introduces branching, deflection, and delocalized damage at the hundreds of micron scale in front of a crack. In addition, these highly mineralised composites are made entirely close to room temperature, including the mineral part, opening a way for manufacturing tough composites under milder conditions.

2:30 PM

(ICACC-FS-014-2024) Architected Composites: From Bioinspired Design to Multi-functionalities (Invited)

T. Magrini*[4]; A. Studart[1]; F. Bouville[3]; C. Daraio[2]

1. ETH Zurich, Switzerland
2. California Institute of Technology, USA
3. Imperial College, United Kingdom
4. Eindhoven Institute of Technology, Netherlands

Lightweight composites have become a key component of energy-efficient automotive- and aviation products. However, the design of composites that combine high strength and high resistance to fracture still constitutes a challenge, as these properties are often mutually exclusive in synthetic materials. In my talk, I will display how we can take inspiration from the internal architecture of biological structural materials to design and manufacture architected composites with unprecedented structural properties. I will highlight new classes of composites, reinforced across multiple lengthscales by a regular arrangement of particles, and how they can be designed to display optical and sensing functionalities in combination with high strength and fracture toughness. Additionally, I will highlight a novel strategy for the design of architected composites, inspired by the non-periodic structure of termite nests and fruit peels. I will display how computer-aided stochastic algorithms can design irregular reinforcing networks in architected composites, and how their connectivity and local assembly influence materials' properties. Finally, I will show how we can tailor the spatial and temporal propagation of fractures in these complex architectures, combining different reinforcing networks into spatially determined meso-scale assemblies.

FS2 Advanced Materials for Thermoelectric and Thermionic Energy Conversion

Focused Session 2: Advanced Materials for Thermoelectric and Thermionic Energy Conversion

Room: Coquina C
Session Chairs: Jeff Snyder, Northwestern University; Sunmi Shin, National University of Singapore

1:30 PM

(ICACC-FS2-008-2024) Tuning and exploring Thermoelectric materials (Invited)

N. Pryds*[1]

1. Technical University of Denmark, Denmark

In recent decades, there has been a significant push to enhance thermoelectric performance through optimization of carrier concentrations and precise determination of material thermal diffusivity. This abstract illuminates two groundbreaking areas in the realm of thermoelectricity. Firstly, it introduces the innovative development of an electric double-layer (EDL) transistor-based gate. This gate, employing ionic liquids, adeptly tunes thermoelectric properties at room temperature. It does so across a broad spectrum of carrier concentrations in the thin films of Nb-doped SrTiO3 (Nb-STO), showcasing versatility and precision. Secondly, the abstract unveils a pioneering method designed for the swift and accurate measurement of thermal diffusivity in individual grains, each with varying orientations. This is expertly achieved through the utilization of the M4PP technique. The proposed method offers rapid and pinpoint thermal characterization and paves the way for an in-depth exploration and understanding of microstructures, presenting a vista of opportunities in the field. Each discovery and method detailed herein contributes to the intricate tapestry of thermoelectricity, providing valuable insights and tools for current and future research endeavours.

2:00 PM

(ICACC-FS2-009-2024) Superior thermoelectric performance of textured calcium cobaltate ceramics via electrospun nanoribbons

A. Feldhoff*[1]; K. Kruppa[1]; I. Maor[2]; F. Steinbach[1]; M. Mann-Lahav[2]; G. Grader[2]

1. Leibniz University Hannover, Institute of Physical Chemistry and Electrochemistry, Germany
2. Technion - Israel Institute of Technology, Chemical Engineering, Israel

Calcium cobaltate $Ca_3Co_{4-x}O_{9+\delta}$ is a promising p-type thermoelectric material with good functional properties for high-temperature applications in air. Its strong anisotropic properties make texturing and nanostructuring mostly favored to improve thermoelectric performance. Mats of flat-shaped nanofibers, designated nanoribbons, were produced by electrospinning, and they were further processed into textured ceramics. Nanoribbons are considered to contribute to sample texturing via aligned orientation of the primary particles and to provide more efficient packing due to their flatness compared to cylindrical nanofibers, allowing higher densification in the green body and ceramic. Texturing in green bodies and ceramics from nanoribbons mats were investigated using X-ray diffraction pole figures. In addition, microstructure, as obtained by scanning electron microscopy and transmission electron microscopy, was correlated to Seebeck coefficient, electrical conductivity and thermal conductivity of the nanoribbon-based samples to evaluate their thermoelectric abilities, which include power factor and figure of merit zT.

2:20 PM

(ICACC-FS2-010-2024) Thermoelectric Properties of $SrTiO_3$/TiN Nanocomposites Consolidated by Spark Plasma Sintering

M. Ohtaki*[1]; S. Umeno[1]; S. Nagasaki[1]; K. Suekuni[1]

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

Nanostructure engineering is nowadays the most important and promising pathway to higher ZT values. However, in principle, the complex nanostructures are thermodynamically non-equilibrial and hence thermally unstable, tending to transform toward energetically favourable coarser and simpler structures through thermal diffusion and grain growth. In this paper, we have examined influences of metal nitrides as heat-resistant diffusion barriers in oxide matrix. Mixtures of n-doped $SrTiO_3$ (STO) and TiN were sintered by using spark plasma sintering. The electrical conductivity of the samples increased with increasing TiN content, reflecting the metallic characters common to metal nitrides. The thermal conductivity of the samples were always higher for the samples containing TiN than that of STO, showing larger electron thermal conductivity for the samples with TiN as expected. However, most interestingly, the lattice thermal conductivity was lowest for the sample containing the largest proportion of TiN; this result can be attributed to enhanced phonon scattering at oxide/nitride hetero-interfaces in the sample. A highest ZT value among the samples was obtained for the sample containing 20 wt% (nominal) TiN as ZT = 0.14 at 800 °C, being 8 times higher than that of the sample without TiN. This work was supported by JSPS KAKENHI JP22H01779.

2:40 PM

(ICACC-FS2-011-2024) Tuning Thermal Conductivity of $SrTiO_3$ through Ruthenium Doping

S. Akhbarifar*[1]

1. The Catholic University of America, Physics, USA

Doping $SrTiO_3$, a perovskite oxide, to optimize its thermoelectric potential through a delicate balance between electrical and thermal conductivity is of paramount importance. This study investigates the thermal conductivity of $SrTiO_3$ doped with ruthenium (Ru) at the titanium site (B-site substitution). Various compositions, i.e., $SrTi_XRu_{1-X}O_3$ (where $0 \leq x \leq 1$), were synthesized using solid-state synthesis and characterized for composition, structure, phase content, morphology, and crystal size through X-ray fluorescence, X-ray diffraction, and SEM/EDX techniques. Thermal conductivity measurements were conducted over a temperature range from 25°C to 300°C, revealing a remarkable reduction at 25°C, dropping from 5.5 W/mK for $SrTiO_3$ to 1.01 W/mK for $SrTi_{0.5}Ru_{0.5}O_3$. This substantial decrease holds the potential to enhance heat-to-electricity conversion by a factor of five. The thermoelectric data were analyzed using quantum physical models to interpret the underlying scattering mechanisms concerning carrier concentrations.

3:20 PM

(ICACC-FS2-012-2024) Solution-Processed Inorganic Thermoelectric Materials: New avenues for material control (Invited)

M. Ibáñez*[1]

1. Institute of Science and Technology Austria (ISTA), Austria

In recent years, solution processes have gained considerable attention as a cost-effective and scalable method for producing high-performance thermoelectric materials. In this strategy, powder material is prepared in a solution, then purified and processed thermally to render the desired dense polycrystalline material. Different from conventional methods, syntheses in solution can produce particles with unprecedented control over their size, shape, crystal structure, composition, and surface chemistry. Such control over the powder properties provides unique opportunities for the synthesis of thermoelectric materials with deterministic microstructural properties. Herein, we propose the use of well-defined nanoparticles as precursors for the production of dense thermoelectric materials as a means to control the microstructure of the dense solid. We will show different examples in which the control over specific nanoparticle properties, such as surface or stoichiometry, translates into very particular properties of the dense material that allow for optimizing its performance. In particular, we will focus on the case of Ag_2Se, a highly relevant thermoelectric material for its use near room temperature.

3:50 PM

(ICACC-FS2-013-2024) Benchtop room-temperature synthesis of high performance thermoelectrics (Invited)

K. Kovnir*[1]

1. Iowa State University, Chemistry, USA

Se-containing materials often synthesized by solution-assisted from toxic and expensive organo-Se precursors. We are reporting a facile alternative utilizing elemental Se which is cheap and non-toxic. Elemental Se reagent has a drawback of low solubility and reactivity resulting in sluggish kinetics at low temperatures. A process of elemental Se activation was developed resulting in stable solution of multiple Se species confirmed with ^{77}Se NMR studies, including R-Se-Se-R and never detected before “naked” Se_2^{2-}. Dynamic equilibrium between the produced Se species allow to perform reactions at wide temperature ranges including room temperature. The developed activated Se precursor allows us to produce Ag_2Se-based materials at room temperature thus avoiding the high-temperature polymorphic phase transitions during the synthesis. The produced materials exhibit flat, almost temperature-independent zT in the 300-400 K range with average zT’s exceeding 1.

4:20 PM

(ICACC-FS2-014-2024) Long-term stable n-type organic thermoelectrics with improved power factor (Invited)

S. Chang[1]; P. Biswas[1]; Z. Tian*[1]

1. Cornell University, USA

Thermoelectrics (TEs) can directly convert heat to electricity. Organic TE materials have the potential to play a unique role in wearable heating-cooling devices and near-room-temperature energy generation for healthcare applications due to their lightweight, low-cost, flexibility, high-throughput fabrication schemes, and environmental friendliness. Yet organic TEs suffer from much lower energy conversion efficiency than inorganic ones. Despite the significant process in p-type organic thermoelectrics, the development of n-type organic thermoelectric materials is rather limited by their low mobility, inferior air stability, and poor doping efficiency. In this talk, we will share our recent development of micron-thick films based on single-wall carbon nanotube (SWNT), where we applied a surfactant to induce the n-type behavior and doped the film to further increase the electrical conductivity. The films were measured over 3 months and retained a stable negative Seebeck coefficient, which proves as a promising candidate for long-term thermoelectric devices.

4:50 PM

(ICACC-FS2-015-2024) Electronic structure of thermoelectric materials SnSe and $SnSe_2$ (Invited)

S. Mo*[1]

1. Lawrence Berkeley National Laboratory, USA

Maximizing the thermoelectric performance of a material is a complex and challenging problem since the parameters constituting the thermoelectric figure of merit (ZT) are intertwined and even conflicting with each other. Although a simple understanding of the thermoelectric performance from an electronic structure point of view may be hard to achieve, establishing a correlation among electronic structure, external stimuli such as doping and defect, and thermoelectric properties could provide valuable insight for the future development of new materials. We have investigated the electronic structures of thermoelectric layered materials, SnSe and $SeSe_2$, using angle-resolved photoemission spectroscopy and first-principles calculations. We found that multi-valley band structure with converging valence band top is the common feature of these materials. We also show that it is possible to engineer the band structure by doping electrons or holes, or by adding defects such as Sn vacancies, to further improve the thermoelectric performance of these materials.

FS3 Nanostructures and Low-Dimensional Materials for Chemical Sensors

Focused Session 3: Nanostructures and Low-Dimensional Materials for Chemical Sensors

Room: Flagler A
Session Chair: Hyung Gi Byun, Kangwon National University

1:30 PM

(ICACC-FS3-012-2024) Multifaceted MOF-based Catalysts with Broad Applicability (Invited)

S. Kim*[1]

1. Korea University, Republic of Korea

Metal-Organic Frameworks (MOFs) are three-dimensional network structures composed of metal ions and organic linkers, with metal ions/clusters connected to organic molecules as ligands, resulting in a variety of sizes and shapes. With a large internal surface area, MOFs can adsorb gas molecules on their surfaces and have gained significant attention lately due to their versatile structure design, high catalytic activity, and environmental friendliness. Based on these advantages, MOFs are widely utilized in diverse cutting-edge applications, including energy storage, gas separation, sensors, and catalysis. In this study, we will elucidate the optimization of 3D structured MOF-74, Co-MOF, and zeolite-imidazolate framework-8 (ZIF-8) through various metal dopings and composite structures for their application as gas sensors and electrochemical catalysts in hydrogen evolution reaction (HER) and carbon dioxide reduction reaction (CO_2RR). Additionally, we will provide an in-depth explanation of the highly esteemed MOF-derived single-atom catalysts (SACs), which are currently gaining prominence as electrochemical catalysts, and engage in a comprehensive discussion of the sophisticated analytical methods involving corrected high-resolution scanning transmission electron microscopy (Cs-STEM), x-ray absorption spectroscopy (XAS), and density function theory (DFT) used to confirm their characterization.

2:00 PM

(ICACC-FS3-013-2024) Effect of noble metal nanoparticles on the gasochromism of WO_3 sol-gel thin film (Invited)

A. Longato[1]; E. Colusso[1]; M. Vanzan[2]; S. Corni[2]; A. Martucci*[1]

1. University of Padova, Industrial Engineering, Italy
2. University of Padova, Chemistry, Italy

Crystalline tungsten trioxide thin films covered by noble metal (Au, Pt) nanoparticles were synthesized by sol-gel chemistry and used as optical sensors for H_2. The optical gas sensing behaviors are strongly influenced by the crystallinity of the film and the nature of the catalyst used. The best performances were obtained with Pt but also Au coated film sowed remarkable sensing activity which resulted strongly influenced by the dimensions: devices sensitized with smaller nanoparticles display better H_2 sensing performance. Computational insight based on Density Functional Theory calculations suggest that this could be related to processes occurring at the nanoparticle-WO_3 interface, in which gold catalyze heterolytic hydrogen dissociative.

2:30 PM

(ICACC-FS3-015-2024) Biocompatible, wearable, and customizable chemical sensor system for real-time health monitoring (Invited)

T. Kim*[1]; S. NajafiKhoshnoo[2]; J. A. Tavares-Negrete[2]; X. Pei[2]; P. Das[3]; S. Lee[4]; J. Rajendran[2]; R. Esfandyarpour[2]

1. Baylor University, Mechanical Engineering, USA
2. University of California, Irvine, USA
3. Siksha O Anusandhan University Institute of Technical Education and Research, India
4. Terasaki Institute for Biomedical Innovation (TIBI), USA

Monitoring the pH of biofluids holds great significance in healthcare. It serves as a useful tool for infection detection, wound monitoring, and disease diagnosis. However, the existing sensor systems have limitations of being expensive and bulky which is inappropriate for real-time health monitoring. A promising solution lies in the development of a sensor system which is biocompatible, wearable, and flexible, and gives continuous and real-time chemical sensing data of user's biofluids, which will advance the personalized medicine. A flexible, customizable, and low-cost chemical sensor system can be developed by 3D printing nanomaterial-based inks. The beauty of 3D printing lies in its customizable and tailor-made solution at an affordable cost. This chemical sensor system can also be operated with a battery-free readout system that realizes a continuous real-time monitoring through a wireless data transmission. Here a pH sensor system developed in this approach shows high sensitivity, specificity, and reliable sensing performance covering the pH range from 3.0 to 10.0. Moreover, it demonstrates excellent biocompatibility and pH monitoring of a wound model successfully. This wearable pH sensor system represents an integrated platform that enables real-time and customizable chemical sensing for health monitoring. It stands as a pivotal advancement of the personalized medicine.

3:20 PM

(ICACC-FS3-016-2024) Ethylene and Putrid Odor Measurement System based on Gas Sensors Arrays (Invited)

H. Byun*[1]; J. YU[1]

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

Changes in the food distribution environment and the increasing demand for fresh produce by consumers are driving the need for technology to control the distribution environment by monitoring the post-harvest physiological activities of agricultural products. The measurement of ethylene gas generated in the process of physiological activities of plants during the distribution of agricultural products can be used to control the quality of agricultural products, so there is a need for sensors and systems that can monitor it in real time. In addition to ethylene sensor, it is also essential to implement a system consisting of an array of gas sensors that can precisely analyses volatile organic compounds that occur during the spoilage of produce, to make the determination of freshness more realistic. We have been developing an odor measurement system composed chemical sensors arrays for ethylene and other substances happening at ripening process, chamber, data acquisition system with microprocessor, and sampling system. The primary experimental results showed that it was possible to develop odor measurement system for discriminate ethylene and other substances in real time.

FS4 Ceramic/Carbon Reinforced Polymers

Focused Session 4: Characterization

Room: Flagler C
Session Chair: Shinji Ogihara, Tokyo University of Science

1:30 PM

(ICACC-FS4-001-2024) Voids in Type IV high-pressure hydrogen tank of carbon fiber reinforced plastic (Invited)

M. Ueda*[1]; T. Hidaka[1]; H. Yang[1]; N. Ichihara[1]; T. Yokozeki[2]; R. Aoki[2]; T. Matsuda[3]; N. Morita[3]; W. Iwase[4]

1. Nihon University, Japan
2. The University of Tokyo, Japan
3. University of Tsukuba, Japan
4. Yachiyo Industry Co., Ltd., Japan

Design safety factor on a high-pressure hydrogen tank needs to be reduced to realize lightweight and low-cost fuel cell vehicles. The high-pressure hydrogen tank is made of carbon fiber-reinforced plastic by means of filament winding. There were many voids in the high-pressure hydrogen tank, which was considered one of the main causes of the large variability in the strength. Therefore, the voids in the high-pressure hydrogen tanks were studied in this study. Internal observation by X-ray computed tomography and cross-sectional observation by scanning electron microscope were performed. The voids were categorized considering their size, shape, and location. Several types of voids were identified; (a) voids within towpreg, (b) towpreg edge voids, (c) crimp voids, (d) voids due to overlap gap, (e) voids due to towpreg gap. The methods to reduce the voids were also discussed.

2:00 PM

(ICACC-FS4-002-2024) Damage mode analysis of CFRP by peak frequency considering the response function of AE sensor (Invited)

T. Sakai*[1]

1. Saitama University, Japan

Carbon fiber reinforced composites (CFRP) are widely used not only in the aerospace industry but also in the automotive industry because of their high strength and high elasticity. Various tests have been conducted to improve the reliability of CFRP, and it is known that the fracture behavior of CFRP is complex, including matrix cracking, debonding, delamination, and fiber breakage. In order to evaluate these phenomena, Acoustic Emission (AE) method has been applied to many CFRP tests, and most of these tests are based on the assumption that the peak frequency corresponds to the damage mode. In this study, we propose a method to remove the influence of the response function of the AE sensor that affects the peak frequency.

