## **The American Ceramic Society**

## **10th International Congress on Ceramics**

## **ABSTRACT BOOK**

## July 14–18, 2024 Montreal, Canada

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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
Opening Remarks and Plenary: Gaurav Sant and Eva Hemmer
Symposium 1: Aerospace Ceramics and Composites
Symposium 1: Aerospace Ceramics and Composites
Symposium 2: Electronic Ceramics and Devices
Symposium 2: Electronic Ceramics and Devices
Symposium 4: Green ceramics for clean energy and sustainability
Symposium 4: Energy Conversion Ceramics 8
Symposium 7: Novel Processing Techniques
Symposium 7: Processing of architected and porous ceramics
Symposium 11: Recent advances and the future of additive manufacturing
Symposium 11: VAT Photopolymerization
Symposium 1: Aerospace Ceramics and Composites
Symposium 1: Aerospace Ceramics and Composites
Symposium 2: Electronic Ceramics and Devices
Symposium 2: Electronic Ceramics and Devices
Symposium 4: Green ceramics for clean energy and sustainability
Symposium 4: Energy Ceramics Synthesis and Manufacturing I
Symposium 7: Novel Processing Techniques
Symposium 7: Dielectric and Induction Approaches to Sintering
Symposium 8: Nanostructured Ceramics
Symposium 8: Processing of Nanostructured Ceramics
Symposium 11: Recent advances and the future of additive manufacturing
Symposium 11: VAT Photopolymerization II

Symposium 1: Aerospace Ceramics and Composites
Symposium 1: Aerospace Ceramics and Composites
Symposium 2: Electronic Ceramics and Devices
Symposium 2: Electronic Ceramics and Devices
Symposium 4: Green ceramics for clean energy and sustainability
Symposium 4: Energy Ceramics Synthesis and Manufacturing II
Symposium 7: Novel Processing Techniques
Symposium 7: Processing Strategies for UHTCs
Symposium 11: Recent advances and the future of additive manufacturing
Symposium 11 VAT Photopolymerization III
Plenary Session
Plenary Session: Chang-Jun Bae
Symposium 1: Aerospace Ceramics and Composites
Symposium 1: Aerospace Ceramics and Composites
Symposium 2: Electronic Ceramics and Devices
Symposium 2: Electronic Ceramics and Devices
Symposium 4: Green ceramics for clean energy and sustainability
Symposium 4: Design of Energy Ceramics
Symposium 7: Novel Processing Techniques
Symposium 7: Innovations in processing and sintering
Symposium 7: Reprocessing and Purification
Symposium 8: Nanostructured Ceramics
Symposium 8: Synthesis of Nanostructured Ceramics
Symposium 11: Recent advances and the future of additive manufacturing
Symposium 11: Material Extrusion I

Symposium 1: Aerospace Ceramics and Composites
Symposium 1: Aerospace Ceramics and Composites
Symposium 2: Electronic Ceramics and Devices
Symposium 2: Electronic Ceramics and Devices
Symposium 4: Green ceramics for clean energy and sustainability
Symposium 4: Energy Ceramics and Green Processes
Symposium 7: Novel Processing Techniques
Symposium 7: Additive, near net shaping and related precursor engineering
Symposium 8: Nanostructured Ceramics
Symposium 8: Synthesis and Properties of Nanostructured Ceramics
Symposium 11: Recent advances and the future of additive manufacturing
Symposium 11: Material Extrusion II
Symposium 1: Aerospace Ceramics and Composites
Symposium 1: Aerospace Ceramics and Composites
Symposium 4: Green ceramics for clean energy and sustainability
Symposium 4: Energy Ceramics and Green Processes II
Symposium 11: Recent advances and the future of additive manufacturing
Symposium 11: Material Extrusion, Binder Jetting, and Novel Methods
Poster Session
Plenary Session
Plenary Session: Diletta Sciti
Symposium 1: Aerospace Ceramics and Composites
Symposium 1: Aerospace Ceramics and Composites
Symposium 3: Educational Trends in Ceramics and Glass
Symposium 3: Educational Trends in Ceramics and Glass

Symposium 5: Global Ceramics and Glass Industry Trends
Symposium 5: Global Ceramics and Glass Industry Trends
Symposium 6: Informatics and data analytics in ceramics and glass
Symposium 6: Informatics and data analytics in ceramics and glass
Symposium 10: Optical and Magnetic Ceramics Materials and Devices
Symposium 10: Optical and Magnetic Ceramics Materials and Devices
Symposium 1: Aerospace Ceramics and Composites
Symposium 1: Aerospace Ceramics and Composites
Symposium 5: Global Ceramics and Glass Industry Trends
Symposium 5: Global Ceramics and Glass Industry Trends
Symposium 6: Informatics and data analytics in ceramics and glass
Symposium 6: Informatics and data analytics in ceramics and glass
Symposium 9: Next Generation Bioceramics and Bioglasses
Symposium 9: Next Generation Bioceramics and Bioglasses
Symposium 10: Optical and Magnetic Ceramics Materials and Devices
Symposium 10: Optical and Magnetic Ceramics Materials and Devices
Symposium 12: Transportation and Infrastructure
Symposium 12: Transportation and Infrastructure
Plenary Session
Plenary Session: Hala Zreiqat 59
Symposium 1: Aerospace Ceramics and Composites
Symposium 1: Aerospace Ceramics and Composites
Symposium 5: Global Ceramics and Glass Industry Trends
Symposium 5: Global Ceramics and Glass Industry Trends
Symposium 9: Next Generation Bioceramics and Bioglasses
Symposium 9: Next Generation Bioceramics and Bioglasses
6 <b>10th International Congress on Ceramics</b>

### Monday, July 15, 2024

### **Plenary Session**

#### Opening Remarks and Plenary: Gaurav Sant and Eva Hemmer

Room: Montreal 1-3 Session Chairs: Lisa Rueschhoff, Air Force Research Lab; Miladin Radovic, Texas A&M University

#### 8:30 AM

## (ICC-PLE-001-2024) A Lanthanide Journey: Navigating Materials from Multimodal Imaging to Miniature Thermometers

E. Hemmer<sup>\*1</sup>

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

The remarkable optical properties of the lanthanides (Ln) make Ln-based materials ideal for applications ranging from biomedicine to optomagnetic, optoelectronic, and energy conversion technology. This is due to the unique electronic properties of the Ln<sup>3+</sup> ions allowing for upconversion, i.e., the emission of UV-visible light under excitation with near-infrared (NIR) light. Moreover, some Ln-based materials emit NIR light under NIR excitation, hence, operating fully in the so-called NIR transparency window, endowing them with potential for biomedical applications. Sodium lanthanide fluorides (NaLnF<sub>4</sub>) are our favorite materials, and we developed a microwave-assisted synthetic approach allowing crystalline phase and size control in the sub 20 nm realm. Having a fast and reliable synthetic route towards NaLnF4 nanoparticles on hand, we now explore various nanoparticle architectures and compositions with the goal to optimize their optical and magnetic properties, ultimately resulting in the design of brighter emitters, biocompatible multimodal imaging probes or nanoscale thermal sensors. This brief journey to the world of lanthanide-based materials will shed light on the microwave-assisted synthesis of core/(multi) shell Ln-based nanoparticles and highlight some examples of potential applications.

#### 9:30 AM

## (ICC-PLE-002-2024) Equatic: Scaling a seawater-based atmospheric carbon removal and hydrogen co-production platform

G. Sant\*1

1. University of California, Los Angeles, USA

The trapping of carbon dioxide (CO<sub>2</sub>) as an aqueous (bi)carbonate ion (e.g.,  $HCO_3^{-}$ ,  $CO_3^{2-}$ ) or as a mineral solid is attractive because of favorable thermodynamics, and the durability and permanence of storage. Herein, I will describe an approach to rapidly precipitate Ca- and Mg- carbonates and hydroxides from seawater to achieve large-scale, cost-effective CO<sub>2</sub> removal. The Equatic process electrolytically forces mineral carbonate precipitation from seawater – thereby consuming  $CO_2$  that is dissolved in seawater by locking it within carbonate minerals, and simultaneously producing alkaline mineral hydroxides that when dissolved in seawater enable the drawdown of atmospheric CO<sub>2</sub>. Here, 1 mol of CO<sub>2</sub> is captured per 2 mol of OH<sup>-</sup> produced by the formation of 1 mol CaCO<sub>3</sub>. Contrastingly, only 1.2 mol of OH<sup>-</sup> are required per 1 mol  $CO_2$  stored as dissolved  $HCO_3^-$  and  $CO_3^{2-}$  ions. This is because when dissolved into seawater, every mol of Mg(OH)<sub>2</sub> leads to the absorption of up to ~1.7 mol of  $\dot{CO}_2$ . In addition, I will describe the translational design, fabrication, commissioning and operations of pilot plants in Singapore and Los Angeles which demonstrate a net energy intensity (NEI) of ~1-to-1.5 MWh per tonne of atmospheric CO<sub>2</sub> removal.

### Symposium 1: Aerospace Ceramics and Composites

#### Symposium 1: Aerospace Ceramics and Composites

Room: Montreal 1-3 Session Chair: Jon Binner, University of Birmingham

#### 10:50 AM

#### (ICC-SYM1-001-2024) Fabrication and characterization of ultra-high temperature ceramics and composites for aerospace applications (Invited)

L. Rueschhoff\*1; J. Kaufman2; C. Wyckoff2; Z. D. Apostolov1; M. Cinibulk1

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

New compositions and/or structures of ultra-high temperature ceramics (UHTC) are needed to meet the increasingly demanding structural and thermal requirements on materials for extreme aerospace environments. Existing well-established UHTC compositions suffer from limited oxidation protection and/or thermal shock resistance in high heat flux conditions. Enhanced understanding and improved performance of oxidation of UHTCs is desired through combined modeling and experimental approaches. UHTC matrix composites (UHTCMCs) offer the potential to improve fracture toughness through fiber toughening mechanisms. Here, we present an overview of work on ultra-high temperature ceramics and composites, including methods utilized to improve and characterize high temperature performance. Both conventional (wet lay-up) and novel (additive manufacturing) processing techniques to achieve UHTCMCs will be presented.

#### 11:20 AM

#### (ICC-SYM1-002-2024) CMCs at GE Aerospace (Invited)

J. Weaver\*1

1. GE Aerospace Research Center, USA

For over 3 decades, GE Aerospace and the GE Aerospace Research Center have been heavily invested in the development and commercialization of CMCs for applications in turbine engines. While the best-known product is the melt infiltrated SiC/SiC in the CFM LEAP and GE9X engines, GE now commercially produces Ox/Ox, C/SiC, and SiC/SiC CMCs for a variety of applications. Challenges experienced in taking CMCs from laboratory development to fullscale industrial manufacturing, along with some of the tools that have accelerated the transition and potential future opportunities for CMCs, will be discussed.

### Symposium 2: Electronic Ceramics and Devices

#### Symposium 2: Electronic Ceramics and Devices

Room: St. Laurent 3 Session Chair: Liam Collins, Oak Ridge National Lab

#### 10:50 AM

## (ICC-SYM2-001-2024) Dislocations in oxides: impact on functional properties and mechanical reliability (Invited)

X. Fang<sup>1</sup>; A.Frisch\*1

1. Technical University Darmstadt, Department of Materials and Earth Sciences, Germany

Dislocations are one-dimensional, line defects in crystalline solids and may have been greatly underappreciated in ceramics. Contrasting the undesirable presence of dislocations in semiconductors for their detrimental role in degrading the electronic device performance, there has been an emerging interest in dislocation-

tuned functional and mechanical properties in various oxides. However, the brittle nature of most ceramics at room temperature makes it difficult to generate and control dislocations without inducing cracks. We have been tackling this challenge, now with a toolbox available to tune dislocations into a range of ceramic materials even at room temperature, providing a robust platform to study the dislocation-tuned functional and mechanical properties. Here in this talk, I will first briefly introduce the room-temperature dislocation engineering toolbox, and then highlight several examples using dislocations to tune the thermal conductivity and the plastic deformation. The proofs-of-concept will be demonstrated on SrTiO<sub>3</sub> and further extended to other oxides to showcase the general applicability. To highlight the potential in high-temperature applications, I will also present the thermal stability of room-temperature-engineered dislocations. Some open questions regarding dislocations in functional ceramics will also be discussed.