2:30 PM

(ICACC-FS4-003-2024) Micromechanisms of defect induced cracking in cross-ply CFRP laminates (Invited)

S. Oshima*[1]; R. Higuchi[3]; S. Kobayashi[2]

1. Tokyo Metropolitan University, Department of Aeronautics and Astronautics, Japan
2. Tokyo Metropolitan University, Mechanical Engineering, Japan
3. University of Tokyo, Department of Aeronautics and Astronautics, Japan

Initiation of transverse cracks is strongly affected by the presence of defects such as voids and contamination. The effects of shape, size, orientation, and location of voids have been taken into consideration more recently by using advanced inspection techniques. However, difficulties arise in the quantitative evaluation because the shape, size, orientation, and distribution of voids are not precisely controllable by the manufacturing conditions. Against this backdrop, in this study, we aim to quantitatively evaluate the effect of defects on transverse cracking in cross-ply CFRP laminates under static and cyclic tensile loading. A single artificial defect was introduced into the 90°

layer of cross-ply CFRP laminate. In situ observation was carried out to characterize defect induced cracking behavior under both static and cyclic loading. Micromechanisms of cracking behavior are discussed herein on the basis of experimental, numerical, and analytical results.

FS5 High Voltage Materials for Advanced Electrical Applications

Focused Session 5: High Voltage Materials for Advanced High Power Electrical Applications

Room: Ballroom 4
Session Chairs: Muhammad Shafiq, Florida State University; Kristina Vailonis, NASA Glenn Research Center; Sean McDarby, NASA Glenn Research Center

1:30 PM

(ICACC-FS5-009-2024) Development of Lightweight Durable Conductors with Metals and Carbon Inclusions (Invited)

V. Shanov[1]; Q. Fang*[1]; K. Joseph[2]; K. Brittingham[2]; V. Kondapalli[2]; M. Khosravifar[2]; A. Raut[2]; H. Tran[1]; M. Lizcano[3]; D. Santiago[3]; A. S. Almansour[3]; D. Mast[4]

1. University of Cincinnati, Chemical and Environmental Engineering, USA
2. University of Cincinnati, Mechanical and Materials Engineering, USA
3. NASA Glenn Research Center, USA
4. University of Cincinnati, Physics, USA

The substitution of traditional copper power transmission wires for cables with lightweight Cu-carbon nanotube (Cu-CNT) composite fibers is critical for reducing the weight, fuel consumption, and CO_2 emission in automobiles and aircrafts. In this work, we created a lightweight Cu-CNT composite fiber through a multistep scalable process including spinning, densification, functionalization, and double-layer Cu deposition. The characterization and testing of the fabricated fiber included surface morphology, electrical conductivity, mechanical strength, crystallinity), and ampacity (current density). The electrical conductivity of the resultant composite fiber was measured to be $0.5*10^6$ S/m with an ampacity of $0.18*10^5$ A/cm^2. The Cu coated CNT fibers were 16 times lighter and 2.7 times stronger than copper wire. They revealed a gravimetric density of 0.4 g/cm^3 and a mechanical strength of 0.68 GPa, suggesting great potential in future applications as lightweight power transmission cables. Further, we introduced silver nanoparticles into the CNT fiber during the spinning phase via a mist infiltration method utilizing an ethanol and silver nanoparticle solution which allowed achieving a uniform silver distribution within the CNT fiber. This inclusion of silver enhanced the CNT fiber electrical conductivity.

2:00 PM

(ICACC-FS5-010-2024) Research and innovation to shape the future of power transmission (Invited)

M. Gandini*[1]; S. Siripurapu[1]

1. Prysmian Group, Italy

In an era of unprecedented technological advances and growing energy demands, the field of electrical transmission is at a critical inflection point. This presentation will explore the critical importance of research and innovation in shaping the future of electrical transmission systems from a manufacturer's perspective. Through an exploration of real-world case studies and emerging trends, we will shed light on how research-driven innovation has the potential to revolutionize the way electricity is transported and distributed. From addressing the challenges of integrating renewable energy sources into the grid to improving the efficiency, reliability, and resilience of transmission networks, the role of research, development, and industrialization is paramount. We will review the major advances of the past 15 years and shed light on the key transformations the industry is pursuing. Taking a closer look at the high-voltage underground and submarine cable sectors, we will delve into the new technology platforms that are able to combine the best electrical performance to date with a reduced environmental impact. By fostering collaboration between academia and industry, we can create an ecosystem that not only drives technological breakthroughs, but also enables seamless adoption and implementation.

2:30 PM

(ICACC-FS5-011-2024) Fabrication of copper/carbon nanotube (Cu/CNT) yarn composite conductor

A. S. Almansour*[1]; M. Lizcano[1]; D. Santiago[1]

1. NASA Glenn Research Center, USA

Sustainable electrified aircraft propulsion (EAP) is likely to lead to an increase in the electrical wiring contained within a single aircraft. Since the electrical resistance and mass of copper (Cu) conductors are associated with power losses, it is desirable to design high-conductivity lightweight conductor materials, to reduce the mass of components like motor windings, low-voltage signal cables, and transmission cables for data and power to improve the overall energy efficiency. This work describes a unique framework for manufacturing metalized carbon nanotube (CNT) composite conductors, measuring their electrical conductivity and strength, and modeling the overall conductivity and current sharing within such composites. Tensile testing was conducted on the processed composite conductor cables with the use of acoustic emission and electrical resistivity to determine stress-dependent-failure mechanisms while monitoring the electrical conductivity. The average of measured electrical conductivities of annealed Cu/CNT samples from batch 5 was greater than theoretical predictions by 9.8 percent and was also greater than the conductivity of pure annealed Cu by 4.8 percent and had comparable ultimate tensile strengths. Additionally, those Cu/CNT samples provide a 13.5% weight saving over current state of the art copper wires.

2:50 PM

(ICACC-FS5-012-2024) Creation and Characterization of Multilayer Graphene – Copper Wires

R. A. Paddock*[2]; M. Tehrani[1]; M. Cullinan[2]

1. University of California San Diego, Structural and Materials Engineering, USA
2. The University of Texas at Austin, Walker Department of Mechanical Engineering, USA

Copper's electrical and thermal properties are why this material is utilized for a variety of products, but the mechanical properties make copper not ideal for high-voltage applications. The addition of multilayer graphene (MLG) using a chemical vapor deposition (CVD) on pure copper foils potentially improves the quality of this material. Controlling the pressure range during the CVD process on the 25 and 50 μm thick copper samples allows for high-quality MLG growth across the 4 by 4-inch foils. Films were consolidated and drawn using a roller wire drawing approach along with repeated heat treatments at 600 °C with flowing hydrogen gas between each cross-sectional area reduction. This research highlights the electrical and mechanical properties of pure copper and MLG-copper wire samples. These properties are correlated to the material's structure measured with microscopy and spectroscopy. Based on the data collected from these measurements and a detailed explanation of how to create wires out of these foils, a description of the overall effects of MLG is given along with recommendations on graphene growth and wire formation.

3:30 PM

(ICACC-FS5-013-2024) A Study in the Effect of High-Graphene-Content Loading on Copper Composite Conductors

Y. Bekele*[1]; M. Cullinan[1]; M. Tehrani[2]

1. University of Texas, Walker Department of Mechanical Engineering, The University of Texas at Austin, Austin, Texas, USA
2. University of California, San Diego, Structural and Materials Engineering, USA

Graphene possesses a high electron mobility, while copper exhibits a high free electron density. Leveraging these material properties offers the potential to create a more efficient conductor compared to conventional copper (Cu) conductors. This study investigates a composite with high graphene (Gr) content, referred to as copper-graphene (Cu-Gr), as a promising alternative to traditional Cu conductors. The Cu-Gr conductor is synthesized by depositing bi-layer graphene onto sub-micron Cu films, resulting in alternating Cu-Gr layers to capitalize on their distinct material characteristics. Transferring high-quality graphene with good coverage onto copper films without oxidation, residue, and de-wetting poses challenges, lowering composite conductivity. The wet transfer process, known for producing high-quality graphene, introduces oxidation or residue concerns on the copper film, while the dry transfer process falls short in providing sufficient coverage. Annealing Cu films to reach bulk conductivity of copper has been a challenge for sub-micron thick Cu films due to the issue of de-wetting and triple-point formations at higher temperatures. However, it has been determined that annealing temperature impacts Cu film conductivity more than annealing time. To address these issues, we explore alternative graphene transfer techniques and a conductivity-temperature study for sub-micron Cu films.

3:50 PM

(ICACC-FS5-014-2024) Boron Nitride Materials Development for High Voltage Power Transmission (Invited)

D. Santiago*[1]; M. Lizcano[1]

1. NASA Glenn Research Center, USA

The unique multifunctional properties of boron nitride (BN) nanomaterials are identified as a parameter that would revolutionize electric propulsion in Aeronautics. Having BN as a part of ceramic or polymer composites can improve the matrix properties by simultaneously achieving high thermal conductivity and low electrical conductivity, along with high mechanical strength, low weight, and chemical inertness. These materials properties are essential for components used in high voltage electric system of electric or hybrid electric airplanes. However, synthesis and processing of these BN-modified composites has proven to be difficult because the chemical inertness and thermal stability of BN prevent filler matrix bonding. Over the years, the evolution of our research has demonstrated significant improvements in composite processing while using different boron nitride materials, by using intercalation, exfoliation, coating and/or chemical functionalization of hexagonal boron nitride or boron nitride nanotubes. Despite the team accomplishments, there are still many areas for improvement that remain in development of composite materials with properties tailored to electrical insulation for high voltage applications of electrified aircrafts. This presentation is an overview of the BN materials developed at NASA Glenn Research Center and their different properties for potential electrical insulation applications.

4:20 PM

(ICACC-FS5-005-2024) Thermal analysis of diamond-like carbon incorporated power substrates

A. Juberi*[1]; O. Faruqe[1]; P. C. Saha[2]; C. Park[2]

1. University of Wisconsin Milwaukee, Electrical Engineering, USA
2. University of Wisconsin-Milwaukee, USA

Power electronics modules are integral components in numerous electronic devices and systems, facilitating the efficient conversion and control of electrical energy. However, as the demand for higher power densities and improved performance continues to grow, so does the challenge of managing the heat generated within these modules. This study explores the significance of heat management challenges in power electronics modules and presents diamond-like carbon (DLC) coating as a promising solution to address these thermal concerns. Diamond-like carbon, also known as DLC, is an amorphous hydrogenated or non-hydronated form of carbon with attractive electrical properties and chemical inertness, among other things. It has very high resistivity and high thermal conductivity and can be used effectively as coating on ceramic substrate or as an alternative to ceramic substrate. This study conducted a comparative thermal analysis by using a basic power model with DLC-coated ceramic and replacing the ceramic with DLC. The finite element analysis (FEA) results show that applying DLC thin films on a ceramic substrate or replacing the ceramic substrate with DLC improves the thermal properties of the power module significantly.

4:40 PM

(ICACC-FS5-016-2024) Highly Engineered Inorganic Coating to Unlock Overhead High Voltage Transmission Capacity and Reduce Carbon Emission

S. Ranganathan*[1]; T. Ochmann[1]; V. Garcia[1]

1. Prysmian Group, R&D, USA

The aging transmission electrical grid in the United States is progressively being challenged with power demands that are impeding the reliability of the system. Each transmission line has a defined maximum thermal operating limit it can safely operate to. The cost to upgrade these lines is significant and the financial impact on the economy is growing as energy rates increase due to power line congestion. Also, it may take several years to upgrade the transmission power grids. An innovative inorganic coating has been developed with the combined property of high emissivity and low solar absorptivity which helps to radiate the heat of conductor and thus reduces the energy losses/carbon emission, which can result in 15-25% increased capacity on an existing transmission line that is thermally limited (rated). Additionally, it also helps to avoid sag violation and power grid energy congestion issues. Durability is tested to withstand transmission line dynamic current cycles and exterior weather conditions like rain, snow, and air pollution. This water-based coating can be cured either by heat or ambient conditions, hence it is designed to be applied in manufacturing or field conditions.

S10 Modeling and Design of Ceramics and Composites

SYMPOSIUM 10: Modeling and design of ceramics and composites

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

1:30 PM

(ICACC-S10-015-2024) Interfacial Superstructure of Segregation in Tungsten Carbide (Invited)

C. Hu*[1]; Z. Yu[3]; J. Luo[2]

1. University of Alabama, Aerospace Engineering and Mechanics, USA
2. University of California, San Diego, USA
3. Fuzhou University, College of Chemistry, China

Interfacial segregation of solute or impurity atoms is a critical thermodynamic process widely observed in ceramics and their associated composites. These segregated atoms tend to arrange themselves into highly ordered structures or patterns, often known as segregation superstructures, at grain boundaries or phase boundaries. Since superstructures have a profound impact on a broad range of materials properties and processing, understanding the formation mechanism of segregation superstructure is of great interest to the materials science community. By employing advanced electron microscopy techniques and computational modeling methods, we investigate the nanoscale mechanisms of solute segregation at WC interfaces and explore how solute segregation influences the stability and associated properties of these interfaces. More specifically, three main topics will be discussed in this talk: (1) a highly asymmetric segregation superstructure in WC general grain boundary, (2) interfacial superstructures in WC-Co interface and their roles in chemical bonding transitions, and (3) segregation-induced interfacial twinning in WC. These findings not only advance our fundamental understandings of segregation behavior at WC interfaces, but also provide valuable insights into the precise control of atomic-level interfacial structures for advanced ceramic materials.

2:00 PM

(ICACC-S10-016-2024) From Computing Grain Boundary Diagrams to Controlling Grain Boundary Transitions with Applied Electric Fields (Invited)

J. Luo*[1]

1. University of California, San Diego, USA

I will first review a series of our studies to compute the grain boundary (GB) "phase" (a.k.a. complexion) diagrams [that were recently reviewed in a perspective article: Interdisciplinary Materials 2:137-160 (2023)]. GB lambda diagrams were constructed to forecast high-temperature GB disordering and related trends in sintering and other properties. In parallel, a lattice model was utilized to construct GB adsorption diagrams. Subsequently, atomistic simulations were used to compute more rigorous and accurate GB diagrams of not only thermodynamic and structural characters but also mechanical properties. To further extend prediction power, machine learning was combined with atomistic simulations to predict GB properties as functions of five GB macroscopic (crystallographic) degrees of freedom plus temperature and composition for a binary alloy in a 7-D space or as functions of four independent compositional variables and temperature in a 5-D space for a given GB in high-entropy alloys. In addition, we have demonstrated that applied electric fields can electrochemically induce GB phase-like transitions to alter microstructural evolution, thereby opening yet another new window to control GBs and tailor microstructures.

2:30 PM

(ICACC-S10-017-2024) Computer-aided analyses for structural reliabilities of ceramic materials (Invited)

T. Ohji*[1]; M. Fukushima[1]; K. Hirao[1]; Y. Nakashima[1]; K. Aoki[2]; S. Ozaki[3]; W. Nakao[3]

1. National Institute of Advanced Industrial Science and Technology (AIST), Japan
2. Chukyo University, Japan
3. Yokohama National University, Japan

Determination of successful manufacturing process of ceramic products still largely relies on human being's experience, skills, etc. In order to sophisticate it beyond this craftsmanship, a national R&D project of Japan, "Development of a Technology Base and Applied Technologies for the Manufacturing Processes of Next-Generation Advanced Ceramics" has been initiated by NEDO. This project focuses on the following four research items. (1) Visualizing the whole manufacturing process and analyzing the microstructural evolution mechanism; (2) Developing the computational tools to comprehensively deal with various phenomena in the manufacturing process; (3) Exploring the innovative manufacturing processes for high performance, product miniaturization, structural complexity, low energy consumption, etc.; and (4) Developing the analytical techniques for ensuring the structural reliabilities, including high-precision flaw characterization, machine-learning-based failure prediction, data-driven evaluation of fracture mechanism, simulation-supported accelerated aging tests, etc. This presentation first gives an overview of this project and then describes the latest achievements, particularly on the fourth research item. This presentation is based on results obtained from a project, JPNP22005, commissioned by the New Energy and Industrial Technology Development Organization (NEDO).

3:20 PM

(ICACC-S10-018-2024) Modelisation and simulation of the dynamic behavior of alumina ceramics

J. Meynard*[1]; A. Cosculluela[1]; P. Pradel[1]

1. CEA, France

Modeling the mechanical behavior of ceramic materials used in shielding solutions when subjected to different mechanical loadings is of interest in order to estimate their energy mitigation ability. When subjected to impact loading, the mitigation ability of ceramics is directly related to their dynamic multi-fragmentation behavior. Modeling and predicting the impact response of ceramics is thus crucial for the design of protective systems. To do that, the Denoual-Forquin-Hild (DFH) anisotropic tensile damage model was used. SHPB tests and plate impact experiments were performed on several materials (alumina ceramics with a wide range of porosity and fiber contents) on a wide a range of strain rates, from 10^2 up to 10^6 s^{-1}. The accuracy of the DFH model was tested by comparing numerical and experimental results. Based on this study, it was observed that for tested strain rates, the tensile damage of studied materials is well reproduced, which is not the case of the plasticity behavior. Modifications of the model were implemented to better fit experimental results and to ensure mesh convergence. Finally, the influence of porosity and fibers on the dynamic response of alumina ceramics was discussed by expressing relationships between these microstructural parameters and some DFH input parameters.

3:40 PM

(ICACC-S10-019-2024) Numerical evaluation of relative density-compression strength relation in brittle foams with 3D microstructures

V. Deshpande*[1]; R. Piat[1]

1. University of Applied Sciences, Darmstadt, Mathematics and Natural Sciences, Germany

Experimentally determining the effect of relative density on compression strength of brittle foams is a costly process of manufacturing and testing statistically significant number of samples of different densities. It also does not reveal the failure mechanisms. This work describes an alternate numerical method of microstructure reconstruction, FE simulation and statistical study. A novel reconstruction algorithm creates artificial microstructures statistically equivalent to the real ones obtained from micro-CT. This follows comparing the simulated effective compression stress-strain behaviour of reconstructed samples with that of the real ones along with experimental data. The effect of length scale on compression strength is determined by simulating the reconstructed samples of different sizes. Further, samples of different densities are reconstructed and their compression stress-strain curves calculated to relate density with compression strength. The failure modes are determined by analysing the evolution of failure site locations. The resulting density-strength relation agrees with the Gibson-Ashby model. Two failure modes: cellular failure below 0.4 relative density and brittle failure above, agree with the available experimental results. The evolution of failure site locations reveals the mechanisms behind the failure modes not accessible through experiments.

4:00 PM

(ICACC-S10-020-2024) Machine Learning Interatomic Potentials (MLIPs) for Preceramic Precursors and Polymer-Derived Ceramics

M. Falgoust*[1]; P. Kroll[1]

1. University of Texas, Arlington, USA

Preceramic polymers (PCPs) such as polycarbosilanes, polysiloxanes, and polysilazanes are the feedstock to produce polymer-derived ceramics (PDCs). In this contribution, we present Machine Learning Interatomic Potentials (MLIPs) for various preceramic precursors in the Si–C–N–O–H system. The MLIPs are fitted to energies, forces, and stresses from previous DFT calculations using the large database of structure models and extended ab initio molecular dynamics (aiMD) trajectories we generated over the last decades. We show that the MLIPs we develop for PCPs and PDCs are as accurate as DFT calculations while computationally far more efficient. Moreover, hitherto non-explored configurations can be added on the fly, allowing systematic improvement of MLIPs through active learning strategies. We demonstrate how the MLIPs are integrated into large-scale molecular dynamics simulations as implemented in the LAMMPS code. Through LAMMPS we can access a variety of static and dynamic properties of PCPs and PDCs at the short and medium-range length scale reaching micro-second times scales.

4:20 PM

(ICACC-S10-021-2024) Ab-initio Molecular Dynamic Simulations of the Pyrolysis of Pre-Ceramic Polymers

P. Kroll*[1]

1. University of Texas, Arlington, USA

Thermal conversion of polysilanes, polycarbosilanes, polysilazanes, and polysiloxanes yields high-performance ceramics such as SiC, Si_3N_4, and SiCO with tailored compositions. The early stages of the polymer-to-ceramic route are characterized by a few fundamental reactions related to cross-linking, insertion, and elimination – and their reaction mechanisms are difficult to decipher. We show that atomistic details of various fundamental reactions are obtained from quantum-chemical (ab-initio) Molecular Dynamic Simulations of silicon-based polymers. We observe intra-chain and inter-chain coupling, cross-linking, and elimination reactions. We discern the role of hydrogen in facilitating the rapid exchange of bonds. We analyze Kumada-type reactions and the Yajima-process that yield the incorporation of carbon into the polymer backbone. Some outcomes are as expected, while others differ from common views. Overall, valuable insight into reaction paths and products can be obtained from these ab-initio Molecular Dynamic Simulations.

S12 Design and Applications of Nanolaminated Ternary Transition Metal Carbides/Nitrides and Borides, Their solid solutions and 2D Counterparts

SYMPOSIUM 12: Design and Applications of Nanolaminated Ternary Transition Metal Carbides/Nitrides (MAX Phases) and Borides (MAB Phases), their Solid Solutions and 2D Counterparts (MXenes, MBenes)

Room: Ballroom 3
Session Chairs: Surojit Gupta, University of North Dakota; Ankit Srivastava, Texas A&M University; Deniz Cakir, University of North Dakota

1:30 PM

(ICACC-S12-014-2024) Instability-induced deformation in MAX phases (Invited)

M. Dujovic[1]; M. Chouksey[1]; M. Radovic[1]; A. Srivastava*[1]

1. Texas A&M University, USA

MAX phases are atomically layered ternary carbides and nitrides. They are lightweight, elastically stiff, thermodynamically stable, refractory, damage-tolerant, pseudo-ductile, and machinable. The key to their properties lies in their atomically layered hexagonal crystal structure with strong intralayer and weak interlayer atomic bonds. Several recent studies have shown that the grain-level deformation and failure mechanisms of MAX phases depend on both the crystallographic orientation and deformation constraint of the grains. Expanding on these studies, we conducted unconventional small-scale tests to further analyze the influence of deformation constraints on their mechanical response. Our tests involved free-standing micropillars, unconstrained microwalls, and constrained microwalls. Our results show that depending on the level of constraint, the material either easily cleaves or undergoes kink-band instability. We will present the results of these tests and crystal plasticity finite element simulations, focusing on correlating the effects of imposed deformation constraints and the onset of instability-induced deformation.

2:00 PM

(ICACC-S12-015-2024) Microstructural defects in MAX phases

A. Guitton*[1]; A. Heinzelmeier[1]; T. Weidner[2]; T. Grosdidier[1]; A. Mussi[2]; J. Guénolé[1]; V. Taupin[1]

1. Université de Lorraine – CNRS – Arts et Métiers ParisTech – LEM3, France
2. Université de Lille, CNRS, INRAE, Centrale Lille, UMET, France

The control of complex micro- and nano-structures is one of the most promising strategies to design materials with tailored properties. $M_{n+1} AX_n$ phases, where n ranges from 1 to 3, are a group of materials composed of a transition metal M, an A-group element A, and nitrogen or carbon X. These materials exhibit a unique combination of properties derived from both ceramics (refractory, high stiffness, low density, low ductility at room temperature) and metals (high thermal and electrical conductivity, resistance to thermal shocks, low hardness, and mechanical strength), making them highly attractive for various potential applications. Additionally, we demonstrated that composites of MAX phases facilitate the creation of complex microstructures capable of enduring severe plastic deformation [1,2]. In this context, recent progress in comprehending micro-/nano-structural defects and their connection to deformation [1,2], diffusion [3], and oxidation [4] mechanisms is elaborated upon.

2:20 PM

(ICACC-S12-016-2024) First-principles study on the oxidation of MoAlB and Cr_2AlB_2 (Invited)

B. Samanta[1]; S. Omotayo[1]; D. Cakir*[1]

1. University of North Dakota, Physics and Astrophysics, USA

MAB phases exhibit excellent resistance to oxidation, attributed to the formation of a protective Al_2O_3 scale on their surfaces. Therefore, it is crucial to study the oxidation behavior of MAB compounds computationally in order to gain a deeper understanding of their oxidation mechanisms and further optimize their performance in high-temperature applications. The initial stage of the oxidation process of the lowest-energy MoAlB and Cr_2AlB_2 surfaces was investigated at different temperatures and oxygen concentrations by utilizing first-principles calculations. Surface energies were calculated for the low-index surfaces. We found that different surfaces exhibited distinct defect energetics, leading to different oxidation mechanisms and products. Diffusion barriers were determined for Al vacancies and O atoms, showing that Al vacancies exhibit lower diffusion compared to the O atoms and play an important role in the oxidation process in MoAlB. However, in Cr_2AlB_2, Al vacancy exhibits sluggish kinetics. We calculated the Gibbs free energy of various reactions to uncover possible oxidation reactions for MoAlB and Cr_2AlB_2. We conducted molecular dynamics simulations at various temperatures and O_2 concentrations to further shed light on the oxidation process. Our study provides valuable insight into the stability, structural properties, and oxidation behavior of MoAlB and Cr_2AlB_2 surfaces at the atomic level.

2:50 PM

(ICACC-S12-017-2024) Anomalous crack growth resistance in atomically layered ternary carbides

M. Dujovic*[1]; S. Celik[1]; M. Radovic[1]; A. Srivastava[1]

1. Texas A&M University, USA

The presence of a notch in a material under mode-I loading conditions acts as a stress concentrator and increases the likelihood of crack initiation and propagation, resulting in premature fracture. However, this work demonstrates that the effect of a notch in an atomically layered ternary carbide with a hexagonal crystal structure, characterized by strong intralayer and weak interlayer atomic bonds, is not always intuitive. To this end, we conducted mechanical tests using notched microcantilever beams, with the basal planes oriented parallel, perpendicular, or at an angle close to 45 degrees relative to the notch. Our results reveal that despite the imposed loading promoting mode-I notch opening in all tests, the initiation and propagation of cracks depend on the crystallographic orientation. We will present the results of these tests and crystal plasticity finite element simulations, focusing on correlating the effects of crystallographic orientation and crack growth response of these materials.

3:30 PM

(ICACC-S12-018-2024) Alumina scale buckling during high temperature oxidation of Cr_2AlC MAX Phase

S. Dubois*[1]; A. Zuber[2]; C. Coupeau[1]; V. Gauthier[1]; G. Parry[3]

1. PPRIME Institute, France
2. Institut PPRIME, Physics and Mechanics of Materials, France
3. Grenoble INP, SIMAP, France

Single-crystal and fine-grained polycrystalline samples of Cr_2AlC were oxidized under dry air flow at temperature in the 1000-1400°C range during 100 h. A continuous alumina layer forms on top of the Cr_2AlC surface whereas a Cr_7C_3 sublayer also appears. In-lab characterization of oxidized Cr_2AlC samples shows strong damaging at the free surface, resulting from the buckling of the alumina scale. In-situ X-ray diffraction measurements under synchrotron radiations were performed to measure the lattice strain during the first hours of oxidation process and further calculate the internal stress in the Al_2O_3 layers. Alumina layers undergo tensile stress during isothermal oxidation, showing that the buckling of the alumina scale does not result from the oxide growth. Such a tensile stress likely results from the Cr_2AlC to Cr_7C_3 phase transformation. During cooling, the tensile stress decreases down to compressive values, due to the thermal expansion coefficient mismatch between the film and the substrate, leading to buckling of the alumina layer. It is demonstrated that the dimensions of the buckles cannot be explained either by gas pressure or by the magnitude of the internal compressive stress in the alumina scale. The discrepancy between the experimental maximum deflection and the one predicted by the elastic theory can only be explained by a significant plastic deformation in the alumina scale.

3:50 PM

(ICACC-S12-020-2024) Synthesis and Tribological Study of PEEK and PEEK-based Composites

M. Malusky*[1]; M. Almarzoogi[1]; S. Gupta[1]

1. University of North Dakota, Mechanical Engineering, USA

A multi-variable tribological study on Poly Ether Ether Ketone (PEEK) and particle-reinforced PEEK-matrix composites is conducted by using different types of lubricants. Additionally, these composites are explored in high-temperature tribological studies to understand how the thermal environment affects the wear characteristics of materials. The reinforcement additives in the PEEK-matrix composites investigated are Cr2AlC and MoAlB, known as MAX and MAB phase ceramics, respectively. The study quantifies the tribological performance of these materials to explore unique applications where sustainable practices can be implemented using ethanol as a green lubricant, and PEEK as a recyclable polymer. As a background, PEEK is a semi-crystalline thermoplastic polymer with high strength, thermal stability, chemical resistance, and wear resistance. The high-performance properties of PEEK are not typically associated with thermoplastics. Performance-demanding applications usually implement thermosets which are not easily recyclable, whereas thermoplastics are. Additionally, investigating the impact MAB and MAX phase ceramic reinforcements have on improving wear performance can lead to insight for other applications utilizing PEEK.

4:10 PM

(ICACC-S12-021-2024) Magnetocaloric properties of bulk Fe_2AlB_2 synthesized by reactive hot isostatic pressing

H. R. Da Igreja[1]; S. Tencé[2]; P. Chartier[1]; S. Dubois*[1]

1. PPRIME Institute, France
2. Institut de Chimie de la Matière Condensée de Bordeaux, France

The nano-laminated transition metal boride Fe_2AlB_2 have gained considerable interest in recent years due to its magnetocaloric properties. In this study, we report the synthesis of Fe_2AlB_2 at 1150°C for 4 h employing the hot isostatic pressing technique. The phase composition and resulting microstructure are characterized. It mainly consists in Fe_2AlB_2 with a few impurities only detected by scanning electron microscopy and energy dispersive X-ray spectroscopy. The fully dense polycrystalline sample exhibits equiaxial grains (with an average size of about 32 μm), with impurities located in the grain boundaries in addition to unreacted B particles. Investigations on the magnetocaloric properties is undertaken via both direct (magnetization curves) and indirect methods (specific heat). The results show an isothermal entropy change of 2.8 $J.kg^{-1}.K^{-1}$ at 2 T and 6.5 $J.kg^{-1}.K^{-1}$ at 5 T. Regarding on the magnetocaloric performance, it is pertinent to point that the composition of Fe_2AlB_2 comprises both light and earth-abundant elements. Additionally, the utilization of the hot isostatic pressing sintering technique facilitates the single-step synthesis process, thereby shaping the material into its final product form. Taken together, these attributes position Fe_2AlB_2 as a highly attractive option for magnetic refrigeration applications.

4:30 PM

(ICACC-S12-022-2024) Design Paradigm for Manufacturing MAX and MAB Phases

G. Ngige*[1]; M. Dey[1]; S. Gupta[1]

1. University of North Dakota, Mechanical Engineering, USA

MAX and MAB phases have emerged as potential materials for numerous applications. Novel manufacturing processes are needed to further evaluate these materials. In this presentation, we present a few of the recent developments in manufacturing of MAX and MAB Phases. Some of the potential methods which will be reviewed are salt encapsulation processes, use of biomass for manufacturing MAX phases and a design paradigm for rapid synthesis of MAB phases, amongst others. It is expected that these new manufacturing processes will further accelerate the economical and large-scale manufacturing of these novel ceramics.

4:50 PM

(ICACC-S12-023-2024) Synthesis Processes of Superconducting (?) MAX phase Ti_2InN

T. Prikhna*[1]; M. Eisterer[2]; A. Bodenseher[2]; B. Büchner[3]; O. Kvitnitskaya[4]; R. Kluge[3]; R. He[3]; L. Kielak[2]; M. Karpets[5]; V. Moshchil[1]; S. Gass[3]; O. Borymskyi[1]; D. Efremov[3]; T. Puig[6]; X. Obradors[6]

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2. Atominstitut, TU Wien, Austria
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4. B.Verkin Institute for Low Temperature Physics & Engineering of the National Academy of Sciences of Ukraine, Ukraine
5. National Technical University of Ukraine «Igor Sikorsky Kyiv Polytechnic Institute", Ukraine
6. Institut de Ciencia de Materials de Barcelona, CSIC, Universitat Autònoma de Barcelona, Spain

Superconductivity (SC) in a nitride of the MAX-phase family was first reported by A.D. Bortolozo et al. in Ti_2InN (a = 0.3074 nm, c = 1.3975 nm) with a transition temperature of 7.3 K. Bulk Ti_2InN MAX phase-based samples with a=0.3075-0.3077, c=1.4012-1.4021 nm were prepared in the present study from Ti_2InN precursor powder (93-95% purity): (1) at 130 bar of N_2 (2) in a quartz ampoule in Ar, (3) by spark plasma sintering, (4) by high pressure (4 GPa) - high temperature sintering. And. All the manufactured samples demonstrated superconducting behavior with T_c onset near 5 K. The samples prepared by SPS and HP-HT methods were highly dense. However, all samples showed a very broad magnetic transition (as susceptibility) not saturating down to 2 K. No macroscopic Meissner phase was established. The magnetization was far too weak to evidence bulk superconductivity of the entire sample and hence of Ti_2InN. The signal may stem either from a minority phase, or from SC surface. In some places of HP-HT sintered samples, a superconducting gap of about 1.2-2.1 mV was estimated from point-contact spectroscopy. Crystalline admixture grains of TiN phase in Ti_2InN matrices are one possible candidate for the superconducting phase in our materials. Alternatively, the small magnetic signal could result from a strong dependence of superconductivity of Ti_2InN on the N stoichiometry or O impurities, which requires additional studies.

S14 Crystalline Materials for Electrical Optical and Medical Applications

SYMPOSIUM 14: Phoosphor, Laser, Isolator, NLO materials

Room: Coquina H
Session Chairs: Luiz Jacobsohn, Clemson University; Takayuki Yanagida, Nara Institute of Science and Technology

1:30 PM

(ICACC-S14-009-2024) Low temperature synthesis of inorganic materials using novel water-assisted solid-state reaction method (Invited)

K. Toda*[1]

1. Niigata University, Japan

Nano-sized inorganic materials were synthesized using a novel water-assisted solid-state reaction (WASSR) method. This novel soft chemical synthesis method is very simple and can synthesize nanoparticle materials just by storing or mixing raw materials added a small amount (typically 10wt%) of water in a reactor at low temperature below 500 K. Typical particle sizes (under 100 nm) of the samples prepared by the WASSR method were smaller than that (2 - 10 micrometer size) of the sample prepared by the conventional solid-state reaction method. We can synthesize nano-sized practical materials such as $LiCoO_2$, $SrMoO_4$, $BiVO_4$, YVO_4, Ba_2SiO_4:Eu, $CsPbCl_3$ and $BaTiO_3$ by the WASSR method.

2:00 PM

(ICACC-S14-010-2024) Effect of metallic chains of nanoparticles on the photoluminescence of MoS_2 monolayer (Invited)

J. Fernandez Martinez[1]; D. Hernández-Pinilla[1]; D. Gallego[2]; H. Van der Meulen[1]; G. Lopez-Polin[1]; P. Ares[2]; J. Gomez-Herrero[2]; M. Ramirez[1]; L. E. Bausa*[1]

1. Universidad Autonoma de Madrid, Fisica de Materiales, Spain
2. Universidad Autonoma Madrid, Física de la Materia Condensada, Spain

Among low dimensional materials, monolayer (1L) Transition Metal Dichalcogenides (TMDs) are promising candidates for the development of next-generation optoelectronics due to their direct optical gap and atomic thinness. In addition, TMD properties can be readily tuned employing strain or electrostatic gating, to name some examples. Here, we analyse the photoluminescent properties of 1L MoS_2 deposited on top of metallic chains of closely spaced Ag nanoparticles (NPs) formed on the domain wall surfaces of a ferroelectric crystal. These NP chains support a spectrally broad plasmonic resonance centered at around 600 nm, overlapping the A and B excitonic transitions of MoS_2. Scanning photoluminescence (PL) microscopy is employed to elucidate the effect of plasmonic chains on the MoS_2 emission. The results show that the PL of 1L MoS_2 is altered in the vicinity of the metallic chains, causing a redshift of the A exciton along with a modification of the relative contribution of the excitons and trions quasiparticles to the PL spectrum. The influence of both strain gradient and electron transfer are discussed to account for the observations. The results show the potential of integrated plasmonic-ferroelectric-2D platforms for the control of strain driven quasiparticle confinement phenomena, opening a pathway for quantum emitters in 2D strained systems.

2:30 PM

(ICACC-S14-011-2024) Laser optical elements fabricated by pulsed electric current sintering (PECS) (Invited)

H. Furuse*[1]

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

Pulsed electric current sintering (PECS) is one of the fascinating sintering technique for fabricating novel laser optics owing to its unique sintering mechanism. Particularly, PECS can sinter the

materials with relatively low sintering temperature under vacuum with uniaxial pressing, results in suppression of grain growth of the ceramics and realization of transparent ceramics with fine microstructure. For laser optics, we have fabricated some novel optical elements including non-cubic laser materials or sapphire/YAG ceramic composite materials by using PECS. In the presentation, the detail of materials preparation, results of microstructure, optical characteristics, and lasing properties concerning to these materials, and the future possibility of PECS for fabricating effective laser optics will be discussed.

3:20 PM

(ICACC-S14-012-2024) DUV optical isolator materials (Invited)

R. Yasuhara*[1]

1. National Institute for Fusion Science, Japan

For silicon devices with high throughput, increasing the output power of deep-ultraviolet laser light sources for exposure light is necessary. In this study, we report on the study of optical isolators for deep-ultraviolet lasers, which are essential for higher output power of laser light sources development, mainly from the viewpoint of materials research. Primarily at 193 nm, the laser output of Ar Excimer used in semiconductor manufacturing equipment, attention must be paid to maintaining long-term optical properties in addition to the initial transmission characteristics. We have successfully developed an optical Faraday isolator for deep-ultraviolet lasers using rare earth-free materials and the rare-earth-based active element-based magnet-optic materials used for the near-infrared and visible regions. In the presentation, these details and future developments will be discussed.