#### 11:20 AM

## (ICC-SYM2-002-2024) Controlling dislocation motion in semiconductors using an electric field *WITHDRAWN*

Y. Zou\*1

#### 1. University of Toronto, Canada

Dislocation motion, an important mechanism underlying crystal plasticity, is critical for the hardening, processing and application of a wide range of structural and functional materials. For decades, the movement of dislocations has been widely observed in crystalline solids under mechanical loading. However, the goal of manipulating dislocation motion via a non-mechanical field alone remains elusive. Here we present real-time observations of dislocation motion controlled solely by using an external electric field in single-crystalline zinc sulfide-the dislocations can move back and forth depending on the direction of the electric field. We reveal the non-stoichiometric nature of dislocation cores and determine their charge characteristics. Both negatively and positively charged dislocations are directly resolved, and their glide barriers decrease under an electric field, explaining the experimental observations. This study provides direct evidence of dislocation dynamics controlled by a non-mechanical stimulus and opens up the possibility of modulating dislocation-related properties.

#### 11:40 AM

#### (ICC-SYM2-003-2024) Nanoindentation studies of semiconductors: Pop-in or not pop-in *WITHDRAWN*

#### Y. Zou\*1

1. University of Toronto, Canada

In this presentation, we present the effect of light illumination on the pop-in phenomenon on ZnS and GaAs. We reveal the fundamental influence about the influence on the light and doping on the dislocation movement on ZnS and GaAg.

### Symposium 4: Green ceramics for clean energy and sustainability

#### Symposium 4: Energy Conversion Ceramics

Room: Montreal 6 Session Chair: Ricardo Castro, Lehigh University

#### 10:50 AM

## (ICC-SYM4-002-2024) Green oxide and chalcogenide semiconductors for renewable energy conversion (Invited)

- O. K. Varghese\*2; L. Schaffer2; D. Waligo2; M. Paulose1
- 1. University of Houston, Department of Physics, USA
- 2. University of Houston, Department of Physics and Texas Center for Superconductivity, USA

The semiconductors formed by combining Group 6 elements with transition and post transition metals are of high relevance to solar energy conversion devices, primarily because of their potentials for light absorption, charge carrier transport or both. Many oxides and chalcogenides in this class are abundant, economic and environmentally friendly. Nevertheless, majority of such materials have not yet become key components of commercial devices for solar power or fuel generation due to their limitations in converting light energy to useful charge carriers. These materials in the zero, one and twodimensional geometries exhibit unique functionalities capable of taking device performance level close to or even beyond the classical limits. We have developed novel oxide and chalcogenide semiconductors using scalable environmentally benign methods for fabricating efficient solar cells and solar photoelectrochemcial cells for fuel generation. Both experimental and simulation approaches have been used to reveal their unique properties. In this presentation, the fabrication methods, exceptional properties and device performance of these oxide and chalcogenide semiconductors are discussed.

#### 11:20 AM

#### (ICC-SYM4-003-2024) Overcoming scale-up challenges for nanostructured photoelectrodes via one-step interface engineering (Invited)

F. L. de Souza<sup>\*1</sup>

1. Brazilian Center for Research in Energy and Materials, Brazilian Nanotechnology National Laboratory, Brazil

Green hydrogen has emerged as a central player in the net-zero economy, with scientists and industrial leaders jumping on the global race to develop high-volume and economical production strategies. Here, we introduce an innovative cost-effective method to manufacture homogeneous metal hematite-based photoelectrodes with areas as large as 156 cm<sup>2</sup>. The method entails the spin- and dip-coating of a dilute polymeric solution containing cationic precursors to fabricate few-grains-thick polycrystalline photoanodes on FTO substrates upon annealing. Based on the theoretical framework regarding dopant designing to mitigate interfacial recombination and bulkpolaronic effects, we introduced Hafnium (Hf<sup>4+</sup>) as a dopant in the polymeric precursor to deliver homogenous photocurrents of >4mA.cm<sup>2</sup> at 1.23V across films as large as 15.75 cm<sup>2</sup>. The Hf addition to the hematite polymeric precursor solution can significantly enhance the connection between the hematite and the FTO substrate which enhances the separation and catalytic efficiency in large areas. Hf addition also decreases the hematite resistivity, probably associated with the reduction of the energy barrier in the grain boundaries. The chemical solution method can represent a step forward in green hydrogen production via photoelectrochemical devices.

### Symposium 7: Novel Processing Techniques

# Symposium 7: Processing of architected and porous ceramics

Room: Montreal 8

Session Chairs: Paolo Colombo, University of Padova; Kathy Lu, Virginia Tech

#### 10:50 AM

(ICC-SYM7-001-2024) Extending the Reach of Porous Ceramics (Invited)

K. Faber\*1

1. California Institute of Technology, USA

In spite of the deleterious role of pores on the mechanical properties of ceramic materials, porosity plays an important, even critical, role in expanding the functionality of ceramic systems. In addition to lowering density, pores have had an impact on bone tissue engineering, membranes, electrochemical cells, and catalysts. The work described here will provide examples of how freeze casting, a straightforward processing technique for porous solids, can be used to move ceramics into new areas of study and utility. The method offers tailorability of pore size, morphology, and fraction. The first Illustration will describe shape memory and superelastic zirconia-based ceramics freeze cast to result in polycrystalline honeycombs which thwart phase transformation-related fracture. A second example will focus on functionalization of pore walls in silicon oxycarbide to create mixed-matrix membranes. The functionalized membranes are of interest in the purification of therapeutic antibodies. Finally, freeze-cast synthetic cryolite, Na<sub>3</sub>AlF<sub>6</sub> can serve as a biocompatible transparent soil in water for the colonization of bacteria, particularly those of interest to sustainability research.

#### 11:20 AM

#### (ICC-SYM7-002-2024) Processing Techniques of Reticulated Porous Ceramics for Environmental and Military applications

J. Ha\*1; H. Lee1; J. Lee1; I. Song1

1. Korea Institute of Materials Science, Republic of Korea

There has been growing interest in porous ceramics in many research areas given their superior thermal and chemical resistance capabilities, unlike porous metals and porous polymers. Among the various types of porous ceramics, reticulated porous ceramics can offer significant industrial potential due to the low density and high permeability of these materials. However, environmental (for filtration) and military applications (for radar absorption) are somewhat rare owing to the rather low compressive strength of reticulated porous ceramics compared to other types of porous ceramics. Therefore, the aim of this study is to determine how to obtain high compressive strength in reticulated porous ceramics by optimizing the process conditions of the solid loading level and the particle size and by using additives in a ceramic slurry sample. The characteristics investigated include the pore characteristics (pore density, pore size and pore structure), the sintering behavior (linear shrinkage), the mechanical properties (compressive strength), and the dielectric properties (dielectric breakdown strength).

#### 11:40 AM

## (ICC-SYM7-003-2024) UV curing assisted drop-casting as a novel technique for producing ceramic microbeads

R. A. Zurowski<sup>\*1</sup>; P. Falkowski<sup>1</sup>; A. M. Wieclaw-Midor<sup>1</sup>; P. Wiecinski<sup>1</sup>; J. Tanska<sup>1</sup>; W. Bulejak<sup>1</sup>; K. Korycka<sup>1</sup>; P. Wiecinska<sup>1</sup>

1. Warsaw University of Technology, Faculty of Chemistry, Poland

The aim of the research was to obtain ceramic microbeads via novel shaping technique - UV curing assisted drop-casting. In this method, the hydrophilic and photocurable ceramic suspensions droplets are exposed to UV irradiation while falling in a hydrophobic non-solvent. Ceramic beads are widely used, i.e., as grinding media in the ultrafine grinding of non-metallic minerals. However, the production of a controlled, repeatable, and cost-effective manner of elements with perfect sphericity and uniform size distribution is one of the significant technological challenges. Moreover, the possibility of applying the final machining is very limited. The proposed method seems to meet the expectations in relation to the aspects mentioned above. The authors present results regarding the optimization of the photocurable ceramic suspensions composition. The influence of the amount and type of dispersants, monomers and photoinitiators on the rheological properties was determined. Numerous rheological tests allowed to develop ceramic suspensions with appropriate properties: high water to monomer ratio, high solid content and low viscosity. The authors also focused on selecting the shaping process parameters to obtain ceramic microbeads with no microstructural defects. Acknowledgements: Research was funded by Warsaw University of Technology - Excellence Initiative: Research University (IDUB) programme.

#### 12:00 PM

#### (ICC-SYM7-004-2024) The influence of precursor molar ratio on aluminium phosphates formed by dehydration and its impact on high temperature stability

E. Valenzuela-Heeger\*<sup>1</sup>; J. Binner<sup>2</sup>

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

This talk investigates the high-temperature stability of aluminium phosphates synthesized with varying precursor molar ratios for use as binders within ceramic slurries. Several studies have previously been conducted on both experimental and theoretical product formation of different precursor molar ratios; however, there is substantial variances across the results. This research aims to define the differences in product formation caused by reaction stoichiometry, followed by an assessment of product thermal stability up to 1500°C via STA. The change in precursor molar ratio demonstrated an impact on product formation over the course of the reaction, including a change in detected reaction products and bond types studied by XRD and FTIR. The final products were made into hydrogels, and clear variances in thermal stability above 600°C were observed.

# Symposium 11: Recent advances and the future of additive manufacturing

### Symposium 11: VAT Photopolymerization I

Room: Montreal 7 Session Chair: Rodney Trice, Purdue University

#### 10:50 AM

(ICC-SYM11-001-2024) Digital light processing 3D printing and post-sintering characterization of silicon nitride (Invited) S. Sobhani<sup>\*1</sup>

1. Cornell University, Mechanical and Aerospace Engineering, USA

Our research group is dedicated to advancing the field of additively manufactured ceramics and architected structures, with a specific focus on their application in high-temperature thermofluidic systems. In this presentation, I will delve into our recent work in 3D printing of silicon nitride using digital light processing (DLP) technology. Mechanical strength and thermal tests were performed to determine suitability of this printing methodology for aerospace systems, specifically motivated by thrusters for small satellite propulsion. Debinding and pressureless sintering protocols were developed using thermogravimetric analysis and density characterization. Furthermore, the impacts of printing methodology and thermal processing on  $\beta$ -phase

9

transition and microstructure were characterized via X-ray diffraction and scanning electron microscopy. Ceramic slurry aging was examined via Fourier transform infrared spectroscopy, and its influence on final part strength was quantified. Our findings offer a promising avenue for the development of high-performance 3D printed ceramic components in aerospace systems.

#### 11:20 AM

## (ICC-SYM11-002-2024) Lithography-based additive manufacturing of co-printed and functional materials (Invited)

S. M. Allan\*1; J. Homa2; M. Schwentenwein2; S. Nohut2

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

Additive manufacturing using lithographic techniques for monolithic advanced ceramics and metals has been well demonstrated for a wide range of structural and functional ceramics. These processes involve printing a green-body using a slurry comprising ceramic powder in a photo-curable resin system as binder. Parts are printed via selective light-induced curing of the slurry. The parts are then thermally debinded and sintered to yield final parts. Many realworld applications require components made of more than one material to leverage the functionality of different bonded materials, such as electrically or thermally conductive pathways, variations in optically transparency, or tailored residual stress states to increase strength, or ensure a part fails in a specific way. Advancement in co-printing of ceramics and metals will be presented, along with discussion of the post-processing challenges that arise from subsequent co-firing and sintering.