3:50 PM

(ICACC-S14-013-2024) Subwavelength spatial confinement of a self-Q-switched solid-state laser (Invited)

M. Ramirez*[1]; P. Molina[1]; D. Hernández-Pinilla[1]; G. Lopez-Polin[1]; F. Leardini[1]; M. Chhowalla[3]; P. Ares[2]; J. Gomez-Herrero[2]; L. E. Bausa[1]

1. Universidad Autonoma de Madrid, Fisica de Materiales, Spain
2. Universidad Autonoma de Madrid, Fisica de la Materia Condensada, Spain
3. University of Cambridge, Materials Science & Metallurgy, United Kingdom

2D materials, rare earth emitters, plasmonics and lithium niobate (LN) are relevant players in the current advancement of optoelectronics and integrated photonics. In this work, we present a novel monolithic architecture that integrates these technologically relevant systems in a single platform to demonstrate a new type of integrated laser light source: a self-Q-switching plasmon-assisted nanolasing. Our system combines: i) a Nd^{3+} doped LN ferroelectric crystal, which provides optical gain in the near infrared spectral region; ii) plasmonic chains of Ag nanoparticles that enable subwavelength spatial confinement of the laser radiation; and iii) a high quality large-area 2D material (monolayer MoS_2) acting as saturable absorber to achieve the temporal confinement. The work opens new avenues to generate efficient and novel coherent light sources simultaneously featuring spectral, spatial, and temporal confinement at technologically relevant wavelengths. Moreover, ultra-compact coherent pulsed light source is achieved without the need of any voltage driven modulation, exhibits high frequency stability, and is compatible with LN-based integrated photonics.

4:20 PM

(ICACC-S14-014-2024) Composite Phosphor Ceramics for Warm White LED Lighting

R. Osborne*[1]; N. Cherepy[2]; R. M. Gaume[3]; S. A. Payne[1]

1. Lawrence Livermore National Laboratory, USA
2. Lawrence Livermore National Lab, Chemistry and Materials Science, USA
3. University of Central Florida, CREOL, USA

Phosphor ceramics offer advantages over traditional phosphor-in-silicone for conversion of blue LED light into white light, including their higher thermal conductivity and improved environmental stability. The high efficacy red phosphor, $K_2SiF_6:Mn^{4+}$, previously consolidated into a red-emitting transparent ceramic phosphor, has now been combined with yellow-emitting $YAG:Ce^{3+}$ particles to form a white light emitting composite phosphor ceramic. The emission spectrum is tunable based on the $YAG:Ce^{3+}$ particle loading, thickness, and Mn^{4+} doping concentration. This composite phosphor ceramic may be useful for high light flux applications due to its improved thermal conductivity and reduced Mn^{4+} concentration for mitigating droop.

4:40 PM

(ICACC-S14-015-2024) Development of a $Nd:SrF_2$ ceramic laser gain medium for high energy applications

T. Rudzik*[1]; N. Cherepy[1]; Z. M. Seeley[2]; S. A. Payne[1]

1. Lawrence Livermore National Lab, Chemistry and Materials Science, USA
2. Lawrence Livermore National Lab, Chemical Sciences Division, USA

As laser technology progresses, work on the cutting edge is requiring ever higher power output, which, in turn, requires higher energy storage capacity and improved thermal management. $Nd:SrF_2$ has been identified as a promising candidate to replace the doped phosphate glass currently used in the highest-energy laser systems. Despite this potential, the difficulties faced during processing have hindered the development of ceramic SrF_2 with sufficiently low optical scatter to be used in high energy laser applications and almost all work on this material has focused on single crystal growth, which may be prohibitively expensive in terms of both cost and fabrication time relative to sintering to form a ceramic. This work presents the first report of $Nd:SrF_2$, formed via powder processing without a liquid phase present, with an optical scatter below 1%/cm. The ability to form doping profiles is also demonstrated, and current progress in laser testing is discussed.

5:00 PM

(ICACC-S14-016-2024) Transparent Ceramic Laser Media Fabricated via Additive Manufacturing

R. Osborne*[1]; A. Drobshoff[1]; W. Rubink[1]; N. Cherepy[2]; I. Phillips[1]; R. Beach[1]; Z. M. Seeley[3]; 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

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, beam quality, and efficiency. Additive manufacturing techniques can be applied to produce ceramic green bodies with predetermined doping profiles that are then processed into transparent ceramic laser media. We have utilized this capability to produce transparent ceramics with many useful doping profiles, such as endcaps, cladding, and gradients in laser rods or even microscale structures such as waveguides. Here we report on some of the recent advances in these techniques and experimental laser results of these materials.

5:20 PM

(ICACC-S14-017-2024) Leveraging the Preferences in Transition Metal-P/Si Bonding to Design Novel Non-linear Optical Materials

K. Kovnir*[1]

1. Iowa State University, Chemistry, USA

Designing of non-centrosymmetric compounds is challenging. We built upon chemical bonding preferences in the transition metal silicon-phosphides TM-Si-P create the asymmetric environment around metal atoms resulting in high abundance of non-centrosymmetric chiral and polar crystal structures. The synthesis of those phases is hampered by inertness of TM and Si in contrast to high reactivity and vapor pressure of phosphorus. We developed a comprehensive synthetic strategy allowing to produce single crystals and phase-pure polycrystalline samples of metal tetrel-pnictides. The correlation between the crystal structure, chemical bonding, and non-linear optical properties will be discussed with focus of earth abundant TM for applications.

S1 Mechanical Behavior and Performance of Ceramics & Composites

SYMPOSIUM 1: Ceramics for concentrated solar–thermal power and industrial process heat II

Room: Coquina E
Session Chair: Kenneth Sandhage, Purdue University

1:30 PM

(ICACC-S1-046-2024) Fabrication and Testing of Receiver Design Feature Specimens in a Simulating Lab Test via Laser Heating

J. Shiang*[2]; D. Dunn[1]; D. Erno[2]; T. DeValk[2]; D. Decesare[2]; H. McGuigan[2]; W. Navojosky[2]; H. Yeong[2]; R. Sarrafi-Nour[2]

1. GE Aerospace, USA
2. GE Aerospace, Research Center, USA

Supported by an award from the Solar Technology Office, US Department of Energy, GE Research in collaboration with Heliogen Holdings Inc, is engaged in the development of ultra-High Operating Temperature SiC-matrix Solar Thermal Air Receiver (HOTSSTAR). We report the results of our study of the effective heat transfer characteristic of several candidate structure motifs, or feature geometries, made of SiC via additive manufacturing in a simulative laboratory test. The SiC structure motifs studied include different permutations of three-dimensional periodic lattices and defined shapes. The solar-thermal simulating laboratory test setup is constructed using a 4kW CO2 laser system with beam shaping optics to apply radiative heating power on one face of 2”-diameter cylindrical feature specimens representing the structure motifs of interest for receiver element design. Using the test setup, simulative test conditions representative of a concentrated solar flux of up to ~2000 suns could be achieved in the lab tests under varying air flow through the test structure. A numerical analysis scheme is developed to extract an effective or compound heat transfer coefficient representative of the test structure under steady-state heat flow conditions.

1:50 PM

(ICACC-S1-047-2024) Development of Ti_3AlC_2 MAX phase composites using reactive melt infiltration for CSP receiver applications

B. Ma*[1]; M. Du[1]; P. S. Chaugule[1]; D. Singh[1]

1. Argonne National Laboratory, USA

MAX phase materials exhibiting high thermal conductivity and superior mechanical properties at high temperatures (>800°C) are promising materials for solar power receivers for the next generation concentrating solar power (CSP) systems. We developed a fabrication process for producing MAX phase Ti_3AlC_2 composite parts by Al reactive melt infiltration. This scalable process uses TiC, TiO_2, and graphite carbon as starting chemicals. Resulting composite parts consists of mainly MAX phase Ti_3AlC_2 and Al_2O_3 phase, with small amounts of unreacted TiC and Al. Scanning electron microscopy of the fracture surfaces revealed dense homogeneous structure with plate-shaped MAX phase Ti_3AlC_2 grains of 0.5 mm thick and 2 to 5 mm in size and spherical Al_2O_3 phase of 1 to 2 mm in size. The resulting bulk materials contain a volume fraction of ≈70% Ti_3AlC_2 phase and ≈20% Al_2O_3 phase as determined by X-ray diffraction analysis. We measured bulk density of 3.9 g/cm^3 by Archimedes’ principle, which corresponds to greater than 91% of the theoretical value. Vickers hardness of 6.1 ± 0.5 GPa were observed at room temperature. Finally, fabrication approaches, including cold-pressing and additive manufacturing, were investigated to produce simple shaped MAX phase components using the reactive melt infiltration technique.

2:10 PM

(ICACC-S1-048-2024) Lifetime reliability prediction tool of ceramic receivers for Concentrated Solar Power

P. S. Chaugule*[1]; B. Barua[1]; M. C. Messner[1]; D. Singh[2]

1. Argonne National Lab, Applied Materials Division, USA
2. Argonne National Lab, USA

Concentrated solar power (CSP) receivers use solar energy to raise the temperature of a heat transfer fluid, that is used to generate electricity. To achieve the SunShot initiative’s 2030 target, next-generation CSP systems need to operate at a higher efficiency i.e. at outlet temperatures as high as 720 ºC. Nickel-based alloys used in current-generation CSP receivers can underdo creep-fatigue damage accumulation at the operating temperatures of next-generation CSP receivers. The current study presents the viability of using ceramic materials as an alternative, as they have better creep and dwell fatigue properties compared to Ni-based alloys. The study addresses the paucity of methods and tools required for designing high-temperature receivers by presenting srlife, an open-source computational tool. The tool can model a CSP receiver and predict its mechanical reliability over its lifetime. To predict reliability, the tool provides a suite of fracture mechanics based models to choose from. The models even account for sub-critical crack growth arising from creep and fatigue types of loading that can reduce the strength of ceramics. The result is a time-dependent reliability of the CSP receiver.

2:30 PM

(ICACC-S1-049-2024) Evaluation of Thermal & Mechanical Properties of Additively Manufactured Monolithic SiC for Solar Receiver Applications

A. Kebbede*[1]

1. GE Aerospace Research, USA

Owing to its high temperature stability combined with excellent thermal and optical properties, SiC has been the material of choice for application in high-temperature solar receivers. State-of-the-art SiC volumetric concentrating solar air receivers such as the honeycomb design have successfully demonstrated in field exit air temperatures approaching 800ºC. However, a successful application of CST systems for the decarbonization of heat reliant industrial sectors requires significant increases in the temperature capability of SiC receiver technology. Supported by an award from the Solar Technology Office (SETO), US Department of Energy (DOE), GE Research in collaboration with Heliogen Holdings Inc, is engaged in the development of an ultra-High Operating Temperature SiC-matrix Solar Thermal Air Receiver (HOTSSTAR) enabled by additive manufacturing. The program objective is to design and demonstrate a techno-economically viable SiC air receiver technology that can achieve exit air temperatures up to 1100ºC for CST applications. We report and discuss the results of our testing and evaluation of thermal and mechanical properties of reaction-bonded SiC fabricated via binder jet printing to support the receiver component design, development, and analyses.

2:50 PM

(ICACC-S1-050-2024) Thermo-Mechanical Modeling and Evaluation of Cracking Response of Additively Manufactured Monolithic SiC Lattice Structures Subjected to Laser Heating

A. Kebbede*[1]

1. GE Aerospace Research, USA

Supported by an award from the Solar Technology Office (SETO), US Department of Energy (DOE), GE Research in collaboration with Heliogen Holdings Inc, is engaged in the development of ultra-High Operating Temperature SiC-matrix Solar Thermal Air Receiver (HOTSSTAR) enabled by additive manufacturing. The program objective is to design and demonstrate a techno-economically viable SiC air receiver to achieve exit air temperature of 1100ºC for CST applications. We present the learnings of a computational study on additively manufactured SiC lattice structures subjected to laser heating. Results will guide design of SiC receivers through a better understanding of lattice structure attributes and their influence on cracking probablity. A heat flux was applied to the surface of a 2”-diameter cylindrical lattice structure to simulate a 4kW CO2 laser. The resulting temperature distribution was applied to a structural model to approximate the stress distribution within the lattice structure and a Weibull analysis was performed to gain insight into the probability of failure. Two different lattice beam spacings were studied. A discussion on modeling assumptions, a comparison with experimental results, and an evaluation of the lattice cracking response is provided.

S3 21th Intl Symp on Solid Oxide Cells Materials Science & Technology

SYMPOSIUM 3: Metal supported cells, interconnect coating and interfaces

Room: Ballroom 1-2
Session Chairs: Federico Smeacetto, Politecnico di Torino;
John Hardy, Pacific Northwest National Laboratory

1:30 PM

(ICACC-S3-049-2024) Interconnect and Balance of Plant Component Coatings for Solid Oxide Electrolysis Stacks and Systems (Invited)

S. Ibanez[1]; K. Adepalli*[1]; G. Merchant[1]; S. Swartz[1]

1. Nexceris, LLC, USA

Solid oxide electrolysis (SOEC) is receiving a lot of interest across diverse industries such as oil & gas, chemical, steel, grid and nuclear companies. SOEC offers unique benefits that no other competing technology would offer in terms of efficiency, fuel flexibility, reversibility, and ability to co-electrolyze steam and CO_2 to produce syngas. Despite these unparalleled features, SOEC often struggles with a wide range of acceptability due to durability and cost concerns. Durability is associated with their operating temperatures, typically 600 to 850 ºC; which limits material choices for alloys that are stable and oxidation resistant. In this presentation, we will show interconnect degradation via Cr evaporation from the steel interconnects and our approach to suppress it by manganese cobalt oxide (MCO) coatings – ChromLok^{TM}. We will also discuss our approach to protect stack and high temperature BOP components. Nexceris developed protective coatings decrease the stainless-steel oxidation rate by about one order in magnitude and decrease the Cr evaporation rate dramatically. We demonstrate coatings compatibility in stacks tested in SOEC, as well as reversible solid oxide cells (rSOC) over a few thousand hours with a low degradation rate. We would also present scalability of these coatings and cost effectiveness when made at scale without increasing the overall stack cost.

2:00 PM

(ICACC-S3-050-2024) Plasma-sprayed low temperature metal-supported solid oxide fuel cells

C. Chang*[1]; C. Tsai[1]; C. Yang[1]; C. Yang[1]

1. Institute of Nuclear Energy Research, Physics Division, Taiwan

This study focuses on the fabrication and performance evaluation of low-temperature metal-supported solid oxide fuel cells (LT-MSSOFCs). These fuel cells utilize bilayer and trilayer solid oxide electrolytes consisting of samarium-doped ceria (SDC) and lanthanum-doped strontium gallate (LSGM) materials. The fabrication technique employed is atmospheric plasma spraying (APS). To improve the performance of the cathode and reduce cathode polarization resistance, two mixed ion-electron conductor (MIEC) cathode materials called $Ba_{0.5}Sr_{0.5}Co_{0.8}Fe_{0.2}O_{3-\sigma}$ (BSCF) and $PrBaCo_2O_{5+\sigma}$ (PBC) were chosen. The electrical testing results indicate that a 5×5 cm^2 LT-MSSOFC with a tri-layer electrolyte exhibits the highest output power density of 462 mW cm^{-2}, 693 mW cm^{-2}, and 858 mW cm^{-2} at 550°C, 600°C, and 650°C, respectively. Similarly, a 10×10 cm^2 LT-MSSOFC with a tri-layer electrolyte provides power outputs of 320 mW cm^{-2} (26W), 560 mW cm^{-2} (44.8W), and 798 mW cm^{-2} (63.9W) at 550°C, 600°C, and 650°C, respectively. Furthermore, the long-term stability tests on the LT-MSSOFCs by subjecting them to a constant current of 300mA cm^{-2} at 550°C. The results show that the degradation rate of the LT-MSSOFCs under these conditions is 0.88% per 1000 hours, indicating reasonable stability over an extended period of operation.

2:20 PM

(ICACC-S3-051-2024) High-temperature oxidation of porous stainless steels and its mitigation strategies

D. Koszelow[1]; S. Molin*[1]

1. Gdansk University of Technology, Department of Functional Materials Engineering, Poland

Our group has been working on the high-temperature properties of porous FeCr alloys for several years, mainly with the potential application in solid oxide cells. Unfortunately, due to the large surface to volume ratio, the long-term application of porous alloys at >700°C is questionable. In this work we will show the oxidation behavior of the unmodified Fe22Cr alloy as well as the pre-oxidized (PO) alloy and the atomic layer deposition (ALD) modified alloy. The pre-oxidation approach aims to produce a well defined chromia scale during the initial high-temperature exposure. This initial exposure is carried out for just a short time (~1 hour) at temperature of 850/900°C, so well above the projected operating temperature (700/750°C). During the pre-oxidation, relatively large chromia crystals grow with fewer grain boundaries, reducing the effective outward cation diffusion and hence the subsequent oxide growth kinetics afterwards. In the atomic layer deposition approach we chose to deposit thin and well controlled layers of alumina to also block grain boundary diffusion. As alumina is not electrically conductive, we are only testing alloys with thin layers (<100 nm), not to form a continuous high resistance layer. Our results show, that by carefully designing the porous alloys with surface modifications (PO/ALD), the lifetime of the alloys can be extended by almost an order of magnitude.

2:40 PM

(ICACC-S3-052-2024) Improved interfaces and innovative joining strategies for high-pressure solid oxide electrolyzer integration

F. Smeacetto*[1]; E. Zanchi[1]; M. Ferraris[1]; M. Salvo[2]; D. Menon[1]; D. Janner[1]; A. Sabato[3]; A. Tarancón[4]

1. Politecnico di Torino, Applied Science and Technology, Italy
2. Politecnico di Torino, Italy
3. IREC, Nanoionics and Fuel Cells, Spain
4. IREC / ICREA, Spain

A stable and durable SOEC operation at high pressure has the potential to provide a cost-effective solution for hydrogen production in the industrial and transport sectors. The main goal of the HyP3D EU-funded project is to deliver a new generation of ultra-compact high-pressure standalone SOEC stacks. Reaching this goal is unquestionably related to the high-pressure resistant joining and integration strategies when considering metallic, ceramic, and glass-based sealing interfaces. The engineering of the interfaces between ceramic and metallic materials is crucial when considering the different stress conditions in the pressurized SOEC stack. A proper surface modification of joining parts, laser machining in metallic interconnect, and 3D printing of the ceramic YSZ electrolytes, can improve dissimilar materials’ interlocking mechanism, thus increasing the joined structure’s shear strength and adhesion. Recent advances in the framework of the HyP3D project, related to novel laser surface modification strategies to enhance the glass-to-metal sealing mechanical robustness, are presented and discussed.