# Symposium 1: Aerospace Ceramics and Composites

### Symposium 1: Aerospace Ceramics and Composites

Room: Montreal 1-3

Session Chair: Valerie Wiesner, NASA Langley Research Center

#### 1:30 PM

#### (ICC-SYM1-004-2024) Microstructural Evolution in a One-Directional Phase Transforming Ceramic Composite (Invited)

M. Large1; C. Stotts2; B. Taylor3; J. Rosales3; C. R. Weinberger2;

G. B. Thompson\*1

- 1. University of Alabama, USA
- 2. Colorado State University, Department of Mechanical Engineering, USA
- 3. NASA Marshall Space Flight Center, USA

The brittle properties of ceramics can lead to catastrophic failures from cracking. This can be overcome through the incorporation of metal phases; however, the metal's low melting temperature inhibits the composite to achieve higher temperature operations. In a condition where survivability from low temperature loading to continuous high temperature operation is required, we describe a one-directional phase transformation processing route to form a single-phase, ultrahigh melting temperature ceramic that is demonstrated through a laminate Zr/ZrC composite. Upon annealing, carbon readily diffuses into the metal ultimately forming single, substoichiometric carbide grains but whose granular structure differs from the starting carbide because of the reaction pathways that lead to nucleation and growth in the metal layers. As carbon depletes from the initial carbide, lattice contraction occurs facilitating grain boundary decohesion and other defects from the elastic strains attempting to retain a fully dense microstructure. While the extent of decohesion was readily evident by the metal-carbide layer ratios in sputter deposited films, it was less ratio evident in carbide powder-metal foil composites. Finally, the carbide powder-metal foil reveals the diffusivity differences of carbon in the transforming metal evident by thin metal Zr lathes along the carbide's rocksalt {111} planes.

#### 2:00 PM (ICC-SYM1-006-2024) Laser assisted joining of SiC/SiC composites

S. De La Pierre<sup>\*1</sup>; D. Basile<sup>2</sup>; M. De Maddis<sup>2</sup>; M. Ferraris<sup>1</sup>

- 1. Politecnico di Torino, DISAT, Italy
- 2. Politecnico di Torino, DIGEP, Italy

Ceramic matrix composites (CMCs), and among them silicon carbide fiber-reinforced silicon carbide matrix (SiC/SiC) composites, are currently under development for high-performance industrial applications such as aerospace and nuclear (both fusion and fission), due to their high-temperature and harsh environment resistance properties. One critical issue for SiC/SiC composites is the possibility of joining them to obtain complex structures, which can fulfill the application requirements. Several technologies and materials have been proposed for joining SiC/SiC components. Among them, laser assisted joining seems to give promising results as a pressure-less, localized heating joining technology, suitable for SiC/SiC composites operating in a high-temperature environment. The present work reports on preliminary results on laser assisted joining of SiC/SiC composites; several joining materials have been used to join SiC/SiC by a diode laser, operating in the infrared (1020-1060 nm). The morphology and micro-structure of joined SiC/SiC plates and tubes will be discussed. Acknowledgments: Part of the research leading to these results has received funding from the European Union's Horizon 2020 research and innovation program under grant agreement No 101059511 - Project SCORPION

#### 2:20 PM

# (ICC-SYM1-007-2024) Effect of BN Interphase Formed by EPD method on Mechanical Behavior of SiC<sub>f</sub>/SiC Composites Fabricated by PIP method

D. Sakakibara\*1; A. Gubarevich2; M. Kotani4; K. Yoshida3

- 1. Tokyo Institute of Technology, Materials Science and Engineering, Japan
- 2. Tokyo Institute of Technology, Japan
- 3. Tokyo Institute of Technology, Laboratory for Advanced Nuclear Energy, Institute of Innovative Research, Japan
- 4. Japan Aerospace Exploration Agency, Japan

Silicon carbide fiber-reinforced silicon carbide matrix (SiC<sub>f</sub>/SiC) composites consist of SiC fibers, SiC matrix, and interphase between them. The mechanical behavior of SiC<sub>i</sub>/SiC composites is greatly influenced by interfacial properties. The objective of this study is to investigate the influence of properties of boron nitride (BN) interphase formed on SiC fibers by electrophoretic deposition (EPD) method on the mechanical behavior of SiC<sub>f</sub>/SiC composites. EPD was conducted using aqueous suspensions of BN submicron particles. By varying BN suspension concentration, BN coatings with different thickness were formed on the surface of SiC fibers. Then polymer impregnation and pyrolysis (PIP) method was applied to form the matrix of SiC<sub>f</sub>/SiC composites by different procedures, resulting in composite samples with different densities. As a result of the EPD coating using suspension of 0.5 and 1.0 wt% BN concentration, average thickness of 210 nm and 360 nm BN layers were formed on SiC fibers. The results of three-point bending tests showed that the bending strength of the sample depended on the thickness of interphase and the density of the sample. Sample with higher density matrix and thicker BN interphase showed the highest bending strength of 418 MPa. These findings suggest that controlling interfacial properties can enhance the strength of composites.

### Symposium 2: Electronic Ceramics and Devices

#### Symposium 2: Electronic Ceramics and Devices

Room: St. Laurent 3

Session Chair: Alexander Frisch, Karlsruher Institute of Technology

#### 1:30 PM

## (ICC-SYM2-004-2024) Nanoscale Insights into Electromechanical Phenomena via Scanning Probe Microscopy (Invited)

L. Collins<sup>\*1</sup>

1. Oak Ridge National Lab, USA

The conversion of electrical to mechanical energy and vice versa is crucial for various technologies like piezoelectric sensors, energy harvesters, and ferroelectric memories. Despite its significance and widespread applications, challenges persist in probing electromechanical (EM) phenomena at the nanoscale. Scanning probe microscopy (SPM) excels in measuring local volume changes at the picometer scale with tens of nanometers lateral resolution, making it ideal for studying EM processes. However, superficial interpretations of certain SPM measurements, like Piezoresponse force microscopy (PFM), can lead to misleading contrasts and misinterpretations of material physics. Domain contrast and signs of polarization switching (e.g., PFM Hysteresis loops) are often misidentified as evidence of ferroelectricity, even in non-ferroelectric materials. Drawing parallels with macroscopic ferroelectric characterization, I will highlight the risks of misinterpreting PFM data, showing how even ordinary almond nuts can display "ferroelectriclike" hysteresis loops, mimicking those attributed to ferroelectric polarization switching. Lastly, I will outline new methods for precise nanoscale EM quantification, emphasizing the need for detailed characterization of ionic and impedance behaviors in interpreting PFM data accurately.

#### 2:00 PM

## (ICC-SYM2-005-2024) High-throughput calculations on oxygen vacancies and their applications" (Invited)

Y. Kumagai\*1

1. Tohoku University, Institute for Materials Research, Japan

Oxygen vacancies play significant roles in various properties of oxide materials. Therefore, insights into the oxygen vacancies can facilitate the discovery of better oxide materials. We recently conducted high-throughput calculations on oxygen vacancies in approximately 1000 oxides, analyzing their structures and formation energies. From this extensive dataset, we developed machine learning regression models to predict these energies and identified superior p-type transparent conducting oxides.

#### 2:30 PM

#### (ICC-SYM2-006-2024) Design of Electro-ceramic Materials using the Combination of Machine Learning Potentials and First Principles Calculations

Y. Iwazaki\*1

1. TAIYO YUDEN CO., LTD., Materials Science Laboratory, Japan

Electronic ceramics, especially ferroelectric materials, are essential for various electronic devices due to their outstanding dielectric and piezoelectric properties. However, their complex atomic structures, including grain boundaries and electrode interfaces, pose challenges for precise analysis using conventional computational approaches. Recent advances in atomic and molecular simulations driven by machine learning (ML) techniques offer promising solutions, allowing large-scale atomic modeling and accommodating chemical reactions. Yet, these methods primarily focus on atomic forces and require first-principles calculations for accurate predictions of electrical properties. This presentation explains the integration of ML potentials and first-principles calculations for effective computational material design. This synergistic approach allows us to comprehensively model intricate structures of electronic ceramics, gaining valuable insights into their electronic behavior, dielectric characteristics, and piezoelectric properties. These approaches advance our fundamental understanding of these materials and facilitate tailored design and optimization for a broad spectrum of electronic applications, demonstrating the potential for a new approach to the electronic research and development of electro-ceramic materials.

### Symposium 4: Green ceramics for clean energy and sustainability

# Symposium 4: Energy Ceramics Synthesis and Manufacturing I

Room: Montreal 6 Session Chair: Claire Dancer, University of Warwick

#### 1:30 PM

## (ICC-SYM4-004-2024) Porous Ceramics Based Composite Phase Change Materials (Invited)

M. White\*1; J. Noel1

1. Dalhousie University, Department of Chemistry, Canada

Phase change materials (PCMs) can be used to efficiently store thermal energy for later release. Two of the main issues preventing wider PCM use are the need for containment (the phase change is generally solid to liquid), and low intrinsic thermal conductivity, especially for organic PCMs. We have addressed both issues through composite materials: freeze-cast porous ceramics impregnated with PCMs. We have shown that many different freeze-cast materials (alumina, titania, carbon black, alumina/carbon black, chitosan, carbonized chitosan) can be used as matrices for PCMs (fatty acids, PEG, paraffin, esters, sugar alcohols, salt hydrates), to prepare a wide variety of form-stable phase change materials. The porous ceramic matrix does not substantially influence the PCM melting points, but does control the fill, thermal conductivity and mechanical properties of the form-stable PCM. Of those investigated, alumina at a 40 mass% loading of PCM gave the best thermal conductivity, up to 3.2 W m<sup>-1</sup> K<sup>-1</sup>at room temperature, significantly enhanced compared with 0.24 W m<sup>-1</sup> K<sup>-1</sup> for the pure PCM, and with excellent hardness and mechanical stability after 1000 cycles. Chitosan and carbonized chitosan scaffolds gave form-stable PCMs with the highest PCM loading, > 90 mass%.

#### 2:00 PM

#### (ICC-SYM4-005-2024) Porous monolithic perovskite ceramics for "green" high-temperature hybrid sensible/thermochemical storage of solar and industrial heat

C. C. Agrafiotis<sup>\*1</sup>; M. Pein<sup>1</sup>; A. Eltayeb<sup>1</sup>; D. Vellas<sup>1</sup>; L. deOliveira<sup>1</sup>; D. Koch<sup>1</sup>; M. Roeb<sup>2</sup>; C. Sattler<sup>1</sup>

- 1. DLR German Aerospace Center, Institute of Future Fuels, Germany
- 2. DLR German Aerospace Center, Germany

Porous monolithic structures like honeycombs and reticulated porous ceramics (RPCs, known as ceramic foams) entirely made out of Ca-Mn-based perovskites are capable of cyclic, reversible, endothermic reduction-exothermic oxidation upon heating/ cooling under air accompanied by significant heat effects. Uniquely among other redox oxides, they also exhibit completely reversible thermochemical expansion/contraction during thermal cycling up to 1100°C. These features during "charging/discharging" allow for their direct hybridization with current state-of-the-art regenerative sensible heat storage/waste heat recovery systems of Concentrated Solar Power (CSP) plants or industrial hightemperature processes that consist of brick-patterned storage media with air channels, achieving much higher energy storage densities in the same heat storage unit volume. Recent research results will be presented, spanning the entire range from first-principles calculations and computational redox enthalpy contribution studies of such perovskite compositions to the demonstration of the feasibility of shaping RPCs made entirely of perovskites, their characterization and application-relevant testing. Functionalities not possible with conventional sensible-only storage systems like "thermal boosting" using the reduced oxide as a "green", non-fossil fuel will be addressed.

#### 2:20 PM

### (ICC-SYM4-007-2024) A novel solar thermal process for manufacturing of high-performance engineering surfaces

- P. Psyllaki\*1; A. Mourlas1; J. Rodriguez2; I. Cañadas Martinez2
- 1. University of West Attica, Department of Mechanical Engineering, Greece
- 2. CIEMAT-Plataforma Solar de Almería, Spain

A green, concentrated solar energy-based approach for the rational elaboration of wear resistant carbide-reinforced surface layers for use in demanding industrial environments is presented. Such composite materials are typically developed onto metallic substrates via arc welding techniques, by partially heating the base metal above its melting temperature; upon cooling hard primary carbides are precipitating, reinforcing the metallic matrix. This process employs high amounts of heat absorbed and dissipated in the material to be treated and is applied in a rather empirical and random way, a fact that often induces the appearance of microstructural flaws like pores and cracks. An alternative approach is to employ Concentrated Solar Energy as a green, high-temperature heat source, capable of controlled and focused delivery of elevated heat flux densities to solid surfaces for the in-situ elaboration of hardfacings that exhibit controlled, flawless microstructure and eventually high performance under severe operating environment. Such "solar" hardfacings have been developed applying pre-deposited TiC, WC and  $Cr_3C_2$  ceramic powders onto common steel and were extensively characterized with respect to their microstructure, hardness and wear resistance, under non-lubricated sliding conditions, simulating their in-service operation in harsh applications (e.g. rock drilling, excavation).

### Symposium 7: Novel Processing Techniques

# Symposium 7: Dielectric and Induction Approaches to Sintering

Room: Montreal 8

Session Chairs: Mark Losego, Georgia Institute of Technology; B.Reeja Jayan, Carnegie Mellon University

#### 1:30 PM

#### (ICC-SYM7-005-2024) Presenting the Case for Scaling up Microwave Sintering of Ceramics (Invited) *WITHDRAWN*

B. Jayan\*1

1. Carnegie Mellon University, USA

The first experiments demonstrating microwave sintering of ceramics took place in the 1960s. They introduced a materials processing tool with a lower environmental impact, consuming less energy than traditional furnace-based heating processes. Despite its advantages, including non-contact heating and shape flexibility, scaling up microwave sintering/processing for commercial use has been difficult. This is primarily due to challenges in managing the complex, multi-scale, multi-physics phenomena that occur when microwaves interact with materials. However, the engineering landscape is changing. Recent advancements in computational modeling, material analysis, control systems, and software are providing new solutions to these old problems. Additionally, the emergence of additive manufacturing has spurred a demand for sustainable sintering methods capable of producing intricate and precisely shaped ceramic parts. These advancements offer a promising pathway to overcoming the historical obstacles of microwave sintering. This paves the way for scaling and wider commercial adoption into a variety of sectors, including energy, healthcare, automotive, and aerospace, where there is a growing need for complex, high-performance ceramic components. My talk will chronicle these advances in the field both from my own lab and others in the Ceramics Processing community.

#### 2:00 PM

### (ICC-SYM7-006-2024) Ceramics based on $SnO_{29}$ ZnO and $Zn_2SnO_4$ prepared by conventional and microwave sintering

P. Simonova\*1; L. Kotrbova1; C. Petit2; W. Pabst1; C. Meunier2; F. Valdivieso2

- 1. University of Chemistry and Technology, Prague, Department of Glass and Ceramics, Czechia
- 2. Mines Saint-Etienne, CNRS, UMR 5307 LGF, France

Materials based on SnO<sub>2</sub>, ZnO and Zn<sub>2</sub>SnO<sub>4</sub> are due to its unique properties used in various applications such as gas sensors, varistors, electrodes, photoelectric devices, and others. In this contribution we present results on ceramics which were prepared from SnO<sub>2</sub> and ZnO and also from their mixtures to obtain Zn<sub>2</sub>SnO<sub>4</sub> phase. These ceramics were sintered from 900 to 1400 °C by conventional (CV) and microwave (MW) sintering. The ceramic microstructure was characterized by determining the phase composition, porosity, bulk density, and grain size. Apart from that, elastic properties of these ceramics were investigated at room temperature but also at high temperature to observe the conventional sintering behavior and temperature dependence of these properties. In the case of ceramics prepared from both oxides the creation of Zn<sub>2</sub>SnO<sub>4</sub> was observed above 900 °C after both CV- and MW-sintering. However, microstructural descriptors such as porosity and bulk density were significantly different depending on the sintering process. Interesting observations occurred in the case of pure SnO<sub>2</sub> ceramics, where the porosity and bulk density remained constant and no shrinkage occurred even at 1400 °C, but the elastic properties were significantly changing on samples sintered at higher temperatures. That is related to the non-densifying sintering mechanisms characteristic for this material.

#### 2:20 PM

#### (ICC-SYM7-007-2024) Electromagnetic Induction-Assisted Process for Pressureless and Additive-Free Sintering of Boron Carbide Ceramics

A. Gubarevich\*1; G. Homma1; K. Yoshida1

1. Tokyo Institute of Technology, Japan

A novel sintering technique is proposed for boron carbide ceramics, utilizing pressureless densification under high-frequency electromagnetic field. In this study, boron carbide pellets were densified using a standard induction heating solenoid coil and a graphite crucible. By varying the thickness of the graphite crucible, the energy supply balance between indirect heating via the crucible and direct induction heating of the boron carbide pellets was investigated. Results indicate that utilizing a graphite crucible with a thickness less than the characteristic skin depth of graphite leads to rapid densification of boron carbide, achieving a relative density exceeding 95% in a few minutes. Microstructural analysis revealed a fine structure with limited grain growth, indicating densification under the influence of electromagnetic forces. This study concludes that boron carbide can be sintered to nearly full density within a short duration and with low total energy consumption under high-frequency electromagnetic field.

### Symposium 8: Nanostructured Ceramics

#### Symposium 8: Processing of Nanostructured Ceramics

### Room: St. Laurent 4

Session Chairs: Ravi Kumar, Indian Institute of Technology Madras; Ricardo Castro, Lehigh University

#### 1:30 PM

#### (ICC-SYM8-001-2024) 1D Lepidocrocite Titania-based Nanomaterials, Their Diverse Morphologies and Exceptional Properties (Invited)

#### M. Barsoum\*<sup>1</sup>

1. Drexel University, Materials Science and Engineering, USA

Recently, we converted 15 binary and ternary titanium carbides, nitrides, borides, into lepidocrocite-based, 1D, sub-nanometer nanofilaments, NFs,  $\approx$  5x8 Å in cross-section by reacting them with TMAH aqueous solution at  $\approx 85$  °C range for tens of hours. One gram of our 1D material spans ≈ 600 million km! Depending on with what and the order the reaction products are washed, the 1D NFs self-assemble into loose, spaghetti-shaped fibers, ≈ 30 nm in diameter, fully inorganic gels, pseudo 2D or porous mesoscopic particles. In all cases, the fundamental building block is 1D lepidocrocite NFs,  $\approx$  3 nm long, that self-assemble into the aforementioned morphologies. At this time, we believe that our materials are the only thermodynamically stable 1D NFs in water, with important implications in photo- and chemical catalysis. The production of hydrogen for times of the order of 6 months with production rates an order of magnitude higher than P25, will be discussed. The adsorption of some cations and dyes in some cases outperform high adsorption clays. We also discovered that some common dyes sensitize the 1D NFs which allows for their degradation using only visible light. This is important in this respect because the band gap energy,  $\approx 4 \text{ eV}$ , of our 1D NFs is a record for titania-based materials due to quantum confinement.

#### 2:00 PM

## (ICC-SYM8-002-2024) High Pressure Sintering of Advanced Structural Ceramics

W. Ji\*1; Z. Fu2

- 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.

#### 2:20 PM

#### (ICC-SYM8-003-2024) Double-layered nanosheets: their preparation, lateral size fractionation, and their application to a pH-responsive material

Y. Sugahara\*1; T. Kamibe1

1. Waseda University, Japan

Double-layered nanosheets are attractive materials because they have nm-level thicknesses and confined spaces between two layers. Their preparation is possible by using a unique structure of layered potassium hexaniobate trihydrate, K4Nb6O17.3H2O, since it has two different interlayers with different reactivities which appear alternately in its stacking direction. By selective organic modification and subsequent exfoliation at one type of interlayer selectively, double-layered niobate nanosheets can be obtained. When outer surface of double-layered nanosheets was modified by using a water-cyclohexane biphasic system, phase transfer of double-layered nanosheets from an aqueous phase to an organic phase was observed, and lateral size fractionation can be achieved by multiple extraction procedures. On the other hand, after one type interlayer was modified with a pH-responsive cross-linked polymer network using atom transfer radical polymerization technique, double-layered nanosheets were obtained by exfoliation at the other type of interlayer. They had the cross-linked polymer network between two layers and were able to be converted into single-layered nanosheets via degradation of the cross-linkers by acid treatment.

#### 2:40 PM

### (ICC-SYM8-004-2024) Processing Advanced Ceramics by Suspension Plasma Spraying (Invited)

#### C. Moreau<sup>\*1</sup>

1. Concordia University, Mechanical, Industrial and Aerospace, Canada

Suspension plasma spray (SPS) is an emerging technology that has undergone extensive research and development in recent decades. SPS involves the injection of a suspension of submicron-sized particles in a liquid, typically ethanol or water, into a plasma plume to produce coatings with distinct microstructures. This innovative approach has attracted significant attention due to the new possibilities and challenges it presents. The control of the microstructure and nanostructure of SPS coatings has been thoroughly investigated. Studies have demonstrated the ability to tailor the porosity of the coating to suit specific applications, ranging from nearly dense coatings optimized for wear resistance to highly porous coatings with controlled pore sizes for the fabrication of ceramic filtration membranes. In this presentation, we aim to provide an overview of SPS technology, along with examples on how this process can be used to manufacture ceramic coatings and devices. We will explore emerging applications, such as superhydrophobic and icephobic coatings, porous filtration membranes, columnar thermal barrier coatings, wettable cathodes for aluminum electrolysis, and photocatalytic surfaces. These examples highlight the versatility and market potential of this ceramic processing technology.

#### 3:40 PM

### (ICC-SYM8-005-2024) Nanoscopic dynamics in zirconia and other oxide polycrystals under flash event (Invited)

H. Yoshida\*1; Y. Yang1; K. Nambu3; H. Masuda1; K. Morita2; T. Yamamoto4

- 1. The University of Tokyo, Materials Science and Engineering, Japan
- 2. National Institute for Materials Science (NIMS), Japan
- 3. Kyushu University, Materials Science and Engineering, Japan
- 4. Nagoya University, Materials Design Innovation Engineering, Japan

It is widely accepted that the diffusion of atoms in oxide is significantly accelerated under flash event, mainly by thermal runaway, as shown by flash sintering in zirconia and other oxide ceramics. We have recently shown from XPS analysis and photoluminescence emission spectra that non-equilibrium point defects are introduced at high concentrations in zirconia and yttria under flash events. It

has also been shown that high-temperature plastic deformation involving grain boundary sliding is athermally enhanced under flash events. Such athermally enhanced dynamics probably lead to diffusion self-joining of tetragonal zirconia bodies using flash event and to the enhancement of anelasticity in cubic zirconia treated with electric field application. Non-equilibrium point defects introduced under flash events are probably responsible for the enhanced dynamics. In this paper, our recent results on changes in nanoscopic state and dynamics in oxide ceramics induced by flash events.