3:20 PM

(ICACC-S3-053-2024) Development of Metal-Supported SOFCs for Non-Hydrogen Fuels (Invited)

M. Abdul Jabbar*[1]; D. Thompson[1]; C. Gumeci[1]; J. Parrondo[1]; Y. Furuya[1]; N. Dale[1]

1. Nissan, USA

Solid oxide fuel cells (SOFCs) are well-known for their fuel-flexible operations; for example, SOFCs can operate on simple and complex renewable fuels (such as ethanol, natural gas, jet fuel, propane, etc.). However, performance and durability are still under investigation and development for applications that need to meet stringent functional requirements. The challenges involve the development of low-resistance and structurally strong cell designs, catalysts for internal reforming, and efficient electrochemical reactions on the electrodes. This article discusses the challenges and remedies to overcome them to develop metal-based SOFCs for highly performing and durable power generators.

3:50 PM

(ICACC-S3-054-2024) Metal-supported solid oxide cells for hydrogen production and direct utilization of methanol, ethanol and methane (Invited)

B. Hu*[1]; Z. Zhu[1]; M. Welander[1]; F. Shen[2]; G. Lau[1]; M. Tucker[2]

1. Lawrence Berkeley National Laboratory, Energy Storage and Distributed Resources, USA
2. Lawrence Berkeley National Laboratory, USA

Recent advancements in the development of metal-supported solid oxide cells (MS-SOCs) at Lawrence Berkeley National Laboratory have enhanced cell performance and stability through improvements in cell structure, catalyst utilization, and mass transport. The unique symmetric architecture design, with porous ZrO_2 ceramic backbones and dense electrolyte sandwiched between thin MS, offers excellent tolerance to redox cycling, extremely fast start-up capability, strong mechanical ruggedness, and low material cost. These advantages of MS-SOC promote various energy applications including electrolysis for hydrogen production, chemical synthesis, vehicle range extenders, emergency power generators, and distributed generation for data centers. Cell structure, including the porosity and layer thickness, greatly affects mass transport and catalyst loading, which is also controlled by process methods and parameters. Infiltration is widely utilized for loading catalysts into MS-SOCs. Firing temperature and number of infiltration cycles were optimized for high performance and durability using high-temperature XRD for phase purity/stability analysis. Operation for 500 h with direct utilization and 1 kh steam electrolysis will be presented. Button cells (4.5 cm^2) have been scaled up to 45 cm^2 planar cells. Degradation mechanisms will be discussed with posttest analysis results.

4:20 PM

(ICACC-S3-055-2024) Optimisation of the electrophoretic deposition technique to process ceramic protective coatings for reversible solid oxide cells

E. Zanchi*[1]; M. Torrell[3]; L. Bernadet[3]; M. Salvo[1]; D. Montinaro[2]; F. Smeacetto[1]

1. Politecnico di Torino, Department of Applied Science and Technology, Italy
2. SolydEra Spa, Italy
3. IREC - Catalonia Institute for Energy Research, Department of Advanced Materials for Energy Applications, Spain

In the reversible SOC stack operation, the exposure to high current density, thermal gradients and corrosive atmosphere can lead to harsh degradation phenomena of the metallic interconnect (IC). Depositing a protective coating is an accepted approach to protect the IC from the main causes of degradation, i.e. high-temperature corrosion and chromium evaporation. Currently, there is a general interest in validating new coating compositions to avoid cobalt as a critical raw material. The choice of the deposition process plays a crucial role in the performance of the obtained materials. The electrophoretic deposition (EPD) method allows to obtain coatings with reproducible properties in a few seconds and at room conditions. In the framework of the EU project 24/7 ZEN, EPD is used to deposit dense and continuous manganese-copper spinel coatings on AISI 441 stainless steel. The co-deposition approach is exploited to process iron-doped spinel coating to evaluate the improvement of thermal stability and protective properties. Furthermore, the synergy between EPD spinel coatings and the sputtering technique to develop multi-layered coating systems is proposed as a possible route to enhance the coating performances in terms of Cr retention capability.

4:40 PM

(ICACC-S3-056-2024) Long-term oxidation (10000h at 750°C) of steel interconnect coated with Fe-stabilized MnCu-oxide spinel

J. Ignaczak*[1]; P. Jasinski[1]; S. Molin[1]

1. Gdansk University of Technology, Department of Functional Materials Engineering, Poland

High-temperature corrosion of steel interconnects of solid oxide cell stacks is one of the main challenges in the development of durable and efficient stacks. The most popular coating material is based on Mn-Co-oxide spinels. Cobalt is one of the critical raw materials, thus its use should be cautious and possibly replacement materials should be found. One of the alternative materials are Mn-Cu-oxide spinels. They possess high electrical conductivity, good thermal expansion coefficient match, but their long-term stability is not yet satisfactorily documented. In this work we show our results concerning 10000 hours oxidation test of Mn-Cu-Feoxide coating prepared by electrophoretic deposition (EPD) method. We have carried out cyclic thermogravimetry, extensive microscopy and structural studies to describe in detail the behaviour of the coated Crofer 22 APU steel. We discuss the relevance of iron stabilization of the single-spinel phase and its effects on the physicochemical properties.

S6 Advanced Materials and Technologies for Rechargeable Energy Storage

SYMPOSIUM 6: Lithium-ion, Lithium-sulphur and all-solid-state batteries

Room: Ballroom 5
Session Chair: William Chuirazzi, Idaho National Lab

1:30 PM

(ICACC-S6-046-2024) Creating efficient Li-ion conducting pathways in composite electrodes for all-solid-state batteries using a liquid-phase process (Invited)

M. Calpa*[1]

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

All-solid-state batteries (ASSBs) based on sulfide solid electrolytes are considered next-generation energy storage systems, offering improved safety and energy density. A key factor determining the performance of ASSBs is the formation of solid ion-conductive pathways in the composite electrodes. A high content of solid electrolyte (> 50 wt.%) in composite electrodes facilitates Li-ion percolation but compromises the energy density of ASSBs. In this work, a liquid-phase process is used to fabricate effective Li-ion pathways in composite electrodes while using low amounts of solid electrolyte (<20 wt.%). A high ion-conductive $Li_7P_3S_{11}$ solid electrolyte with a small particle size (<1μm) was attained by a liquid-phase synthesis and used as Li-ion conductive additive in a composite electrode comprising NMC ($LiNi_{1/3}Mn_{1/3}Co_{1/3}O_2$) as the active material. The solid electrolyte coating on active materials (NMC or graphite) by an electrolyte dissolution-reprecipitation approach was also investigated. The homogeneous distribution of solid electrolyte in composite electrodes, achieved by using a nano-sized solid electrolyte or by the solid electrolyte coating on active materials, resulted in a marked enhancement in the performance of ASSBs.

2:00 PM

(ICACC-S6-047-2024) Improvement of peeling efficiency of cathode active materials from lithium-ion batteries by the direct electric pulse discharging disassembly method

A. Narita*[1]; T. Kurihara[1]; T. Koita[1]; K. Oyama[1]; S. Higuchi[1]; C. Tokoro[1]

1. Waseda University, Japan

We are developing the disassembly method of various devices, including Recycling lithium-ion secondary batteries (LiBs), in which high-power electric pulse discharging is applied to the samples directly. This method has advantages over electrodynamical fragmentation, such as easier aimed parts separation with lower energy cost. We reported that the method enables the peeling of active cathode materials from aluminum foils at once, which suggested that the pulsed discharge is an effective separation method in direct recycling. And we are applying the energy density to standardize the discharging conditions for various shapes of the samples. However, discussing how to solve the uneven peeling, which sometimes happens, is necessary. This study discussed the effect of chemical conditions on peeling behavior. The peeling behavior of cathode sheets from LiBs (including active materials, carbon, lithium salts, organic components, and aluminum) with various millage histories in vehicles was observed after direct pulse discharging. While the millage history of the cells did not affect the peeling behavior, the time from the disassembly of the LiB cells affected the one. The results indicate that rapid electrical pulse treatment of LiBs enables them to overcome chemical variability with their used histories on peeling behavior.

2:20 PM

(ICACC-S6-048-2024) Silica Depleted Rice Hull Ash (SDRHA) Stabilizes Cycling in Li-S Batteries

R. M. Laine*[1]; P. Y. Kim[2]

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2. University of Michigan, Macromolecular Science and Eng., USA

As part of our effort to develop sustainable materials as components of lithium -ion batteries, we have learned to use rice hull ash (RHA) in a variety of applications.[1–3] RHA is a byproduct of the combustion of rice hulls to generate electricity at gigawatts/yr in the U.S. alone. RHA typically consists of nanocomposite mixtures of silica and hard carbon (90 wt % SiO_2). We have learned to distillatively remove this silica in one step by reaction of RHA with hexylene glycol and catalytic base.[4] The resulting material, $SDRHA_{44}$ (44 wt % SiO_2) can have SSAs ≥ 500 m^2/g and 4-5 nmave pore sizes. Here, we discuss adding sulfurized polyacrylonitrile[5] (≈ 40 wt % S) with SDRHA to formulate Li-S systems that exhibit improved capacities and stability at 1 & 2 C for > 100 cycles. The work to be presented is supported by NSF DMR Grant 1926199 and Mercedes Benz R&D. 1. Temeche, E. Yu, M., Laine, R. M. Green Chemistry. 2020;22:4656–4668. 2. Yu M, Temeche E, Indris S, Laine RM. Green Chem. 2021;10.1039. D1GC02084F. 3. Zhang X, Temeche E, Laine RM. LixSiON (x = 2, 4, 6); Green Chem. 2020;10.1039.D0GC02580A. 4. Laine RM, Furgal JC, Doan P, Pan D, Popova V, Zhang X. Avoiding Carbothermal Reduction: Distillation of Alkoxysilanes from Biogenic, Green, and Sustainable Sources. Angew Chem Int Ed. 2016;55(3):1065–1069. 5. Wang J, Yang J, Wan C, Du K, Xie J, Xu N. Adv Funct Mater. 2003;13(6):487–492.

2:40 PM

(ICACC-S6-049-2024) New Technology of Electrolyte Development for Extreme Fast Charging

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Realizing extreme fast charging (XFC) in lithium-ion batteries for electric vehicles is still challenging due to the insufficient lithium-ion transport kinetics, especially in the electrolyte. Herein, a novel high-performance electrolyte (HPE) is proposed and tested in pilot-scale, 2-Ah pouch cells. Moreover, the origin of improved electrochemical performance is comprehensively studied via various characterizations, suggesting that the proposed HPE exhibits high ionic conductivity and excellent electrochemical stability at high charging rate of 6-C. Therefore, the HPE-based pouch cells deliver improved discharge specific capacity and excellent long-term cyclability up to 1500 cycles under XFC conditions, which is superior to the conventional state-of-the-art baseline electrolyte.

S7 18th Intl Symp on Functional Nanomaterials & Thin Films for Sustainable Energy Harvesting

SYMPOSIUM 7: Nanomaterials for energy conversion, storage and catalysis

Room: Coquina B
Session Chair: Sedat Ballikaya, Istanbul University

1:30 PM

(ICACC-S7-027-2024) Tailor-Made White Photothermal Fabrics: A Bridge between Pragmatism and Aesthetic (Invited)

J. Yuan*[1]

1. Stockholm University, MMK, Sweden

Maintaining human thermal comfort in cold wild is crucial for diverse outdoor activities, e.g., sports and recreation, health-care, and special occupations. To date, advanced clothes are employed to collect solar energy as heat source to stand cold climates, while their dull dark photothermal coating may hinder pragmatism in wild and visual sense considering fashion. Herein, tailor-made white webs with strong photothermal effect are proposed. With the embedding of Cs_xWO_3 nanoparticles (NPs) as additive inside nylon nanofibers, these webs are capable of drawing both NIR and UV light in sunlight for heating. Their exceptional photothermal conversion capability enables 2.5-10.5 °C warmer than that of a commercial sweatshirt of 6 times thicker under different climates. Remarkably, this smart fabric can upgrade its photothermal conversion efficiency by more than doubled in a wet state, which makes it a competitive candidate for quick-drying of sports clothes frequently wetted by sweat under solar light. Obviously, this smart web with considerable merits of shape retention, softness, safety, breathability, washability and on-demand coloration, provides a revolutionary solution to realize energy-saving outdoor thermoregulatory and simultaneously satisfy the needs of fashion and aesthetics.

2:00 PM

(ICACC-S7-028-2024) MXene/cellulose composite cloth for integrated functions in personal heating and steam generation

J. Chang*[1]; B. Pang[1]; H. Zhang[1]; K. Pang[1]; M. Zhang[1]; J. Yuan[2]

1. Stockholm University, Department of Materials and Environmental Chemistry, Sweden
2. Stockholm University, MMK, Sweden

Given the abundant solar light available on our planet, the development of advanced fabric capable of simultaneously providing personal thermal management and facilitating clean water production in an energy-efficient manner has emerged as a promising avenue for research. In this study, we present the fabrication of a photothermally active, biodegradable composite cloth composed of MXene and cellulose through an electrospinning method. This composite cloth exhibits favorable attributes, including chemical stability, mechanical performance, structural flexibility, and wettability. Notably, our 0.1-mm-thick composite cloth raises the temperature of simulated skin by 5.6 °C when compared to a commercial cotton cloth, which is five times thicker under identical ambient conditions. Remarkably, the composite cloth demonstrates heightened solar light capture efficiency (87.7%) in a wet state. Consequently, this cloth functions exceptionally well as a high-performance steam generator, boasting a superior water evaporation rate of 1.34 kg m^{-2} h^{-1} under one-sun irradiation. Moreover, it maintains its performance excellence in solar desalination processes. The multifunctionality of these cloths opens doors to a diverse array of outdoor applications, including solar-driven water evaporation and personal heating, thereby enriching the scope of integrated functionalities for textiles.

2:20 PM

(ICACC-S7-029-2024) Nanocomposites for Mechanical Energy Harvesters and Sensors Towards Building Flexible and Wearable Electronics

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1. Universidad de Chile, Mechanical Engineering, Chile
2. Universidad de Concepcion, Electrical Engineering, Chile

Realizing the potential of Internet-of-things (IoT) technology, the demand of flexible electronic devices led the scientific community to develop novel electroactive materials for various sensing and energy harvesting devices to realize them in the potential applications in health monitoring, robotics, electronic skin and diagnostics. A self-sustainable device which can work independently is essential for fulfilling the requirements of miniaturization and to deliver sustainable energy using the different ambient forms. The harvesting of renewable energies by piezo-, thermo-electric forms; and simultaneously sensing the dynamic responses which can be realized for self-powered sensors applications due to their self-sustainable capability, which can function by built-in electric potential without the need of an additional power source batteries. Materials for flexible electronics sensors generally consist of electroactive polymers and ceramics as dielectric layer. Polymers such as poly (dimethylsiloxane), polyimide, polyvinylidene fluoride and its copolymers are widely used which are flexible, robust and have low dielectric properties. On the other hand, various dielectric nanocomposites were developed with superior energy harvesting and sensing performance and have wide applications in flexible and wearable electronics in electromechanical and biomedical applications.

2:40 PM

(ICACC-S7-030-2024) Exploring novel 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 ofelectrochemistry where new generation of chalcogenides and phosphide have been shown to be highly effective. We shall like to report on exploring less common types of metal chalcogendies which through rational design with a good control in the synthesis of the material with pre-determined structures and morphologies, one is able to extract unique properties which may not be known previously. For example, we will show that metal chalcogenides such as SnS showspromise as thermoelectric materials with high power factor at room temperature while in the phosphide family, 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 CuS has potential in PEC applications.

S9 Porous Ceramics Novel Developments and Applications

SYMPOSIUM 9: Porous Ceramics for Environmental, Energy, Biological and Functional Applications II

Room: Coquina D
Session Chairs: Gisele Laure Lecomte-Nana, ENSCI;
Enrico Bernardo, University of Padova

1:30 PM

(ICACC-S9-027-2024) A new high-performance lightweight composites made of hollow ceramic particles by spark plasma sintering method: preparation and characterization

J. Ozolins[1]; V. Abramovskis[1]; I. Zalite[1]; I. Steins[2]; V. Lapkovsis[2]; A. Shiskin*[2]

1. Institute of General Chemical Engineering, Latvia
2. Institute of Materials and Surface Technologies of the Riga Technical University, Latvia

Obtaining of new materials with reduced is crucial for new technologies and products design. One of the methods to reduce density can be the production of a composite as a syntactic foam. Cenospheres (CS) can be used as a closed cell matrix [1]. Cenospheres have many important properties, such as low heat transfer coefficient, high hardness, lack of electrical conductivity and complete chemical inertia, low density: the true is 0.65-0.7 and bulk density 0.39-0.42 g/cm^3. In the experimental CS can was coated using physical vapor deposition method (PVD) by metal different metals and alloys – Cu, Ti6Al4V and 316 stanless steel which acts as a binder during the sintering process. The lightweight composite material, using spark plasma sintering method was produced. The influence of the SPS process parameters (different sintering temperatures) on the apparent density of the composite, compressive strength was investigated. The obtained material is specified by: compression stress is in range from 26 to 91 MPa; bulk density form 0,7 to 1.5 gcm^{-3} bulk density; characterised by mechanical stability up to 1000 °C. Developed composite materials are promising for aerospace and defence sectors use.

1:50 PM

(ICACC-S9-028-2024) Production of periodic open cellular structures (POCS) by additive manufacturing for catalytic applications (Invited)

R. Balzarotti*[2]; S. Bottacin[2]; M. Pelanconi[2]; G. Bianchi[2]; A. Ortona[1]

1. SUPSI, MEMTi, Switzerland
2. SUPSI, Department of Innovative Technologies, Switzerland

The development of structured reactors is a hot topic in heterogeneous catalysis, as it represents one of the most promising solutions to overcome most of limitations of traditional packed bed reactors. In literature, supports such as honeycomb monoliths, foams, and meshed wires have been reported as valuable candidates for the intensification of non-adiabatic processes. Nevertheless, the recent advances in additive manufacturing (AM) have enabled a new degree of freedom in structured catalyst design, allowing to control precisely both the shape and the material used in the production process. The present work aims at providing insights regarding the use of AM for the development of tailored-made structured catalysts for heterogeneous processes. The investigation of a two-steps procedure for carbon-based POCS backbone production is herein reported: first, a porous polymer-based preform was produced by selective laser sintering (SLS); then, the lattice was infiltrated with the precursor of the final material and pyrolyzed. Finally, two washcoating procedure (i.e., electrophoretic deposition and slurry coating) have been compared for the deposition of thin Al_2O_3 layers onto the POCS surface.