#### 4:10 PM

## (ICC-SYM8-006-2024) Reactivity and processing properties of barium titanate nanoparticles in different chemical environments

K. Neuhauser<sup>\*1</sup>; H. Razouq<sup>1</sup>; E. Neige<sup>1</sup>; T. Berger<sup>1</sup>; O. Diwald<sup>1</sup>

1. Paris Lodron University Salzburg, Chemistry and Physics of Materials, Austria

BaTiO<sub>3</sub> is a perovskite material with outstanding ferroelectric properties. Hence, it is used in many electronic applications like sensors or random-access memories. Furthermore, BaTiO<sub>3</sub> is subject to promoting the photocatalytic reactivity of established photocatalysts. In this work, we investigated how different formulation approaches applied to BaTiO<sub>3</sub> nanoparticle powders that were grown by flame spray pyrolysis (FSP) affect the particle stability and the charge separation properties. As an example, processing 1-dimensional BaTiO<sub>3</sub> composite nanostructures in an aqueous environment gives rise to dramatic changes in the microstructure. This is attributed to the reaction of the barium containing species with dissolved carbon dioxide in the aqueous environment. We analysed the formation of the resulting carbonate structures with combined X-ray diffraction and electron microscopy experiments. Moreover, we compared different approaches to assess the separation of photogenerated charges inside BaTiO<sub>3</sub> structures of different dimensionality, i.e. from nanoparticles to 2D layers of sintered grains.

#### 4:30 PM

#### (ICC-SYM8-007-2024) One-Dimensional Titanium Oxide Lepidocrocite Nanofilaments: Properties and Details on their Simple and Scalable Synthesis Procedure

A. D. Walter\*1; G. R. Schwenk1; M. Barsoum1

1. Drexel University, Materials Science and Engineering, USA

One-dimensional titanium oxide lepidocrocite filaments (1DLs) behave as an inorganic polymer. As a material with a base unit of just 3x2 titanium octahedra in cross section, they exhibit confinement in two dimensions providing a fascinating mix of properties. The ease of fabrication for 1DLs is a paradigm shift in low dimensional materials. Their scalable fabrication process will be discussed in detail, including a presentation of some of the primary characterization techniques for 1DLs. Their unique polymeric, electronic, and surface properties will be discussed. Some surface area driven application results will be introduced including their self-assembly and high adsorption capacity for inorganic and organic species.

# Symposium 11: Recent advances and the future of additive manufacturing

### Symposium 11: VAT Photopolymerization II

Room: Montreal 7 Session Chair: Peigang He, Institute for Advanced Ceramics

#### 1:30 PM

#### (ICC-SYM11-004-2024) How additive manufacturing is becoming a game-changer for the production of clean hydrogen (Invited) R. Gaignon<sup>\*1</sup>

1. 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 program starting this year called HYP3D, AM is helping to improve the standards of hydrogen production with pressurised 3D printed SOEC stacks. The challenges that come from this technology lie in 3 main domains : production, storage, and supply chain. 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, and the metallic interconnects are voluminous and complex in shape. 3DCeram's SLA technology makes it possible to print thin corrugated ceramic cells, increasing the surface area by 60% which in turn enhances the performance of the cells. It's also reducing the size of the interconnects and therefore the stack as a whole. This is another example of how 3D printing is bringing to life more disruptive designs and helping to enhance system performances.

#### 2:00 PM

#### (ICC-SYM11-005-2024) DLP 3D printed Hierarchical Hybrid Ceramic Filters: Design, Fabrication, and Performance Analysis

M. Gorbar<sup>\*1</sup>; R. Kontic<sup>1</sup>; P. Marmet<sup>2</sup>; L. Holzer<sup>2</sup>; D. Penner<sup>1</sup>

- 1. ZHAW, IMPE Institute of Materials and Process Engineering, Switzerland
- 2. ZHAW, ICP Institute of Computational Physics, Switzerland

Hybrid ceramic structures, consisting of multiple layers with varying material mixtures, offer a promising avenue for efficient media-transport through the structure. This study presents an analysis of the design, fabrication, and performance of examples of such layered geometrical complex structures. The ceramic bodies exhibit a hierarchical microstructure, tailored to meet specific requirements and can be shaped in 3D utilizing a Digital Light Processing (DLP) route. The hierarchical microstructure of ceramic filter plays a crucial role in its overall performance. Through precise control of microstructural features, including pore distribution, channel arrangement, and particle size, the filter's filtration efficiency, flow rate, and selectivity can be tailored to specific requirements. This tunability allows for the optimization of the filter's performance across a wide range of applications. Experimental analysis and numerical simulations provide insights into the filter's performance under various operational conditions. Findings highlight the filtration efficiency and longevity of the hybrid ceramic filter, surpassing conventional alternatives. Additionally, the 3D printing capability of the hybrid ceramics via the DLP route presents numerous advantages. The filter can be printed in separate pieces, allowing for complex geometries and customized designs.

#### 2:20 PM

#### (ICC-SYM11-006-2024) Ceramics in the Digital Age: Precision Engineering with Photopolymerization and 3D Printing

A. Kulkarni\*1; H. Yazdani Sarvestani1; A. Sohrabi1; T. Lacelle1;

- V. Karamzadeh<sup>1</sup>; A. Shashoua<sup>1</sup>; Y. Martinez-Rubi<sup>1</sup>; M. Jakubinek<sup>1</sup>; B. Ashrafi<sup>1</sup>
- 1. National Research Council, Canada, Canada

Ceramic materials, known for their high mechanical strength and environmental stability, face challenges in structural applications due to inherent brittleness. Polymer-derived ceramics offer a solution, overcoming shape constraints in traditional processing. This paper explores the formulation of commercial silicon oxycarbides (SPR 684) for 3D printing via stereolithography (SLA) using photopolymerization. The preceramic polymer, combined with additives, allows versatile SLA printing. Pyrolysis of the printed polymer produces a ceramic shape, but issues like porosity arise. Computed tomography and compression experiments unveil the impact of formulation on crack initiation and propagation in 3D-printed architectures. This low-cost SLA method is ideal for thin features and customized structures, promising bio-inspired, architected ceramics.

#### 2:40 PM

#### (ICC-SYM11-007-2024) A Suspension-Enclosing Projectionstereolithography Process for Complex Ceramic Component Fabrication without Building Support Structures

X. Song\*1

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

In recent years, ceramic fabrication using stereolithography (SLA) has attracted a lot of interest due to relatively high accuracy and density that can be achieved in final part production. One of the main challenges in ceramic SLA is that support structures are required to build overhanging features. Since support structures have to be manually removed after fabrication, fracture tips are typically introduced at the location where a support structure was originally built. Fracture tips may decrease surface quality, introduce stress concentration and consequently increase the probability of component damage. In this research, we present a Suspension-Enclosing Projection-stereolithography process (SEPS), which employs the unique non-Newtonian rheological behavior of its high-viscosity feedstock material to self-support overhanging features without building extra support structures. Different feedstock materials are studied to identify the most suitable rheological properties required for supporting overhanging features. Several test cases were constructed to showcase the developed fabrication process.

# Symposium 1: Aerospace Ceramics and Composites

#### Symposium 1: Aerospace Ceramics and Composites Room: Montreal 1-3

Session Chair: Jared Weaver, GE Research

#### 3:30 PM

# (ICC-SYM1-008-2024) 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 ecocomposites were inoculated by Al-Nb-V-Zr master alloy and subjected to thermo-mechanical treatment (cold worked/homogenized/hot worked/aged at 450°C and 480°C 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 ecocomposites were analyzed using Optical Microscope, Scanning Electron Microscope (SEM), X-ray Diffractometer (XRD), and Energy Dispersive Spectroscopy (EDS).

#### 3:50 PM

(ICC-SYM1-009-2024) Phase and microstructure study of boron doped allylvinylhydridocarbosilane derived bulk SiBC ceramic

K. Lu<sup>\*1</sup>; R. Anand<sup>2</sup>

- 1. University of Alabama at Birminghngham, USA
- 2. Virginia Tech, USA

In this study, a commercially available precursor "SMP-750" containing allylvinylhydridocarbosilane and Si nanoparticles was blended with 5 mol% boron using borane dimethylsulfide and crosslinked followed by pyrolysis at 1200 °C to 1800 °C in argon atmosphere for the synthesis of SiBC. The pyrolyzed sample contains primarily amorphous SiC phase and Si metal nanoparticle at 1200 °C. At higher pyrolysis temperature, Si combines with free carbon present into SiC matrix and forms a reaction bonded  $\beta$ -SiC and thus reduces the free Si amount into the SiC sample. X-ray diffraction phase analysis showed that the addition of boron to SiC ceramics significantly influences the high-temperature behavior of the ceramics in terms of evolution of a nanocrystalline phase, coarsening of the separated amorphous phases, crystallization of SiC and its decomposition. Interestingly, the B-containing carbon phase does not convert into crystalline boron carbide, even upon annealing at very high temperatures and despite the high boron content. Further, scanning electron microscope evidence that the boron incorporation into SiC helps to obtain a dense, and crack-free bulk SiC ceramic.

#### 4:10 PM

## (ICC-SYM1-010-2024) Oxide-oxide Ceramic Matrix Composites based on Unidirectional Towpreg for Automated Fibre Placement

T. Nelson\*1; J. Binner2; I. M. Edmonds3

- 1. University of Birmingham, School of Metallurgy and Materials, United Kingdom
- 2. University of Birmingham, Ceramic Science & Engineering, United Kingdom
- 3. Rolls-Royce, United Kingdom

Traditional oxide-oxide ceramic matrix composite (CMC) manufacture can involve manual slurry infiltration into precut fabric sheets, followed by hand lay-up onto a mould of required shape. Automated fibre placement (AFP), currently used for production of polymer matrix composites, promises a capability for highly reproducible laminate production of complex geometries using unidirectional towpreg feedstock. This presentation will show results of a processing study of Al<sub>2</sub>O<sub>3</sub>/Al<sub>2</sub>O<sub>3</sub> CMC laminates manufactured with a commercial towpreg system. One key aspect of processing this material is the level of 'tack', or ability of the towpreg to adhere to a mould or to itself. Multiple methods for quantification of tack will be explored. This commercial towpreg system has also been used to successfully produce components of more complex geometry, using an out-of-autoclave process, that demonstrate the ability of towpreg to form components of interest for aerospace.

#### 4:30 PM

### (ICC-SYM1-011-2024) A novel evaluation method for processability of slurry-based prepregs

G. Puchas\*1; F. Lindner1; S. Schafföner1

1. University of Bayreuth, Ceramic Materials Engineering, Germany

Fabrication methods applying pre-impregnated fibers (prepregs) are often used for the manufacturing of ceramic matrix composites (CMC). The lamination, i.e. the stacking and subsequent pressing of the prepregs, is pivotal for the properties of the CMC. During processing, insufficient tack or too high pressure can lead to rebound of the prepregs and thus to delaminations. Prepregs with a polymer-based matrix system also show a dependance of tack on the humidity. In this study, prepregs for the fabrication of oxide fiber composites (OFC) by a cold roll lamination process were investigated, which comprised a ceramic slurry instead of a polymer matrix. Common rheological values, such as the shear rate dependent viscosity and thixotropy, proved to be insufficient for the prediction of the processability of prepregs with varying slurry compositions. Therefore, a novel test method was developed, which enabled the measurement of the slurry tack as well as the estimation of the prepreg rebound after lamination, which is paramount for the resulting microstructure and fiber volume content. The investigated material system comprised Nextel<sup>™</sup> 610 fabrics (DF-19) and slurries of varying compositions. The results obtained on mini-composites with this evaluation method were compared with OFC fabricated by the cold roll lamination process and showed good agreement between the test method and the actual manufacturing process.

#### 4:50 PM

### (ICC-SYM1-012-2024) Influence of porosity on the properties of a weak matrix ceramic matrix composite

L. Wagner\*1; G. Puchas1; S. Schafföner1

1. University of Bayreuth, Ceramic Materials Engineering, Germany

Oxide fiber composites (OFC) are a class of ceramic matrix composites (CMC) offering a quasi-ductile fracture behavior due to embedded ceramic fibers in a ceramic matrix. OFC offer high strength for application temperatures up to 1100 °C depending on the used fibers. According to the work of He and Hutchinson (1989), it depends on the fracture energy ratio as well as the Young's modulus ratio if a crack is deflected at a surface between two materials or propagated from one to the other. To maintain a damage tolerant behavior using the weak matrix concept, the porosity of the matrix enables crack deflection due to the low Young's modulus and fracture energy. In this work, the change of properties and the loss of the damage tolerant behavior with decreasing porosity were investigated. In order to do this, a NextelTM 610/Al2O3-ZrO2 composite was infiltrated with zirconium-n-butoxide to gradually decrease the matrix porosity from 47 - 34 %. The microstructure of the samples was investigated in regard to possible porosity gradients due to reinfiltration and the effects of matrix densification. Different mechanical testing methods were carried out to determine the influence of the porosity on the mechanical properties of OFC and matrix, respectively. A loss of the damage tolerant behavior at a matrix porosity of 34 % was determined.

### Symposium 2: Electronic Ceramics and Devices

#### Symposium 2: Electronic Ceramics and Devices

Room: St. Laurent 3 Session Chair: Alexander Frisch, Karlsruher Institute of Technology

#### 3:30 PM

## (ICC-SYM2-007-2024) Enhanced Charge Transfer via Dielectric Interface in Rechargeable Batteries (Invited)

T. Teranishi\*1; Y. Toyota1; R. Yamanaka1; S. Kondo1; A. Kishimoto1

1. Okayama University, Japan

Rechargeable batteries with drastically enhanced power density/ energy density are promising candidates to power next-generation vehicles, contributing a future sustainable society. A breakthrough involving dielectric insulating layer to assist efficient charge transfer at battery interface has been newly proposed. In the lithium ion batteries (LIBs), experimental and computational results proved that solvated Li are preferentially involved in the adsorption and desolvation process on the dielectric surface, followed by Li intercalation near the dielectrics-active materials-electrolyte triple phase interface (TPI). In fact, high rate capabilities of LIB was drastically improved via the BaTiO<sub>3</sub> nano-powder incorporation into the cathodeelectrolyte interface. The high rate capability was further strengthened with increasing TPI density for nano-cube (NC) BaTiO<sub>3</sub> modified cathodes. The architecture was also applied to the charge accumulation into activated carbon (AC) micropores in lithium ion capacitors (LICs). The optimized capacity of the BT-AC composite (annealed at 300°C for 20 h) was 35% higher than the capacity of the bare AC. The improved capacity characteristics were responsible for the promoted charge-transfer reaction activity at the electric double layer, involving adsorption/desorption and solvation/desolvation processes via the dielectric interface.

#### 4:00 PM

### (ICC-SYM2-008-2024) All-solid-state Li-ion batteries prepared by water processing (Invited)

S. Yasui\*1

1. Tokyo Institute of Technology, Japan

The development of sulfide-type solid electrolytes has been investigated for the practical application of all-solid-state batteries with high energy density. However, sulfide-type solid electrolytes are known to react with moisture in the atmosphere to generate hydrogen sulfide. On the other hand, atmospherically stable oxidetype solid electrolytes are investigated for safer use. In addition, completely new high-conductivity electrolytes have been proposed, such as hydrate melts and silica-ionic liquid composites, which have attracted much attention due to their aqueous processes, lowtemperature operation, and rapidly expanding process/application windows. More recently, flexible oxide-polymer composite solid electrolytes with excellent electrochemical properties have been reported. We have developed a series of oxide-based solid electrolytes with flexible properties and superior ionic conductivity in the order of several mS/cm to realize environmentally benign and formable solid-state lithium-ion batteries. Although "water-prohibition" is common in the battery world, we dared to use water processing in consideration of environmental harmonization and cost performance. As long as water is used, control of electrolysis of water is important, but this can be cleared by precise control. We will report on the characteristics of all-solid-state batteries fabricated using the developed solid electrolytes.

#### 4:30 PM

### (ICC-SYM2-009-2024) Impact of $TiO_2$ at the Grain Boundaries in Lithium Lanthanum Titanate Solid Electrolytes

J. C. Madrid Madrid\*1; A. Jonderian2; E. McCalla2; K. K. Ghuman3

- 1. Institut National de la Recherche Scientifique
- 2. McGill University, Chemistry, Canada
- Institut National de la Recherche Scientifique, Énergie Matériaux Télécommunications, Canada

The enhancement of Li-ion conductivity within the perovskite Li-La-Ti-O samples (LLTO) by adding TiO<sub>2</sub> remains unexplained in the literature. Herein, microscopy shows that TiO<sub>2</sub> appears at the grain boundaries (GB) of the perovskite, prompting a comprehensive Molecular Dynamics investigation. In this work, we analysed symmetric and mixed LLTO GBs, as well as LLTO/TiO<sub>2</sub> interfaces, to understand the impact of the secondary phase on Li-ion conductivity compared to other factors, such as disorder or strain present in the samples due to the GBs. The rigid-ion Buckingham-type potential combined with a long-range Coulombic term was used to accurately model ionic interactions. The investigation of diffusion mechanisms through Mean Squared Displacement (MSD) analysis unveiled that disordered TiO<sub>2</sub> phases significantly enhance Li-ion mobility compared to more orderly Sigma 5 or Mixed GBs. This suggests that disorder may create additional pathways for ion diffusion not present in symmetric GBs or crystalline LLTO samples. Furthermore, the enhanced Li-ion diffusion through LLTO/TiO<sub>2</sub> interfaces was indeed observed in the calculations and is attributed to the presence of TiO<sub>2</sub> phases and, to a lesser extent, to the highly disordered interface formed between them. These insights into the intricate migration mechanisms of Li-ion could advance the development of efficient solid-state electrolytes.

# Symposium 4: Green ceramics for clean energy and sustainability

### Symposium 4: Energy Ceramics Synthesis and Manufacturing II

Room: Montreal 6

Session Chair: Christos Agrafiotis, DLR - German Aerospace Center

#### 3:30 PM

# (ICC-SYM4-008-2024) Non-sintering Preparation and Properties of Solid Heat Storage Materials for Clean Energy (Invited)

K. Liu\*1

1. Institute of Process Engineering, Chinese Academy of Sciences, Materials and Environmental Engineering, China

In recent years,  $CO_2$  emission of urban heating is huge consuming 214 million tons of standard coal in the nern China alone in recent years. Heat storage is a kind of energy, storage, and solid heat storage technology can make full us to be an energy and regulate the power grid. However, commended the neat storage materials (SHSMs) have the problems such us that carbon emission (8t  $CO_2$  for 1t magnesia parts) and high tos in production process, which limit the large-scale applitude. This study uses calcined bauxite as the aggregate and a mixture of magnesium silicate hydrate gel and calcium aluminate cement as composite binder. The results show that the prepared non-sintering SHSMs have good mechanical strength, creep resistance and thermal cycling properties at room temperature and middle to high temperature. The thermal conductivity of SHSMs is 2.23 W/(m•K), and the bending strength after 20 thermal shock cycles is more than 10 MPa, which can meet the requirements of high-temperature thermal storage application (>1000 °C).

#### 4:00 PM

# (ICC-SYM4-009-2024) Optimisation of cold sintering processing routes for solid-state batteries and recycled ceramic waste materials

C. E. Dancer\*1; R. Yavari1; D. Dabera1

1. University of Warwick, Warwick Manufacturing Group, United Kingdom

The cold sintering process is attractive for processing ceramic materials primarily due to the low temperatures required. In this presentation we will present recent research examining the optimisation of the cold sintering process for two ceramic materials for different applications. We demonstrate densification of the oxide SSE aluminium doped lithium lanthanum zirconium oxide (Al-LLZO) which is a strong candidate for use in solid state battery electrolytes. Using an additive and optimised cold sintering processing conditions we obtain a bulk density of >95% and strong electrochemical performance. Secondly, we will discuss our work on the cold sintering of mixed recycled ceramic waste for reuse in structural applications such as domestic worksurfaces, comparing our findings to those of existing materials in use. Here we found that a knowledge of the composition of the waste stream is critical to obtaining good results, particularly where mixed ceramic-polymer waste streams are introduced. An initial analysis of the energy consumption and supply chain advantages of the use of ceramic waste material will also be presented.

#### 4:20 PM

### (ICC-SYM4-010-2024) Microstructure evolution of blacklight sintered ferroelectric ceramics

L. Fulanovic\*1; M. Scherer1; J. Rödel1

1. TU Darmstadt, Nonmetallic-Inorganic Materials, Germany

Demands for efficient energy and time densification processes have conditioned the advancement of the thermal treatment of polycrystalline ceramics, specifically sintering. The recently developed blacklight sintering technique embodies a highly energy-efficient thermal process. Specifically, thermal energy is only transferred to the sample, and a furnace containing the thermal energy is not required. This novel rapid densification technique provides extreme heating and cooling rates of 100 K/s, thus, the entire sintering cycle lasts a minute. Furthermore, it is simple to operate as it doesn't require complex sample holders or protective atmospheres. Our previous work demonstrated the feasibility of blacklight sintering for barium titanate ceramics, achieving a relative density of 90 % and established electrical properties. The current study focuses on the microstructural evolution of barium titanate. Powder compacts were thermally treated to obtain a relative density of 65 to 90 %. Temperature-dependent density curves were compared to reference furnace-sintered samples. Additionally, the microstructures were analyzed at each sintering stage. In the case of blacklight, samples exhibited profound microstructural gradients, particularly in the second sintering stage. Furthermore, the characteristic abnormal grain growth of barium titanate was comparably strong for both blacklight and furnace-sintered samples.

#### 4:40 PM

### (ICC-SYM4-011-2024) Manufacturing of large-scale Solid Oxide Electrolyzer Cells at Topsoe

S. Pirou\*1; K. Brodersen1; C. Gadea1; A. M. Molina1; B. T. Dalslet1; I. Ritucci1

1. Topsoe, Power-to-X, Denmark

Topsoe's vision is to be recognized as the global leader in carbon emission reduction technologies by 2024. Our energy-efficient solid oxide electrolyzer cell (SOEC) technology is integral to future Power-to-X plants and downstream processes. As one of the few global companies with wide-ranging expertise and technologies, we transform renewable electricity, steam, biomass, and waste into zero-emission fuels and chemicals, contributing to a sustainable future. To support electrolysis capacity, we're constructing an SOEC manufacturing facility in Denmark, set for operation in 2025, which will produce electrolysis stacks and modules with an annual capacity of 500 MW, expandable as needed. This presentation will address the main challenges in the fabrication and large up-scaling of our solid oxide electrolyzer cells. The main processing steps of the cell manufacturing will be described and the key parameters for a successful cost-effective and scalable manufacturing processes will be discussed.

#### 5:00 PM

# (ICC-SYM4-012-2024) Synthesis, microstructure, and electrical properties of green, sustainable, and eco-friendly fly ash-based geopolymer material

M. Yadav<sup>\*1</sup>; E. Varathan<sup>1</sup>

1. CSIR-National Physical Laboratory, Bhartiya Nirdeshak Dravya(BND), India

Fly ash geopolymer binder, a recent innovative advancement, are inorganic ceramics composed of alumino-silicates. They form rigid gels under ambient conditions, yielding network structures convertible into crystalline or glass-ceramic materials. Geopolymer binders are synthesized from aluminosilicatesl(fly ash) and alkaline activator. It offers diverse applications and lower CO2 emissions compared to traditional cement. Fly ash, a coal industry by-product often discarded in landfills, can be repurposed in geopolymers. The synthesis involves using appropriate alkaline activator (e.g., sodium/ potassium hydroxide with silicate) to dissolve aluminosilicate, causing atom reorganization to form polymer backbone, releasing water, and forming final geopolymer. Factors like alkali concentration and activator-to-fly ash ratio influenced geopolymerization. The molarity of potassium hydroxide was fixed at 12 M, while ratio of fly ash to activator varied from 1.0 to 1.75. Characterization was performed through techniques such as XRF, XRD, FTIR, and FESEM. FTIR identified functional groups, FESEM examined morphology, XRD analyzed crystalline phases, and XRF provided oxide composition. Electrical properties were studied through an impedance analyzer to assess suitability for insulation.