2:20 PM

(ICACC-S9-029-2024) The Effect of Slurry Parameters on the Properties of Spray Dryed FCC Catalyst Granules

E. S. Bukcu*[1]; I. Okten[2]; C. Aciksari[2]; S. Celebi[2]; U. Savaci[1]; S. Turan[1]; E. Ayas[1]

1. Eskisehir Technical University, Materials Science and Engineering, Turkey
2. Tupras R&D, R&D, Turkey

Improving the catalytic activity, selectivity and accessibility of zeolite-based FCC catalysts has become one of the major research issues in the recent past, driven by the environmental regulations on the quality and greenhouse gas emission levels of liquid fuels such as gasoline. These catalysts are produced through spray drying and their efficiency strictly depends on the process conditions and slurry characteristics. While there are many studies on the rheological behaviour of zeolite and clay systems separately, the impact of slurry characteristics of zeolite-clay-binder systems on the final granule properties is still unexplained. This study aims to address this gap in the existing literature by exploring how various factors - including zeta potential, yield stress, binder/clay ratio, dispersant level, and ZSM-5 silica-to-alumina ratio – impact the properties of granules produced through spray drying. Specifically, the granule shape, density, porosity, attrition index and other relevant characteristics are examined. Observations have revealed that the interparticle interactions within the slurry exert a determinative influence upon the internal structural disposition within the granule during spray drying which subsequently impacts on both the accessibility and attrition resistance of the resultant catalyst materials.

2:40 PM

(ICACC-S9-030-2024) Biomass-metakaolin based porous matrices for insulation and thermal comfort in buildings

E. Kamseu*[1]

1. MIPROMALO, Research, Cameroon

H. TCHENEGHON MOTCHEYO[1,2], L.K. Tiogning Djiogue[3], P. Melatagia[2], E. Kamseu[1,5], S. Rossignol[4], Cristina Leonelli[5] Biomass is recycled into the design of porous insulating and hygroscopic matrices. The amorphous and reactive bio sourced silica allows to monitor the pore network. Spatial geometrical distribution of pores, size and interconnectivity of nano, meso and millipores are used to predict the efficiency of the insulation and thermal comfort. Based on the data collected on the interactions between the pores network system and the hygroscopic behaviors, Artificial Intelligence tools are used to validate the correlation between the pore network systems and the hygroscopy and insulating parameters. Consequently the biomass based porous systems hold immense potential in the area of building energy Conservation and thermal comfort regulation.

S6 Advanced Materials and Technologies for Rechargeable Energy Storage

SYMPOSIUM 6: Advanced anode and cathode materials for sodium battery and capacitors

Room: Ballroom 5
Session Chairs: Huiwen Ji, University of Utah; Marcela Calpa, National Institute for Materials Science (NIMS)

3:20 PM

(ICACC-S6-050-2024) Development of Nanosized Na_5FeO_4 for Cost-effective Sodium-Ion Battery Cathode Using Anionic Redox

S. Kodaki*[1]; H. Kobayashi[2]; I. Honma[1]

1. Tohoku University, Institute of Multidisciplinary Research for Advanced Materials, Japan
2. Hokkaido University, Department of Chemistry, Faculty of Science, Japan

These days, given the increasing demand for lithium-ion batteries (LIBs), it is concerned that such as resource shortages and high prices (particularly for Li or Co). Sodium-ion batteries (SIBs) have gaining attention due to the economical advantage of Na. As a novel cathode material, Na-rich transition metal oxides are expected to have a large energy density per weight. In this work, we synthesized Na-superrich Na-Fe oxide Na_5FeO_4 by room temperature ball milling method and it suggested higher energy density than other cost-effective SIB cathodes. β-$NaFeO_2$ and annealed Na_2O were added into an yttria-stabilized ZrO_2 (YSZ) ball milling jar with YSZ balls at the molar ratio of Na : Fe = 5 : 1 and milled by using ball milling. For structural characterization, XRD, SEM and XAS (X-ray Absorption Spectroscopy) were performed. The cathode was prepared by mixing Na_5FeO_4 with Ketjen Black and polytetrafluoroethylene, the anode and electrolyte were Na metal and 1 M $NaPF_6$, respectively. The XRD pattern suggests that amorphous Na_5FeO_4 was synthesized by ball milling. The charge-discharge tests show high reversible capacity of approximately 230 mAh g^{-1}, corresponding to two-electron reaction. At the discharge, three plateaus were observed. This suggests that Fe and O redox arose, corresponding to the XAS results. This sample showed higher energy density than other cost-effective SIB cathodes.

3:40 PM

(ICACC-S6-051-2024) Li_2CO_3 coated hematite Fe_2O_3 nanocomposite for Li-ion rechargeable batteries

A. Midouni*[1]; A. Hamzaoui[1]

1. CNRSM, Tunisia

In this work, we proposed a new strategy for the first time using a facile one-step coprecipitation process at room temperature to obtain an amorphous Li_2CO_3-coated nanocrystalline α-Fe_2O_3 hierarchical structure. Several techniques of characterization, such as differential thermal and thermogravimetric analysis (DTA/TG), fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), EDS analysis, transmission electron microscopy (TEM), X-ray diffraction analysis (XRD), Raman spectroscopy, electron paramagnetic resonance (EPR) and electrochemical measurements were applied. XRD and EPR show that the mesoporous α-Fe2O3 synthesized by solution combustion and the $Li_2CO_3@Fe_2O_3$ nanocomposite have the same structure. All the main diffraction peaks can be indexed to the hexagonal crystal structure hematite (a=b=0.5038 nm, c=1.3772 nm) with the R-3c space group. The prepared Li_2CO_3-coated nanocrystalline α-Fe_2O_3 manifested high stable long-term cyclability as it delivers a reversible capacity of 975 mAh g^{-1}, high columbic efficiency (97%-99%), after 700 cycles at 100 mA g^{-1} and high specific discharged capacity. At a high rate of 2000 mA g^{-1}, its reversible capacity still remains at 550 mAh g^{-1}, superior to the uncoated counterpart (297 mAh g^{-1}). The superior performance of the $Li_2CO_3@Fe_2O_3$ nanocomposite mainly depends on the strong interaction of amorphous Li_2CO_3 and hollow α-Fe_2O_3.

4:00 PM

(ICACC-S6-052-2024) Electrically Conductive Ultrasmall $CuMn_2O_4$ Cathodes: A Novel Approach to Room-Temperature Magnesium Rechargeable Batteries

R. Iimura*[1]; H. Kobayashi[4]; H. Watanabe[2]; T. Mandai[3]; H. Imai[2]; I. Honma[1]

1. Tohoku University, Japan
2. Keio University, Japan
3. National Institute for Materials Science (NIMS), Japan
4. Hokkaido University, Japan

Magnesium rechargeable batteries stand as promising alternatives to lithium-ion batteries, offering advantages like cost-effectiveness, superior safety features, and a higher volumetric energy density. Despite this promise, designing cathode materials that can achieve both high energy and high-rate capabilities remains an ongoing challenge. Among various cathode materials, oxide-based spinels have become a focus of attention because they offer higher voltage operational capacities. However, most prior studies have been limited to high-temperature conditions, mainly due to the slow diffusion rate of magnesium ions. In this study, we successfully fabricate an electrically conductive, ultrasmall $CuMn_2O_4$ spinel with dimensions less than 5 nm through an alcohol reduction process. This newly developed 'ultrananospinel' demonstrates a semi-reversible phase transition during the Mg ion insertion and ejection process. Additionally, it features a dual-redox system involving both copper and manganese ions. Notably, our ultrananospinel cathode material showcases high-voltage operation exceeding 1.5 V and possesses a theoretical discharge capacity of 225 mAh/g, along with impressive high-rate capabilities. These findings open new avenues for the development of high-performance, room-temperature magnesium rechargeable batteries.

4:20 PM

(ICACC-S6-053-2024) Gadolinium tailored energy storage in $BiFeO_3$–$BaTiO_3$ based relaxor ferroelectric ceramics

R. Chien*[1]; J. Lin[5]; R. H. Montecillo[2]; C. Chen[3]; P. Chen[4]; C. Tu[5]

1. Ming Chi University of Technology, International Ph.D. Program in Innovative Technology of Biomedical Engineering and Medical Devices, Taiwan
2. Ming Chi University of Technology, Innovative Technology of Biomedical Engineering and Medical Devices, Taiwan
3. Hwa Hsia University of Technology, Mechanical Engineering, Taiwan
4. Ming Chi University of Technology, PhD Program in Biomedical Engineering and Medical Devices, Taiwan
5. Fu Jen Catholic University, Physics, Taiwan

Solid-state dielectric capacitors have become significant components for energy-storing applications due to their high-temperature stability and fast charging-discharging speed. High configuration-entropy driven compositions in perovskite-based oxides have been widely studied to increase the recoverable energy density (W_{rec}) and energy storage efficiency (η). Lead-free $BiFeO_3$-$BaTiO_3$ (BF-BT) system is considered as promising materials for static electric energy storage owing to the high polarization in $BiFeO_{3,}$ and the relaxor nature of BF-BT at the morphotropic phase boundary. $(Bi_{1-x}Gd_x)FeO_3$-$BaTiO_3$ ceramics (x = 0 - 0.4) were studied through tailoring remanent polarization and breakdown electric field strength (E_b) to achieve high W_{rec} and η. W_{rec} ~ 6.5 J/cm^3 (at E_b = 260 kV/cm) and η ~ 75% (at E_b = 280 kV/cm) were attained at x = 0.15 and x = 0.3, respectively. The E_b was significantly enhanced from ~120 to ~260-280 kV/cm by adding Gd_2O_3 in BF-BT ceramic via increasing the configuration entropy from 1.22R to 1.69R. The rising configuration entropy disrupts the long-range order in the ferroelectric phase and stablizes a relaxor ferroelectric state charaterized by multi-phase lattice distortions with the formation of nanoscale domains and polar nanoregions. A-site Gd-modified BF-BT ceramics have demonstrated to be promising candidates for electric energy-storing capacitors.

4:40 PM

(ICACC-S6-099-2024) Superb electric energy storage achieved in relaxor ferroelectric $BiFeO_3$-$BaTiO_3$-$NaNbO_3$ ceramics via O_2 atmosphere

R. H. Montecillo*[1]; C. Chen[3]; R. Chien[1]; P. Chen[4]; C. Tu[2]

1. Ming Chi University of Technology, Innovative Technology of Biomedical Engineering and Medical Devices, Taiwan
2. Fu Jen Catholic University, Physics, Taiwan
3. Hwa Hsia University of Technology, Mechanical Engineering, Taiwan
4. Ming Chi University of Technology, PhD Program in Biomedical Engineering and Medical Devices, Taiwan

Lead-free perovskite dielectric materials have been widely investigated for energy storage applications due to its high polarization and reversible phase. However, their energy density and stability are limited by their low electric resistivity. In this study, the (1-x)$(0.7BiFeO_3$-$0.3BaTiO_3)$-x$NaNbO_3$ ceramics (x = 0 – 0.2) were prepared using two sintering atmospheres (air and oxygen). A superb recoverable energy-stored density W_{rec} of 8.2 J/cm^3 and an efficiency η of 70% at 300 kV/cm were achieved at x = 0.15 sintered in O_2 atmosphere. The superior W_{rec} is due to the structural coexistence (ferroelectric rhombohedral R3c, nonpolar cubic Pm-3m, and antiferroelectric orthorhombic Pbam symmetries), tailored via antiferroelectric $NaNbO_3$ addition. The improved breakdown strengths (up to 325 kV/cm) are due to the increased interatomic bonding linked to the BO_6 octahedron and lattice network. Altogether, $NaNbO_3$-substitution and O_2 sintering synergistically resulted in excellent energy storage and pave a scheme of antiferroelectric-tailoring $BiFeO_3$-$BaTiO_3$–$NaNbO_3$ relaxor ferroelectrics for dielectric capacitor.

FS1 Bioinspiration/Green Processing & Related Technologies of Advanced Materials

Focused Session 1: Bioinspiration, Green Processing, and Related Technologies of Advanced Materials

Room: Ponce de Leon
Session Chair: Florian Bouville, ETH Zürich

3:20 PM

(ICACC-FS-015-2024) Monitoring cation-vacancy interactions to describe electrochemical properties of energy storage materials (Invited)

J. Kim*[1]

1. Stevens Institute of Technology, Chemical Engineering & Materials Science, USA

For grid storage applications, batteries made of earth-abundant elements, such as sodium (Na) and potassium (K), have emerged as alternatives to lithium (Li)-based systems. In this talk, recent findings about critical factors that dictate electrochemical properties of Na and K storage materials will be highlighted. Na storage materials with a layered oxide framework 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. I will discuss how TM chemistry affects the stability of desodiated sodium TM oxides. A highly-desodiated phase that exhibits peculiar oxygen stacking to afford alternating octahedral and prismatic Na layers, namely OP2 stacking, will be demonstrated. I will also introduce opportunities and challenges of K-ion batteries. Developing cathodes that host large K ions requires a different perspective from developing materials to store smaller ions. I will show how cation and anion mixing affect the K-K interactions and resulting electrochemical properties of $KVPO_4F$ by introducing titanium to partially substitute vanadium or oxygen to partially substitute fluorine.

3:50 PM

(ICACC-FS-016-2024) Unveiling Novel Semiconductors: Bioinspired Approaches and Advanced Characterization (Invited)

P. Gharavi*[1]

1. UCF, MSE, USA

The rapid advancement of the electronics industry has led to a growing demand for sustainable and efficient semiconductor materials. In response to this challenge, our research focuses on the development of novel bioinspired semiconductor materials with the potential to revolutionize the field. This study is an exploration of characterization techniques that offer a fresh perspective on semiconductor material. The semiconductor materials we investigate are designed to mimic natural structures and processes. This is an integral part of our research, enabling the development of materials that exhibit unique properties, such as energy efficiency. To characterize and understand these advanced semiconductor materials, we employ state-of-the-art electron microscopy techniques. High-resolution imaging, chemical analysis, and structural investigations are conducted to gain insights into the atomic-level properties of these materials. The data obtained through advanced electron microscopy is critical in unraveling the secrets of these novel materials.

4:20 PM

(ICACC-FS-017-2024) Fly Ash Bricks: An Ecofriendly Construction Material, its Properties and Uses in different Environmental Areas

M. Kaur*[1]

1. Maharishi Markandeshwar University, Mullana, Ambala, Haryana, India, Agriculture, India

The unwanted material that is produced on combustion of coal in thermal power plants to generate electricity is known as fly ash. This pozzolanic material although considered as waste material but it has advantages over the substances like cement, clay bricks etc. that has been used in making buildings, houses, restaurants etc. form decades in different parts of the world. Various researchers studied that heat emitted from cement plays a significant role in global warming enhancement. Besides this, red clay bricks consumes higher content of clay that results in removal of top soil / land degradation. Moreover, wet dumping of fly ash causes ground water pollution, soil pollution, health issues to living beings. The bricks made of fly ash are relatively lighter weight and stronger than common clay bricks. Fly ash is also a good fertilizer as it improves the efficiency of essential minerals uptake such as calcium, magnesium, iron, zinc, molybdenum, sulfur and selenium by crops. Nowadays, to overcome the environmental issues like soil infertility, solid waste, etc., fly ash has been recycled into eco-friendly products like fly ash bricks that has been used as construction material in various countries including India.

FS4 Ceramic/Carbon Reinforced Polymers

Focused Session 4: Processing and Stress Analysis

Room: Flagler C
Session Chair: Masahito Ueda, Nihon University

3:20 PM

(ICACC-FS4-004-2024) Burst Behavior and Damage Progress Evaluation in Burst Test of CFRP Hydrogen Tank with Bonded Dome and Cylinder Structure for Fuel Cell Vehicles

S. Katsumata*[1]; M. Lee[2]; T. Ogasawara[2]; N. Hirayama[3]; K. Sakata[3]; U. Kiyoshi[4]

1. Tokyo University of Agriculture and Technology, Graduate School of Engineering, Japan
2. Tokyo University of Agriculture and Technology, Japan
3. Nihon University, Japan
4. Kanazawa Institute of Technology, Japan

Carbon fiber reinforced plastic composite (CFRP) is widely used in high-pressure hydrogen tanks for fuel cell vehicles (FCVs) because of excellent specific strength and stiffness. To promote FCVs in near future, it is essential to reduce the cost of tanks and improve productivity. We propose dome/cylinder split molded structures that are more productive than the conventional filament winding (FW) method. This structure is advantageous in greatly reducing the amount of carbon fiber used. In this study, design, prototype fabrication, and rupture tests were performed for tank structures in which multi-step butt joints were applied without using adhesives at the dome-cylinder joints. In particular, the dome-cylinder joints, which require dramatic improvement in strength reliability was focused on. In addition to the burst tests of prototype tankes, tensile tests and in-situ observations were performed on coupon specimens simulating the joint of the tanks. Failure mechanisms were investigated by evaluating the bond strength and the microscopic fracture behavior. In addition, the consistency of strength and fracture mechanism between the prototype tanks and the coupon specimens was discussed.

3:40 PM

(ICACC-FS4-005-2024) CFRP etching process to enhance the joint strength

A. Benelli*[1]; S. De La Pierre[1]; M. Ferraris[2]

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

The objective of this study is to enhance the strength of Carbon-Fiber-Reinforced Polymer (CFRP) composite joints through a surface treatment aimed at selectively removing the matrix and exposing the first layer of fibers. Rectangular specimens of CFRP with high tensile strength were subjected to varying process parameters and subsequently bonded using a polymeric adhesive. The efficacy of this process was assessed through profilometry and microscopic examination of the joints cross-section. Furthermore, the maximum shear strength of the composite joints was determined using a single lap offset mechanical test. Mechanical test results were analyzed, and a correlation between the surface etching process and the mechanical stress resistance of the composite joints was extrapolated. The effectiveness of the etching process on the mechanical resistance of the composite joints was found to depend on several factors, including the process parameters of the etching procedure and the chemical composition of the composite substrate. When the appropriate parameters are carefully optimized, a significant increase in the mechanical resistance of the joints can be achieved.

4:00 PM

(ICACC-FS4-006-2024) Preparation of polystyrene coated silica particle by ball milling process

Y. Nakashima*[1]; M. Fukushima[1]

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

Polymer coating on the powder can improve the dispersibility of the powder in organic solvents, which improves the properties of the final products, such as films and plastics. Many polymer coating methods have been reported; however, these require complex procedures and excessive energy input to the powder. In this presentation, we propose a new method that focuses on surface radicals to achieve a simple and mild polymer coating using a rotating ball milling technique. In this study, amorphous silica powder with a particle size of 400 nm was used as the starting powder. Silica particles were added to a polypropylene vessel with styrene, consisting of zirconia balls with diameters of 5 and 10 mm. After adding the desired volume of styrene between 0.5 and 5 ml into the vessel, rotating ball milling was conducted at a rotating speed of 150 rpm for various times from 6 to 48 h. Polystyrene-coated silica particles were prepared using these experimental procedures, and polystyrene was selectively formed on the silica powder surface. The reaction ratio reached approximately 50% in the 24 h mill and remained constant during further milling. The most suitable volume of styrene was 1 ml, which is considered to depend on the uniform distribution of styrene in the system and the ratio of surface radicals to styrene. Part of this study was supported by the Hosokawa Powder Technology Foundation.

4:20 PM

(ICACC-FS4-007-2024) Reactivity, pyrolysis, mass-loss kinetics and carbon residue of phenol-formaldehyde resins with different hexa-contents and derived CFRPs

N. Langhof*[1]; F. Wich[1]; W. Krenkel[1]; S. Schafföner[2]

1. University of Bayreuth, Ceramic Materials Engineering, Germany
2. University of Bayreuth, Chair of Ceramic Materials Engineering, Germany

Phenolic resins are important thermoset materials in applications such as MDF boards, brake pads, heat ablation materials, refractories and as C-precursors for Ceramic Matrix Composites. The cross-linking and pyrolysis mechanisms of phenolic resins are complex and part of current investigations. The most used hardener for novolak is hexamethylenetetramine ("hexa"). This work investigated the influence of the hexa-content (0-15 wt.-%) on the reactivity, pyrolysis reactions, mass-loss kinetics and residual carbon amount for phenolic resin. Furthermore, the pyrolysis/degradation of a 0/90° C-fabric reinforced CFRP was studied in a microscopic heating cell. The reactivity and viscosity of resins was studied in a rheometer. Pyrolysis reactions, mass loss kinetics and residual carbon content were investigated by using TG-DSC-FTIR-GC/MS. The obtained results show that the reactivity, viscosity and the residual carbon amount increase with increasing hexa-content. If the amount of hexa was varied, the pyrolysis reactions change significantly. As an important outcome, a suitable resin-hardener mixture for the desired application can be selected. The deeper understanding of the pyrolysis of phenolic resins and CFRPs lead to distinct time- and cost-savings for future applications in several industrial sectors.

4:40 PM

(ICACC-FS4-008-2024) Application of ply refinement technique to the variational stress analysis of composite laminates with ply discontinuities (Invited)

M. Fikry[2]; V. Vinogradov[3]; S. Ogihara*[1]

1. Tokyo University of Science, Japan
2. Tokyo University of Science, Mechanical Engineering, Japan
3. Newcastle University, United Kingdom

This work extends the applicability of a recent variational model that has been used for analyzing cracked laminates, to determine the stress state in a laminate with alternating material properties in the longitudinal direction, subject to loading. For a simpler formulation, a plane-stress problem was first assumed in this study. The laminate was subdivided in both the longitudinal (loading) and thickness directions of the specimen. Subdivision in the thickness direction works similar to the ply-refinement technique for a more accurate solution, while subdivision in the longitudinal direction is used to accommodate different material properties in several regions along a ply. To account for continuity boundary conditions between divided regions, a Lagrange multiplier strategy was used. Closed-form expressions for the energy function derived from using the strategy led to a very concise solution for determining the approximate stresses in the laminates. As an example, a laminate with ply discontinuity was analyzed, and the resultant stress distributions were then compared to the previous variational analysis model and finite element analysis (FEA). The stress distribution in all directions, i.e., axial, transverse and shear stresses, demonstrated an excellent agreement between the FEA when a higher number of ply subdivision is used.

5:10 PM

(ICACC-FS4-009-2024) An innovative contactless Method for the Control of CFRP panels using Ultrasonic Lamb Waves (Invited)

L. A. Lecointre*[1]

1. The University of Tokyo, Japan

The improvement of Non-Destructive Testing (NDT) methods efficiency for controlling Carbon Fibers Reinforced Polymers (CFRP) is an important research field, particularly for aerospace structures manufacturing. One of the most promising leads of improvement of classic Ultrasonic Testing (UT) methods which have been identified from the 1960s is the use of Lamb Waves. Lamb Waves are mechanical waves which have the ability to propagate in plate-like structures over a relatively long distance. They represent a great improvement compared to classic UT as it removes the necessity of scanning the full parts. Developing efficient and reliable methods for the control of CFRP using Lamb Wave has been an important field of research over the last years. In particular, the design of non-contact methods and of robust Signal Processing algorithm is of utmost importance. In this research, an innovative and fully non-contact method for the generation of Lamb Waves in CFRP panels has been investigated. In order to adapt this new method to NDT in industry, the generated Lamb Waves have been characterised, the detection of delamination have been investigated and several Signal Processing algorithms have been designed. The development of such method and the demonstration of its efficiency in a wide range of industrial applications might be a disruptive change in the field of NDT of Composites.

S1 Mechanical Behavior and Performance of Ceramics & Composites

SYMPOSIUM 1: Mechanical testing and characterization of ceramics

Room: Coquina E
Session Chairs: Meelad Ranaiefar, NASA Glenn Research Center; Zachary Tuchfeld, NASA

3:30 PM

(ICACC-S1-059-2024) Multi analytical techniques of anthropoid wooden coffin from late period Egypt (Invited)

A. Mohammed*[1]

1. Fayoum University, Conservation and Restoration Department, Egypt

The present study aims to identify the pigments, ground layer, and previous restoration materials used in a polychrome wooden coffin using multi-analytical techniques. It utilized optical mi-croscopy, technical imaging, scanning electron microscope attached to X-ray dispersion unit, X-ray diffraction, Raman spectroscopy, and Fourier transform infrared spectroscopy. The results showed the use of yellow as goethite α-FeOOH, blue as Egyptian blue [Cuprorivaite (CaCuSi4O10)], red as haematite (α-Fe2O3), and white as calcite (CaCO3). The black pigment as carbon(C) and the binding medium in both the calcite-based plaster layers and the polychrome layers was identified as animal glue. The previous consolidation material was primal AC33.

4:00 PM

(ICACC-S1-060-2024) Transformation-Induced Plasticity of ceria-doped zirconia: In-situ testing of micro-pillars with Laue-Diffraction and Scanning Electron Microscopy (Invited)

M. Magalhaes[1]; t. Douillard[1]; S. Dassonneville[2]; H. Reveron[3]; T. Cornellius[2]; o. Thomas[2]; J. Chevalier*[1]

1. INSA Lyon, Materials Science, France
2. Aix-Marseille Université, France
3. Univ Lyon, MATEIS UMR5510, Insa de Lyon, Ceramics and Composites Group, France

Most ceramics generally fail at low strains with a much larger scatter in strength values than metals and require statistical approaches to failure. However, we recently investigated the mechanical behavior laws of ceria-doped zirconia ceramics and composites exhibiting a high degree of stress-induced transformation. They present, to some extent, mechanical behavior analogous to a metal, by displaying a significant amount of transformation-induced plasticity (TRIP), a high flaw tolerance and much less dispersion in strength data than any other ceramic. They potentially open new application avenues in situations where the advantages of ceramics were dampened by their brittle failure. They might represent a real alternative to, or combined with, strategies developped with long-fibers composites or bio-inspired ceramics. The transformation criteria of such Ce-doped zirconia are not fully described, in particular in relation to the crystal orientation. Thus, in this work, single-crystal micro-pillars were tested during in-situ loading in a Scanning Electron Microscope but also with Laue Diffraction (ESRF, Grenoble, France) in order to investigate their mechanical behavior laws, the onset of plasticity (or failure) and the conditions under which plasticity by dislocation motion or TRIP or brittle failure may occur.

4:30 PM

(ICACC-S1-062-2024) Characterization of Ceramic Materials using Air-coupled Ultrasound

A. Bodi*[1]; R. Steinhausen[2]; G. Urbanek[3]

1. SONOTEC GmbH, Non Destructive Testing, Germany
2. Non Destructive Testing, Forschungszentrum-Ultraschall, Germany
3. RHI MAGNESITA, R&D Material Testing, Austria

The stringent demands on the quality of high-performance ceramics necessitates quality control of 100% of the end products. Commonly used inspection methods are of limited suitability for this application; non-contact ultrasonic testing without couplant opens up new possibilities. Air-coupled ultrasonic testing enables not only quality control of the finished ceramics, but also in-process testing of green bodies before sintering, an innovative new application. The inspection of green bodies is not possible with standard inspection methods as they lack the necessary structural integrity. This lecture will provide an insight into the wide-reaching possibilities of air-coupled ultrasonic testing. The suitability of the process as well as its limitations will be illustrated with the help of real life examples. The detection of cavities or foreign bodies is achieved with amplitude analysis, a procedure widely used in recent years. As there was previously no established method for the evaluation of structural inhomogenities using air-coupled ultrasound, a special process has been developed in a cooperation project between RHI Magnesita, FZ-U and SONOTEC. To accelerate process automation, the influence of the air gap is compensated using a new technique which is unique in the world. The suitability of this method is demonstrated on real ceramics, provided by RHI Magnesita.

4:50 PM

(ICACC-S1-063-2024) Mechanical Characterization of Boron Nitride Nanotube - Polyvinylidene Fluoride Composite Films

J. J. Yonkauske*[1]; A. Hatfield[3]; R. Srinivasaraghavan Govindarajan[2]; C. Park[5]; S. Chu[5]; T. Xu[4]; D. Kim[1]

1. Embry-Riddle Aeronautical University, Aerospace Engineering, USA
2. Embry-Riddle Aeronautical University, USA
3. Old Dominion University, USA
4. Old Dominion University, Mechanical and Aerospace Engineering, USA
5. NASA Langley Research Center, Advanced Materials and Processing Branch, USA

Excellent structural, thermal, and piezoelectric properties of boron nitride nanotubes (BNNTs) make them attractive nanofillers for composites for applications in extreme environments. This research aims to expand knowledge of material properties of BNNT-polymer matrix composites by conducting a mechanical characterization of BNNT-polyvinylidene fluoride (PVDF) composite films. The films, of various BNNT concentrations, are cast via a dry-curing process. Samples undergo tensile testing and dynamic mechanical analysis to assess key mechanical and viscoelastic properties. Fourier-transform infrared spectroscopy (FTIR), Raman spectroscopy, and differential scanning calorimetry are used to characterize polymer chain conformation and crystallinity of the films. Thermogravimetric analysis evaluates thermal stability and degradation behavior. Scanning electron microscopy is used to examine film surface morphology and homogeneity of BNNT nanofiller dispersion. Preliminary results show increasing Young's modulus and yield stress corresponding to increasing BNNT concentration. BNNT inclusion also reduces recrystallization temperature and increases melting temperature. FTIR shows presence of β and primarily γ phase PVDF. These results suggest inclusion of BNNT in PVDF matrix improves the mechanical properties and thermal stability of the overall composite, and might influence conformation.

5:10 PM

(ICACC-S1-064-2024) The tribological performance of a novel metal-ceramic-hybrid-brake disc – A development for electric vehicles (EVs)

N. Langhof*[1]; T. Opel[1]; W. Krenkel[2]; S. Schafföner[1]

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

C/C-SiC is a friction material, known for its superior friction performances, low wear rates and fine dust emissions. Therefore, the ceramic brake disc is an environmentally friendly, life-time component with increasing annual production, but also, they are very expensive. To find a cost-efficient alternative to introduce C/C-SiC in EVs a metal-ceramic-hybrid-brake-disc approach was developed. The novel disc consists of an aluminum carrier body, where 11 circular aligned C/C-SiC friction segments (screw joint) on each side. Topic of this study was to evaluate whether the gaps between the friction segments of the metal-ceramic hybrid disc (Æ 410 mm) influence the tribological performance in comparison with a reference C/C-SiC full ceramic disc. Reference pads (“LowMet”), C/C and C/C-SiC pads (30x30x10 mm^3) were studied on dynamometer test rig with a fly wheel ($J = 96\ kgm^2$), with an initial speed of 20 m/s and braking pressures of 1-3 MPa. The COFs and the wear rates were evaluated. All pads show significantly more wear on the segmented hybrid disc, up to 20 times for the reference and 3 times for the C/C pads. The COF decreases with increasing braking pressure, except for the C/C pads. The reasons for the observed behavior were analyzed by SEM investigations (friction film resp. 3rd-body), discussed and the future potential of the hybrid approach will be shown.

S7 18th Intl Symp on Functional Nanomaterials & Thin Films for Sustainable Energy Harvesting

SYMPOSIUM 7: Synthesis, functionalization and assembly of inorganic and hybrid nanostructures

Room: Coquina B
Session Chair: Andreu Cabot, Catalonia Institute for Energy Research

3:30 PM

(ICACC-S7-031-2024) DFT study on the strain and electronic effect on the catalysts for ORR (Invited)

X. QI*[1]; Q. Xue[1]

1. Chongqing University of Technology, China

Challenges for theory are to understand the relationship between their structure and catalytic function and to provide guidelines to design new catalysts that take advantage of materials property. Density functional theory (DFT) is used to calculate the energetics of oxygen reduction reaction (ORR) on various catalysts, including the crystal, alloy, clusters and single atoms. The relationship between binding energy and d band center is very important. The effect of both geometric and electronic on the reaction free energy of ORR was also studied. It can be found that the surface strain effect plays an very important role for the metal catalysts, while the decoration on the single atom catalysts can improve their ORR performance.

4:00 PM

(ICACC-S7-032-2024) Evaluation of Hydroxypropyl cellulose With Nano Silica, Nanocellulose, Nano calcium oxide, and Nano Primal for Consolidation of Wooden Samples

A. Mohammed*[1]

1. Fayoum University, Conservation and Restoration Department, Egypt

Preservation of weak and deteriorated wood has become a significant global issue. The present study focuses on evaluating the efficiency of many Nanocomposite materials used for consolidating wooden artifacts, Nanocomposite was added to improve the Hydroxypropyl cellulose (Klucel G) properties and increase its ability to consolidate and protect the wooden Artefacts. In this study, acacia wood samples were prepared, and mud and gesso layers were applied. Finally, colored materials were applied. hematite for the red color, goethite for the yellow color, calcium carbonate for the white color, and Egyptian blue for blue colour. and artificial aging was performed on the colored samples. Aged wooden samples were consolidated by brushing. The treated samples were then subjected to artificial aging to test the efficiency of the consolidation materials. investigation using analytical methods was conducted in the evaluation process. Microscopic investigation using a USB digital microscope was used to examine the surface morphology. Color changes were then evaluated. It has been proven that the treated sample with Klucel G / calcium oxide nanocomposites 0.5% in ethyl alcohol gave the best results, suitable to use of this nanocomposite for the consolidation of wooden artifacts covered with layer and colored materials.

Friday, February 2, 2024

FS2 Advanced Materials for Thermoelectric and Thermionic Energy Conversion

Focused Session 2: Advanced Materials for Thermoelectric and Thermionic Energy Conversion

Room: Coquina C
Session Chairs: Armin Feldhoff, Leibniz University Hannover; James Hodges, Pennsylvania State University

8:30 AM

(ICACC-FS2-016-2024) Topological Thermoelectric Materials (Invited)

Q. Li*[1]

1. Brookhaven National Laboratory, USA

Recent discoveries of topological phenomena due to ultra-relativistic fermions across vastly different energy and length scales have led to a fascinating convergence between condensed matter physics, high energy physics, and quantum information technology [Nature Physics 12, 550 (2016); Nature Materials 18, 1267 (2019); Nature Materials 20, 329 (2021)]. Topological materials, such as Dirac/Weyl semimetals have a linear dispersion that leads to the electrons near the Fermi energy behaving like ultra-relativistic Dirac fermions. Many of the topological materials of current interests originated from the thermoelectric compounds well known in 1950s and 1960s. In this talk, I will present our studies on the Dirac/Weyl semimetals, with a view on thermoelectric applications in the past, present and future. Dirac linear dispersion can give rise to large Seebeck effect and Nernst effect in a magnetic field. An example is given on a recent discovery of colossal Nernst power factor and high Nernst figure-of-merit in topological semimetal $NbSb_2$ single crystals [Nat Commun 13, 7612 (2022)]. We also report on-going studies of thermoelectric properties of magnetic Weyl semimetals in both single crystals and polycrystalline samples. Combined with an ultrahigh carrier mobility, topological thermoelectric materials may be exploited for thermomagnetic refrigeration, thus open a new avenue for the solid-state heat pumping below liquid nitrogen temperature.

9:00 AM

(ICACC-FS2-017-2024) Novel ion substitution approach for high performance thermoelectric materials (Invited)

T. Katase*[1]

1. Tokyo Institute of Technology, Japan

Conventional approach to develop high performance thermometric materials is to use the aliovalent ion substitution for high electronic conductivity (σ), and also the heavy mass ion substitution for reduction of lattice thermal conductivity (κ_{Lat}). In this talk, I will introduce new ion substitution approach and mechanism for the simultaneous realization of high σ and low κ_{Lat}, using exmples of isovalent Te ion substitution of SnSe and hydride anion (H^-) substitution of $SrTiO_3$. Isovalent Te doped SnSe bulk, i.e. $Sn(Se_{1-x}Te_x)$ with x = 0.4, increased σ by three orders of magnitude and also drastically reduced κ_{Lat} to less than one-third at room temperature. The large Te ion in SnSe forms weak Sn-Te bonds and the high-density Sn vacancy, leading to high hole concentration. The weak Sn-Te bonds also reduces the phonon frequency and enhances the phonon scattering. We also demonstrated H- substitution of polycrystalline $SrTiO_3$, i.e. $SrTiO_{3-x}H_x$ with x = 0.216, largely reduced κ_{lat} to less than half and realized high σ comparable to single-crystal materials without any deterioration in electron conduction across grain boundaries. The H^- substitution forms a mixture of strong Ti-O bonds and weak Ti-H bonds and the randomly distributed Ti-(O,H) bonds strongly enhances phonon scattering. We expect these new ion substitution approach would lead to a new design route for high performance thermoelectric materials.

9:30 AM

(ICACC-FS2-018-2024) Thermoelectric property of hexagonal $BaTiO_3$ (Invited)

S. Yasui*[1]

1. Tokyo Institute of Technology, Japan

Thermoelectric materials have been widely investigated for one of the green energy candidates in our lives. Application devices consisting of Bi-Te and Pb-Te based thermoelectric materials are used owing to their superior properties (figure of merit (ZT)). However, those materials are toxic and not suitable for environmental-friendly. Through previous investigations of candidate materials, thermal conductivity would be one of very important parameters to obtain superior ZT. This parameter is basically dependent on phonon and career (electron and hole) in the structure. In this study, we focus on semiconductive hexagonal $BaTiO_3$ (h-BTO). Single crystal h-BTO was prepared by Floating-Zone (FZ) method. As a result, electrical and thermal conductivities increased with increasing oxygen vacancy, while Seebeck coefficient decreased. In total, ZT increased with increasing oxygen vacancy. To investigate crystal orientation dependence of physical properties, (001)-oriented h-BTO showed smaller thermal conductivity than (100)-oriented h-BTO one. This reason is explained by anisotropic h-$BaTiO_3$ crystal structure.