### Symposium 7: Novel Processing Techniques

### Symposium 7: Processing Strategies for UHTCs

Room: Montreal 8

Session Chairs: Jon Binner, University of Birmingham; Bernadette Sanchez, Sandia National Laboratories

#### 3:40 PM

#### (ICC-SYM7-008-2024) Processing Strategies for Compositionally Complex Ultra-High Temperature Ceramics (Invited)

W. Fahrenholtz\*1

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

Ultra-high temperature ceramics have melting temperatures in excess of 3000°C, which makes them among the most refractory materials that are known. The high melting temperatures result in the need for extreme temperatures for the synthesis and densification of these materials. Reaction-based processing techniques that involve in-situ synthesis of the desired phases provide several advantages compared to conventional ceramic powder processing approaches. This presentation will present case studies of the synthesis and densification of compositionally complex ultra-high temperature ceramics. The first will focus on high entropy carbides that were produced at significantly lower temperatures than other processing routes, yet retained strength to higher temperatures than other reports. The second case study will compare two synthesis routes for ultra-high temperature ceramics containing two phases, one boride and one carbide. These so-called dual-phase ceramics were produced by two different methods that will be compared. The presentation will conclude with a perspective on promising research directions for synthesis and densification of ultra-high temperature ceramics.

#### 4:10 PM

(ICC-SYM7-009-2024) Synthesis and processing of metalorganic Hf and Zr precursors for the deposition of UHTC films and analysis of thermal oxidation using advanced XPS

J. D. Boissiere<sup>\*1</sup>; H. Root<sup>1</sup>; F. El Gabaly Marquez<sup>1</sup>; L. Treadwell<sup>1</sup>; B. H. Sanchez<sup>1</sup>

1. Sandia National Laboratories, USA

Ultra-high temperature ceramics (UHTCs), such as metal borides and carbides of Zr or Hf, are of broad interest to industries including aerospace, nuclear energy, and electronics due to their thermochemical and mechanical properties. Bulk processing or traditional sputter deposition techniques are not practical to meet current component manufacturing needs for these materials. Therefore, new routes that enable the deposition of films and coatings of UHTCs must be developed. We have synthesized a series of metalorganic complexes including Zr- and Hf-alkyl, alkoxide, siloxide, and boryl ligands, which are being utilized as preceramic precursors for the production of metal carbides, silicides, and borides. These complexes are synthesized from commercial halide and cyclopentadienyl Zr and Hf precursors. Molecular dynamics simulations of the metalorganic species are used to inform decomposition pathways and direct ongoing precursor development and processing methods. These outcomes are coupled with thorough chemical analysis and advanced materials processing to yield ceramic products. Sandia has developed an ambient pressure XPS with in-situ sample heating capabilities, which is used to evaluate the resulting chemical properties and investigate the thermal oxidation processes of targeted UHTC materials.

#### 4:30 PM

(ICC-SYM7-010-2024) Use of 2D carbides (MXenes) in processing ultra-high temperature ceramics (Invited)

B. Wyatt<sup>1</sup>; S. Nemani<sup>1</sup>; B. Anasori<sup>\*1</sup>

1. Purdue University, USA

The family of 2D nanocarbides, known as MXenes, has emerged since 2011, many of which have compositions similar to ultra-high temperature ceramics (UHTCs), such as TaC. MXenes have been mostly studied as nano-functional materials. However, we believe they can be used to process and design novel UHTCs structures and composite materials with high precision. MXenes (1-nm-thick 2D sheets of carbides) are scalable and hydrophilic, solution-processed 2D nanomaterials that can be applied as a coating by a simple airbrush, like paint. These coatings and films can be heat treated to transform into UHTC coatings with controllable morphology, texture, and layering. Additionally, as-synthesized MXenes have negative surface charges, which make them stable as colloidal solutions. In parallel, mixing many ceramic particles in acidic solutions of pH ~ 4-5 can lead to the protonation of their surfaces and positive surface charges. Consequently, mixing MXene flakes with ceramic particles leads to their self-assembly and coverage of the ceramic particles with MXenes. By processing and sintering the resulting mixed materials, we can manufacture UHTCs with homogenous dispersion of the additives within the matrix. In this talk, we will present our recent findings on MXenes' high-temperature phase transformation, their integration into UHTCs matrices and reactions during the sintering processes, and their resulting properties.

#### 5:00 PM (ICC-SYM7-011-2024) Refractory Ceramics Using High Char Polymers (Invited)

J. D. Sitter\*1; M. Laskoski1

1. United States Naval Research Laboratory, High Temperature Materials, USA

Ultra-high temperature ceramics (UHTCs) demonstrate significant promise in many high-performance applications. However, existing processing typically produce macrocrystalline ceramics that are too brittle and expensive to incorporate into aerospace and military systems. Our approach produces dense, nanocrystalline, monolithic UHTC composites with precursor flexibility that can maximize density while altering hardness, and durability under high temperatures. This process is based on a novel synthesis route using compressed powder mixtures of metal precursors and high charyielding that yield shaped carbides, nitrides, and borides under mild heat treatment. Incorporation of various metals, fibers, and ceramics into these composites can improve their mechanical, electrical, and thermal properties and adapt them for many emerging applications.

# Symposium 11: Recent advances and the future of additive manufacturing

#### Symposium 11 VAT Photopolymerization III

Room: Montreal 7

Session Chair: Richard Gaignon, 3DCERAM SINTO

#### 3:30 PM

(ICC-SYM11-008-2024) Thermal and structural characterization of joined alumina components manufactured via digital light polymerization

C. Albunio<sup>\*1</sup>; S. Sobhani<sup>1</sup>

1. Cornell University, USA

The ability to join 3D printed parts allows for the creation of larger, more complex structures while reducing print time and potential print errors. Moreover, joining parts extends the print volume capability, enabling fabrication of larger objects that surpass the size constraints of the build plate. This represents a major step toward increasing the relevance of ceramic additive manufacturing beyond current applications. In this work, several joint designs and methodologies are examined. Joining is done in the green state using alumina photosensitive slurry or after sintering using a common ceramic joining material - ceramabond. The thermal conductivity and flexural strength of the parts before and after thermal cycling are characterized.

#### 3:50 PM

#### (ICC-SYM11-009-2024) Reciprocating Wear of DLP Additively Manufactured Al<sub>2</sub>O<sub>3</sub>-based Ceramics

A. M. David\*1; M. Y. Amegadzie1; P. Siahpour1; K. P. Plucknett1

1. Dalhousie University, Mechanical Engineering, Canada

Digital light processing (DLP) is a 'layer-by-layer' additive manufacturing (AM) technology based on a vat polymerisation approach. DLP has been used to print alumina ( $Al_2O_3$ ) based ceramics for assessment of their reciprocating wear response. The effects of both the printed layer thickness (from 10 to 75 µm) and the build angle (from 0 to 90°, varied in 15° increments) have been investigated for sintered  $Al_2O_3$  formed using DLP. For the actual 'bottom-up' DLP processing, samples are subjected to pre-conditioning and burnout steps, followed by sintering in air (typically 1650 °C for 2 h). The surface roughness of as-sintered parts was assessed using confocal laser scanning microscopy prior to wear testing. Linear reciprocating wear tests were conducted at a frequency of 5 Hz, for a maximum duration of up to 60 minutes. The tests were performed at room temperature, under loads up to 60 N. It was apparent that the lowest

#### 4:10 PM

#### (ICC-SYM11-010-2024) Development and Characterization of Low Sintering Temperature Additively Manufactured Aluminum Nitride Ceramics for Aerospace Applications

G. D'Orazio\*1; S. Sobhani1

1. Cornell University, Mechanical and Aerospace Engineering, USA

Planned human exploration of terrestrial bodies beyond the Earth's sphere of influence requires considerable power generation for propulsion, life support, and science equipment. Many studies explore the use of a megawatt range nuclear reactor to meet these demands. Such a system requires extensive heat rejection technologies, such as heat pipes, pumped fluid loops, and massive radiator installations. This work examines the development of an aluminum nitride (AlN) and photosensitive resin feedstock to produce 3D printed heat pipes. Using AlN as the envelope material can extend the heat pipe's operational temperature range beyond typical aluminum and titanium-water heat pipe systems. A low temperature, pressureless, AlN formulation using yttria and calcium zirconate sintering aids was developed for digital light processing 3D printing. Exposure settings and slurry rheology were optimized for high fidelity parts with minimum features in the 300 µm range. Ceramic slurry lifespan was examined via Fourier transform infrared spectroscopy, exploring partial polymerization due to overcuring. Thermogravimetric analysis informed the development of a nitrogen atmosphere debinding curve, and parts were subsequently sintered at 1600°C. The surface quality of as-printed parts were characterized in terms of surface roughness and emissivity.

#### 4:30 PM

# (ICC-SYM11-011-2024) Effect of sintering temperature on feature resolution and flexural strength of ceramics fabricated via vat photopolymerization additive manufacturing

L. Bezek<sup>1</sup>; K. Lee<sup>\*1</sup>

1. Los Alamos National Laboratory, USA

Employing additive manufacturing (AM) to fabricate ceramic parts offers the opportunity to fabricate complex, high-resolution parts for diverse, functional applications. However, one ongoing challenge in qualifying ceramic AM is optimizing the post-process, particularly sintering, conditions to consistently produce geometrically-accurate and mechanically-robust parts. This work investigates how sintering temperature (varied between 900 and 1300°C) affects feature resolution and flexural properties of silica-based parts formed by vat photopolymerization AM. Test artifacts were designed to evaluate features of different sizes, shapes, and orientations, and three-point bend tests were used to evaluate mechanical properties. Deviations from designed dimensions often increased with higher sintering temperatures and/or larger features. Many features exhibited defects, often dependent on geometry and sintering temperature. Higher sintering temperatures yielded parts with higher strength and lower strain at break. These results serve as design guidelines to achieve more predictable part performance when considering the future design and manufacture of complex ceramic parts.

#### 4:50 PM

#### (ICC-SYM11-013-2024) Atomic Layer Deposition (ALD) for Nanoscale SiC AM Feedstock Improvement

C. Gump\*1; D. Lindblad1; J. Gauspohl1; C. Christopher1

1. Forge Nano, R&D, USA

Forge Nano (FN) has applied ALD to modify the surface of SiC AM feedstocks. Long used in the semiconductor industry to coat silicon wafers, ALD uses self-limiting surface reactions to deposit nanoscale films onto surfaces. The film thickness depends digitally on the number of ALD cycles performed. FN has previously coated metal AM feedstocks, including Ti64 and AlSi10Mg, with 1-3 nm of Al<sub>2</sub>O<sub>3</sub> ALD films and demonstrated increased feedstock oxidation resistance and flowability, as well as improved mechanical properties of as-printed and hot isostatically pressed parts. Using our novel CRISP chemistry process for the deposition of SiO<sub>2</sub>, 100 g batches of SiC were coated with multiple thicknesses of SiO<sub>2</sub> ALD in FN's research fluidized bed reactor (FBR). Elemental analysis by ICP and LECO C and O proved ineffective at characterizing the films, but a FIB liftout/STEM imaging and elemental mapping analysis technique showed that the particles were uniformly coated, with the film thickness scaling with the number of ALD cycles performed. The deposition process was further scaled to 3 kg batches in FN's pilot FBR. We expect this material to exhibit improved flowability, densification and mechanical properties in binder jet and other similar AM applications. Further scaling to 100-300 kg batches is possible in FN's large scale semi-batch rotary coating tools, and even further using a continuous vibrating bed reactor.