10:20 AM

(ICACC-FS2-019-2024) Relationship between durability and mechanical properties of high temperature-resistant thermoelectric modules (Invited)

R. Funahashi*[1]; T. Urata[1]; Y. Matsumura[1]; H. Murakami[1]; H. Ikenishi[1]; T. Sekine[2]

1. National Institute of Advanced Industrial Science & Technology, Japan
2. Akita Industrial Technology, Japan

In order to spread thermoelectric (TE) systems widely to solve energy and environmental problems, the long lifetime is one of the indispensable abilities of the TE modules. The mechanical properties at the junctions between the TE materials and the electrodes should be important to not only the power output but also the lifetime of the TE modules. The relationship between mechanical and TE generation properties has been investigated for the high temperature-resistant TE modules composed of the oxide and half-Heusler (HH) materials. The mechanical properties evaluated by the tensile and the shear strength of the individual TE devices composed of one piece of TE material and the electrodes depend on the preparation conditions and the compositions of the Ag-based paste as the bonding material. The degradation of the power output and the internal resistance are observed by heat cycling and vibration because of weakening of the joining strength. The accumulated fatigue caused by the dynamic stress should be paid attention to discuss the durability of the thermoelectric modules. TE generation test was carried out using a small sized biomass burner. The TE unites composed of the 16 pieces modules consisting of the oxide and HH materials. The electrical and thermal output power reaches 80 W and 11 kW from one TE unit at 1253 K of the combustion gas temperature.

10:50 AM

(ICACC-FS2-020-2024) Body heat harvesting for Type 1 diabetes (Invited)

W. Kim*[1]

1. Yonsei University, School of Mechanical Engineering, Republic of Korea

In this talk, we are going to introduce our efforts on operating wearable sensors and actuators based on body heat harvesting. We demonstrate that conventional inorganic materials can be used in wearable systems despite their bulky and rigid nature. In particular, we proposed a bracelet-like modular design of a thermoelectric module with a heat sink integrated with Li-S battery for body heat harvesting. This continuously produces power up to 378 μW, operating a commercial glucose sensor (64 μW) and storing the remainder in the Li-S batteries [1]. In addition, we show our recent demonstration that chargeless power operates continuous monitoring of glucose level through a sensor and insulin injection when necessary with a designed actuator for targeting type I diabetes patients [2]. A battery can be charged through body heat harvesting. For personal thermoregulation, we propose a mat-like flexible thermoelectric system based on rigid inorganic bulk materials. Using portable batteries as power sources, the refrigerated skin temperature was lowered by several degrees which is adequate for humans to perceive coldness, according to theoretical analysis. These show potential for wearable refrigeration and body heat harvesting. [1] J. Kim, S. Khan, P. Wu, S. Park, H. Park, C. Yu and W. Kim, Nano Energy, 79, 105419, 2021. [2] J. Kim, S. Khan, E. Kim, H. Kil, J. Park, J. Yoon and W. Kim, Nano Energy, 113, 108553, 2023.

11:20 AM

(ICACC-FS2-021-2024) Thermoelectric Materials for High Power Density Cooling Applications (Invited)

A. Nozariasbmarz*[1]

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

Future advancements in high-power electronics require robust thermal management methodology. A thermoelectric cooler (TEC) is an ideal solution to address this thermal management problem. The solid-state TEC provides a lightweight, high coefficient of performance (COP), high power density, and high reliability. In this talk, we will provide the design of TECs for electronics cooling using a computational model and experimental validation. Materials parameters are essential to manipulate the scattering processes within materials to decouple the transport of phonons and electrons. Besides, key device parameters such as device thickness, leg density, and contact resistance were studied to understand their influence on the performance of TECs. Our results show that it is possible to achieve high cooling power density by enhancing the TE power factor and optimizing the TE leg height and packing density. Scaling of TECs and contact resistance reduction provide ultra-high cooling power density.

11:50 AM

(ICACC-FS2-022-2024) High performance thermoelectric devices towards compact and local cooling applications

S. Shin*[1]

1. National University of Singapore, Department of Mechanical Engineering, Singapore

Albeit of the rapidly growing needs of personalized and compact thermal regulation, today's thermoelectric device has persisted with a unified design consisting of arrays of straight thermopiles, electrodes, and ceramic packaging. It has not been long ago when researchers have started looking into the thermoelectric cooling applications, compared to the long history of material-level development. In this talk, I will introduce the structural impact to achieve high thermoelectric performance for cooling and new strategies to enhance the cooling performance while maintaining compact device designs.

S10 Modeling and Design of Ceramics and Composites

SYMPOSIUM 10: Modeling and design of ceramics and composites

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

8:30 AM

(ICACC-S10-022-2024) Understanding the role of grain boundaries in polycrystalline ceramics (Invited)

K. K. Ghuman*[1]

1. Institut National de la Recherche Scientifique, Énergie Matériaux Télécommunications, Canada

Addressing the critical challenges posed by climate change hinges on our ability to identify champion materials that can be used to construct sustainable, and environmentally friendly devices. With this aim in mind, my research leverages leading-edge computational techniques and a multidisciplinary approach to understanding the intricate behaviors exhibited by complex polycrystalline materials utilized in fuel cells and batteries, subsequently optimizing them for the development of next-generation devices. Speciifcally, in this presentation, I will delve into our recent findings concerning the microstructural, ionic, and electronic behaviors observed at the grain boundaries and interfaces present in the commonly used ceramics. By integrating the classical and quantum simulations, this work prepared realistic models of the working-class materials and provided the long-sought explanation for the experimentally observed phenomenon. Additionally, in this talk, I will discuss some of the challenges in identifying the overall impact of all the microstructural defects present in polycrystalline materials and the role that machine-learning tools can play in resolving them.

9:00 AM

(ICACC-S10-023-2024) Continuous phase-field simulation of powder compaction and subsequent sintering

T. Takaki*[1]; A. Nakazawa[2]; S. Sakane[1]

1. Kyoto Institute of Technology, Faculty of Mechanical Engineering, Japan
2. Kyoto Institute of Technology, Graduate School of Science and Technology, Japan

The properties of sintered products are greatly affected by the green body produced by powder compaction and the conditions during sintering. In this study, a continuous simulation method for powder compaction and subsequent sintering was developed using the phase-field method. Here, green compacts were fabricated through uniaxial compression, and then sintered under a constant temperature. Both simulations were performed on a large-scale with parallel computations using multiple graphics processing units. Through the simulations, the sintering behavior of the powder aggregates as a function of compaction was investigated. This presentation is based on results obtained from a project, JPNP22005, commissioned by the New Energy and Industrial Technology Development Organization (NEDO).

9:20 AM

(ICACC-S10-024-2024) First-principles study on the surface stability and water adsorption behavior of X_2-RE_2SiO_5 (RE=Lu, Yb, Tm, Er, Ho, Dy, Tb)

J. Wang*[1]; M. Liu[1]; J. Wang[1]

1. Institute of Metal Research, Chinese Academy and Sciences, Advanced Ceramics and Composites Division, China

X2-RE_2SiO_5 has attracted extensive attentions as the top coating material in environmental barrier coatings (EBC), due to its good corrosion resistance and phase stability at high temperature. Resistance to water vapor corrosion in combustion environment is the critical protection function of EBC. However, the mechanism of water vapor corrosion of RE_2SiO_5 is still unclear. Investigation of the interaction between RE_2SiO_5 and water molecules at the atomic scale is essential for understanding the water vapor corrosion process at high temperature. In this work, the surface stability and water molecular adsorption behavior of X2- RE_2SiO_5 (RE= Lu, Yb, Tm, Er, Ho, Dy, Tb) were studied by first principles method. Based on the BFDH rule and the analysis of structural characteristics, 13 RE_2SiO_5 surface configurations covering 4 types of low index surfaces were selected, and the stability of these 4 types of surfaces was predicted by calculating the surface energy. The adsorption sites and adsorption energies of water molecules on the stable surfaces are then compared. The results of this work show that the active O atom on the surface of RE_2SiO_5 is the key factor of its interaction with water molecules, which provides an important theoretical basis for establishing the mechanism of water vapor corrosion resistance.

9:40 AM

(ICACC-S10-025-2024) Revisiting the SOVS model and Parameterization to Alumina (Invited)

E. Hernandez*[1]; M. C. Guziewski[1]; M. C. Golt[1]; Z. Wilson[1]

1. DEVCOM Army Research Laboratory, Sciences of Extreme Materials, USA

The Skorohod and Olevsky for viscous sintering (SOVS) model is a continuum mechanics model that simulates shrinkage and densification during the sintering process based on the materials viscous flow. It has been successfully used to simulate sintering of ZnO under free and forge conditions, as well as sintering of bilayered plate parts. While the SOVS model has been shown to accurately capture complex evolution phenomena, it employs multiple numerical parameters that could influence evolution. In this work, we perform a brief sensitivity analysis to better understand the influence of these parameters. An implicit implementation of the SOVS model is used in this study, which has been shown to improve accuracy with a marginal increase in computational cost. Lastly, parameterization of the SOVS model for Al_2O_3 is performed. The model is compared to experimental master sintering curve data.

10:30 AM

(ICACC-S10-026-2024) Direct computation of effective composite properties from non-periodic images (Invited)

G. L. Vignoles*[1]; B. Dubroca[1]; G. Mangeon[1]

1. University Bordeaux, LCTS - Lab for ThermStructural Composites, France

Computing effective properties of composite materials is at the heart of any materials design strategy. Here, not only the volume amount of the phases, but also their spatial arrangement, have to be considered. This has motivated countless studies on image-based computations, like “homogenization” or “volume averaging” techniques. Actually,

many of these techniques bear the limitation of requiring computations in periodic unit cells instead of statistically representative unit cells. This shortcoming becomes evident when working with real images (2D or 3D) of actual composites, which are not periodic. In this presentation, we discuss, on the basis of the specific case of Ceramic-Matrix Composites preforms receiving increasing amounts of matrix by the Microwave-assisted Thermal-Gradient Chemical Vapor Infiltration process, several techniques for the direct computation of effective properties from micrographs and tomographs of the raw or partly infiltrated preforms. Principles, implementations and results of these techniques will be given. Part of this work has been funded by the European Commission in the frame of the Horizon 2020 project "CEM-WAVE" under Grant Agreement no. 958170.

11:00 AM

(ICACC-S10-027-2024) Functionalized 3D fiber reinforcements for all-oxide CMC: Modelling of mechanical characteristics using the NASA Multiscale Analysis Tool

F. Jung*[1]; B. A. Bednarcyk[2]; T. Markus[1]; M. R. Welsh[1]; T. Gries[1]

1. RWTH Aachen University, Institut für Textiltechnik, Germany
2. NASA Glenn Research Center, USA

Compared to metallic turbine materials, oxide ceramics have a higher oxidation stability and temperature resistance. However, in order to make these materials suitable for turbine applications, it is necessary to reinforce the ceramics with complex continuous fiber reinforcements, thereby significantly increasing the damage tolerance of the composite. The reinforcement architecture applied poses a major challenge for the production of the CMC using colloidal manufacturing routes. Therefore, a novel 3D-braided reinforcement architecture was developed at RWTH Aachen University's Institut für Textiltechnik to reduce the filtering effect of dense textiles and allow the production of ceramic composites using a pressure slip casting route. To evaluate the performance of the braided structure, a micromechanical model using the NASA Multiscale Analysis Tool (NASMAT) will be created, which can predict the equivalent properties of the composite structure and perform progressive damage modeling. These equivalent properties and damage accumulation can be coupled with an FEM model at the component level to obtain an overall structural response under load.

S14 Crystalline Materials for Electrical Optical and Medical Applications

SYMPOSIUM 14: Scintillator materials

Room: Coquina H
Session Chairs: Kenji Toda, Niigata University; Mariola Ramirez, Universidad Autonoma de Madrid

8:30 AM

(ICACC-S14-018-2024) Synthesis of Scintillation Glasses and Glass Ceramics with Hf by Using Floating Zone Furnace (Invited)

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2. Nara Institute of Science and Technology, Japan

One of the ways to measure ionizing radiation is achieved by the process of converting high-energy radiation into a large number of photons. To convert the ionizing radiation to photons, phosphors called a scintillator have been used. Recently, glasses have become attractive materials owing to their prominent advantages, such as low cost and easy formability, and these advantages are preferable for manufacturing scintillators. However, glass scintillators suffer from a rather low energy transfer efficiency under ionizing radiation irradiation, which results in a low light yield compared with single crystalline scintillators. Furthermore, glasses generally have a low density, which causes a reduction in X-ray absorption efficiency than single crystals. In this study, to achieve the development of the glass scintillator which has a high sensitivity for X-rays and light yield, we focused on HfO_2–Al_2O_3–SiO_2 glass. Hf is the source of micro-crystal formation in the glass and provides crystallized glass through appropriate heat treatment. Crystallization of glass may increase the probability that ionized electrons and holes produced by radiation will reach the luminescent center ions. Therefore, Hf is a suitable element for both heavy elementalization and crystallization of glass. In this presentation, the physical, optical, and scintillation properties of the glass will be presented.

9:00 AM

(ICACC-S14-019-2024) Rare-earth-ions-doped LiF/CaF_2 eutectic composites for radiation dosimetry and thermal neutron detection (Invited)

N. Kawaguchi*[1]; H. Kimura[2]; T. Kato[1]; D. Nakauchi[1]; T. Yanagida[1]

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Radiation detection and dosimetry are one of the important applications of inorganic luminescent materials. Scintillators immediately emit light under radiation excitation and are used for radiation detection combining with photodetectors. Thermoluminescence, optically-stimulated luminescence, and radiophotoluminescence materials can show emissions after radiation irradiation, with intensities that depend on the cumulative radiation doses. The searches for new materials have been going on for decades and continues today. Single crystals, sintered ceramics, and glasses are widely used for such applications. We are interested in eutectic luminescent materials. Eutectics are phase-separated composites obtained by simple melt-solidification methods and tend to have lower melting points than their constituent compounds. Therefore, it can be expected that eutectics show the lower production cost than single crystals, sintered ceramics, and glasses. In this presentation, we will show scintillation and dosimetric properties of rare-earth-ions-doped LiF/CaF_2 eutectic composites which were obtained by melting at 850 °C and solidifying.

9:30 AM

(ICACC-S14-020-2024) Development of heavy crystal scintillators for X- and gamma-ray detectors (Invited)

T. Yanagida*[1]; T. Kato[1]; D. Nakauchi[1]; N. Kawaguchi[2]

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2. Nara Institute of Science and Technology, Graduate School of Materials Science, Japan

Scintillators are one of the luminescent materials, and have a function to convert a quantum of ionizing radiation to thousands of low energy photons immediately. When the target of detection is high energy photons such as X- and gamma-rays, heavy materials are preferable for scintillators since the detection efficiency against high energy photons depends on density and effective atomic number. In the conference, some recent results of development of heavy scintillators mainly containing Lu, Hf, Ta and Tl are introduced. These materials were synthesized by some single crystal growth techniques such as the floating zone and bridgeman method. After the synthesis, they were examined on optical (photoluminescence excitation and emission spectra and decay curve) and scintillation (radioluminescence spectrum, decay curve, afterglow and pulse height) properties. These results will be presented and discussed.

10:20 AM

(ICACC-S14-021-2024) Material design of fast scintillators using CsCl-based compounds exhibiting Auger-free luminescence (Invited)

M. Koshimizu*[1]

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Auger-free luminescence (AFL) is caused by electronic transition from valence band to the outermost core hole levels and has long been used as fast scintillation process in BaF_2. The AFL wavelength of BaF_2 is around 200 nm, which is not appropriate for generic photon detectors such as photomultiplier tubes or photodiodes. On the contrary, CsCl exhibits AFL at longer wavelength than that of BaF_2 owing to the smaller gap between the valence band and the outermost core levels, which is advantageous for scintillators. Therefore, in this study, fast scintillators were developed using AFL of CsCl-based compounds. From a viewpoint of the basic process, AFL competes with Auger process, in which the energy of the electronic transition from valence band to the outermost core hole levels is deposited to another electron in the valence band. If the energy is smaller than the band-gap energy, Auger process does not occur. AFL is effective in insulators having such electronic structure. In the lecture, scintillation properties of CsCl-based compounds are discussed from the viewpoints of electronic structure.

10:50 AM

(ICACC-S14-022-2024) Scintillation Mechanisms in a $(Lu_{1/4}Y_{1/4}Tb_{1/4}Gd_{1/4})_3Al_5O_{12}$:$Ce^{3+}$ Single Crystal

R. Lalk*[1]; Y. Tratsiak[1]; L. Stand[1]; C. Melcher[1]; M. Zhuravleva[1]

1. University of Tennessee, Scintillation Materials Research Center, USA

Compositional engineering has led to the discovery of Ce-activated garnet scintillators of the form $A_3B_5O_{12}$ with improved light yield and scintillation kinetics, most notably in $Lu_xY_{1-x}Al_5O_{12}$ and $Gd_3Ga_xAl_{1-x}O_{12}$, where admixture of A- and B- site cations allows for band edge engineering in order to bury shallow electron traps. The multicomponent (high-entropy) approach to compositional engineering offers additional opportunities to design garnet compositions. In this work, we investigate the structure, transport, and luminescence of a $(Lu_{1/4}Y_{1/4}Tb_{1/4}Gd_{1/4})_3Al_5O_{12}$:Ce 1 at% multicomponent garnet grown by the Czochralski method. Phase purity is confirmed by X-ray diffraction. Scintillation mechanisms are investigated using photoluminescence excitation and emission, wavelength-resolved thermoluminescence, and scintillation kinetics measurements. Photoluminescence emission excited at 275 nm results in both Tb^{3+} and Ce^{3+} emission, indicating an energy transfer from the host to Ce^{3+} emission centers. Thermoluminescence shows three groups of traps released at approximately 50K, 140K, and 400K. Wavelength-resolved thermoluminescence indicates that electrons from the traps released at 50K predominantly recombine through Tb^{3+} 4f-4f centers, and this mechanism is responsible for the scintillation kinetics. Meanwhile, electrons from deeper traps recombine through Ce^{3+} centers, so afterglow is relatively fast.

11:10 AM

(ICACC-S14-023-2024) Scale-up of high-aspect-ratio pixelated transparent ceramic scintillator via additive manufacturing for x-ray imaging

J. Smith*[1]; R. Osborne[2]; T. Yee[2]; C. J. Mcnamee[2]; P. Kerr[1]; N. Cherepy[2]; Z. M. Seeley[2]; S. A. Payne[1]

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The current capabilities for field x-ray radiography using thin phosphors on standard TFT arrays could be improved by switching to the GLO (Europium-doped Gadolinium Lutetium Oxide) transparent ceramic scintillator by increasing sensitivity. A pixelated GLO array with a high aspect ratio would increase the interaction length while maintaining spatial resolution, and therefore further improve diagnostic capabilities. Using direct ink write, pixelated GLO arrays have been fabricated and scale-up efforts are underway.

Author Index

*** Denotes Presenter**

Author Index

*Denotes Presenter

Author Index

Author Index

Author Index

Author Index

*Denotes Presenter