### Tuesday, July 16, 2024

#### **Plenary Session**

#### **Plenary Session: Chang-Jun Bae** Room: Montreal 1-3

#### 8:30 AM

## (ICC-PLE-003-2024) Learning from the Past, Looking to the Future of Ceramic Additive Manufacturing

C. Bae\*1

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

Ceramic additive manufacturing (C-AM) promises to revolutionize industries by enabling direct fabrication of complex ceramic structures. Challenges include defects and distortions during green body formation and binder burn-out. We introduce an in-situ sensing system for defect monitoring and quantitative analysis, shedding light on fabrication dynamics. Additionally, we investigate binder burn-out through numerical simulations and experimental characterizations, revealing spatial and temporal evolution of thermal degradation behavior. Our research aims to enhance reliability, efficiency, and versatility in ceramic fabrication processes.

# Symposium 1: Aerospace Ceramics and Composites

#### Symposium 1: Aerospace Ceramics and Composites

Room: Montreal 1-3 Session Chairs: Gregory Thompson, University of Alabama; Christopher Weinberger, Colorado State University

#### 9:50 AM

#### (ICC-SYM1-014-2024) Hardness of Transition Metal Carbides: Processing, Microstructure and Properties (Invited)

B. R. Watkins<sup>1</sup>; C. Blacksher<sup>2</sup>; G. B. Thompson<sup>2</sup>; C. R. Weinberger<sup>\*1</sup>

- 1. Colorado State University, Department of Mechanical Engineering, USA
- 2. University of Alabama, USA

Transition metal carbides are well known UHTCs that exhibit both extreme melting temperatures and high hardness. The mechanical properties of these materials are well known to be dependent on the carbon content as well as the transition metal type. In this talk, we examine the role structure, microstructure and processing play in controlling the hardness of these transition metal carbides. Specifically, we examine how chemistry affects hardness using density functional theory and dislocation modeling, focusing on the transition metal and carbon concentration. Furthermore, we demonstrate that this hardness can be further controlled through processing and grain size control by comparing bulk sintered transition metal carbides and magnetron sputtered film. These results shed light into the anomalous hardness observed in these materials as well as points to processing routes for ultrahard materials.

#### 10:20 AM

### (ICC-SYM1-015-2024) Liquid phase sintering of C fiber reinforced ultra-high temperature ceramics composites

L. Zoli\*1; D. Sciti1; A. Vinci1

1. CNR-ISSMC, DSCTM, Italy

Ultra-high temperature ceramic matrix composites (UHTCMCs) are the next generation of reusable materials for application in extreme environments associated with aerospace and military applications. Preferably, UHTCMCs are constituted of just two essential elements that are integrated appropriately, e.g.: a carbon fiber (Cf) fabric and an ultra-refractory matrix. These UHTCMCs are meant to be reusable owing to in situ formation of a protective scale during exposition. To produce such revolutionary materials, we have developed a process consisting of powder slurry impregnation of fiber fabrics followed by sintering. Densification through sintering at high temperature is the distinctive feature of these UHTCMCs, but it also the most critical aspect of the process because over sintering may also lead to embrittlement. In this study, the hypothesis driving the present research is: Liquid phase sintering (LPS) is more effective for densification of UHTCMCs with carbon fibers than solid state sintering because it allows for maintaining pristine fibers. Four compositions (42 vol% ZrB2, 40 vol% Cf, 12 vol% LPS aid, 6 vol% SiC) were obtained by slurry impregnation of UD fabrics followed by hot pressing sintering at 1700 °C, 30 MPa using Si<sub>3</sub>N<sub>4</sub>, Si<sub>3</sub>N<sub>4</sub>:2B<sub>2</sub>O<sub>3</sub>, ZrSi<sub>2</sub> and Y<sub>2</sub>O<sub>3</sub> as LPS aids. Densification curves, microstructure, and mechanical properties up to 1500 °C were analyzed and compared.

#### 10:40 AM

### (ICC-SYM1-017-2024) Reactive sintering of dense titanium diboride-based composites

- D. Kozien\*<sup>1</sup>; Z. Pedzich<sup>1</sup>; D. Salamon<sup>2</sup>; D. Valasek<sup>2</sup>; P. Tatarko<sup>3</sup>; M. Hičák<sup>3</sup>;
- O. Hanzel<sup>3</sup>; P. Nieroda<sup>1</sup>; K. Pasiut<sup>1</sup>; L. Chlubny<sup>1</sup>
- 1. AGH University of Krakow, Faculty of Materials Science and Ceramics, Poland
- 2. Central European Institute of Technology, Brno University of Technology, Czechia
- 3. Institute of Inorganic Chemistry, Slovak Academy of Sciences, Department of Ceramics, Slovakia

Composites basing on boron carbide (B<sub>4</sub>C) have gained significant interest due to their exceptional physical and chemical properties. This study utilized boron carbide, carbon and intermetallic compounds from Ti-Si system to produce a composite containing more than 99% of TiB<sub>2</sub>, TiC and SiC. For densification three different methods were used: pressureless sintering (PS), hot-pressing (HP) and spark plasma sintering (SPS). Temperatures of consolidation were different: PS 1650-1750°C; HP 1500-1550°C and SPS 1400-1450°C, respectively. A type of used intermetallic affected chemical reactions mechanisms, but there was discovered that using intermetallic phases such as TiSi, TiSi<sub>2</sub>, and Ti<sub>5</sub>Si<sub>3</sub> in combination with appropriate molar proportions of B<sub>4</sub>C and C led to dense composite material of very simple phase composition. This process resulted in the elimination of free carbon typically coming from commercial raw materials. Finally achieved high-temperature refractory TiB2-TiC-SiC composite exhibited high mechanical strength and fracture toughness. The chemical reaction that occurred during sintering was highly effective, 99% of the initial phases decomposed and allowed to form a new TiB<sub>2</sub>, SiC and TiC phases, which were well-densified at relatively low temperatures. Acknowledgments: This research was supported by a grant from the National Center for Research and Development (LIDER XIII; Grant No. 0024/L-13/2022

#### 11:00 AM

### (ICC-SYM1-018-2024) Ablation resistance of titanium diborate based composites obtained via reactive sintering

- Z. Pedzich<sup>\*1</sup>; D. Kozien<sup>1</sup>; D. Salamon<sup>2</sup>; D. Valasek<sup>2</sup>; P. Tatarko<sup>3</sup>; M. Hičák<sup>3</sup>; O. Hanzel<sup>4</sup>; P. Nieroda<sup>1</sup>; K. Pasiut<sup>1</sup>; L. Chlubny<sup>1</sup>
- 1. AGH University of Krakow, Faculty of Materials Science and Ceramics, Poland
- 2. Brno University of Technology, Central European Institute of Technology, Czechia
- 3. Institute of Inorganic Chemistry, Slovak Academy of Sciences, Department of Ceramics, Slovakia
- 4. Institute of Inorganic Chemistry, Slovak Academy of Sciences, Ceramics, Slovakia

Presented results of studies concern ablation resistance of TiB<sub>2</sub>-TiC-SiC composites made of B<sub>4</sub>C, C and intermetallic compounds via reactive sintering by the means of SPS technique. Intermetallics from two systems were used Ti-Si (TiSi, TiSi<sub>2</sub>, or Ti<sub>5</sub>Si<sub>3</sub>) and Ti-Al (Ti<sub>3</sub>Al, TiAl, and TiAl<sub>3</sub>). Composites were sintered at the temp. range of 1000-1700°C. Density was determined using Archimedes' method. Phase compositions were studied utilizing Panalytical/Philips X'Pert Pro MD XRD diffractometer. X-ray absorption spectroscopy (XAS) measurements were performed at PIRX beamline of the Solaris National Synchrotron Radiation Centre, Krakow, Poland. Ablation resistance was tested using an oxyacetylene flame at 1800°C. Material was exposed for flame during 60 s. Ablated surfaces were examined using a confocal microscope (Lext OLS 3100, Olympus) to determine the depth of the ablation zone. The linear ablation rate of the material was determined. Microstructure and chemical composition of ablated surfaces were examined by the means of SEM/EDS method (Thermo Scientific Scios 2). Generally, investigated composites showed good resistance

for ablation. In individual cases this resistance significantly exceeded values of commercially available materials. Acknowledgments: This research was supported by the National Center for Research and Development - Programme LIDER XIII; Grant No. 0024/L-13/2022.

#### 11:20 AM

#### (ICC-SYM1-019-2024) Influence of Metal Type and Process Conditions on the Hardness of Transition Metal Carbide Films

C. H. Blacksher<sup>1</sup>; C. R. Weinberger<sup>2</sup>; G. B. Thompson<sup>\*1</sup>

- 1. University of Alabama, USA
- 2. Colorado State University, Department of Mechanical Engineering, USA

Transition metal carbides (TMCs) are used in abrasive and other extreme environments. Requisite to these applications is a sufficient high hardness. Using the same rocksalt crystal structure, we report how changes in the metal sublattice and processing conditions alter the hardness of these films. Here Zr, Nb and Ta have been reactively sputter deposited with ethylene to form nanocrystalline TMC films with ZrC, NbC, and TaC being approximately 10, 28, and 35 GPa, respectively. The lower hardness of ZrC is explained by a microstructure being less dense which inhibited cracking in the carbide grains. In contrast, prevalent cracking was observed throughout the NbC and TaC films. While the atomic mass of Ta (Z=73) would contribute to higher atomic peening effects to densify the carbide film, Zr (Z=40) and Nb (Z=41) are very similar but have drastic hardness differences. Thus, the means of the carbide film to accommodate deformation during growth is important. These differences in hardness are discussed in terms of the deformation mechanisms between group IVB and VB TMCs as well as growth rate and controlled increases in pressure to change the arrival energy during growth.

### Symposium 2: Electronic Ceramics and Devices

### Symposium 2: Electronic Ceramics and Devices

Room: St. Laurent 3 Session Chair: Yu Kumagai, Tohoku University

#### 9:50 AM

#### (ICC-SYM2-010-2024) Nano-scale local crystal phase analysis in bismuth ferrite thin film by atomic-scale scanning transmission electron microscopy (Invited)

Y. Sato\*1

1. Kumamoto University, Research and Education Institute for Semiconductors and Informatics, Japan

Bismuth ferrite (BiFeO<sub>3</sub> (BFO)) thin films have been intensively studied because of its potential application to spintronics, memory, and energy conversions. Since BFO can have many crystal phases and the different phases can exhibit different physical properties, it is important to identify the crystal phase. However, structural identification is often a challenging task, because BFO film can have complex multiple-phases nanostructure, depending on film thickness, substrate choice, film growth method, etc. In the present study, I will introduce our recent investigation on local crystal phase analysis in BFO by accurate lattice parameter measurement using atomic-scale electron microscopy. Multiple crystal phases in nanoscale regions have been characterized from atomic positions in scanning transmission electron microscopy images.

21

#### 10:20 AM

## (ICC-SYM2-011-2024) Thin Film Multilayered Ferroelectrics in the $Zn_{1,y}Mg_yO$ System

R. Spurling\*<sup>1</sup>; C. Skidmore<sup>1</sup>; J. Hayden<sup>1</sup>; J. Nordlander<sup>1</sup>; J. Casamento<sup>1</sup>; K. Kelley<sup>2</sup>; J. Maria<sup>1</sup>

Pennsylvania State University, Materials Science and Engineering, USA
 Oak Ridge National Lab, USA

We report on observations of ferroelectric switching in thin film heterostructures based on wurtzite  $Zn_{1-x}Mg_xO$ . We discuss sputter deposition synthesis and electrooptic property characterization of  $Zn_{1-x}Mg_xO$ -based thin film stacks. We observe that ferroelectric switching is achieved through polar non-ferroelectric film layers through the presence of an interface with ferroelectric  $Zn_{1-x}Mg_xO$ . We observe this phenomenon for  $Zn_{1-x}Mg_xO/ZnO$  interfaces as well as in oxide/nitride wurtzite film stacks, such as  $Zn_{1-x}Mg_xO/AlN$ . Remnant polarization and coercive field measurements support the conclusion that switching occurs through the entire film stack. We propose that ferroelectric domains nucleating in the  $Zn_{1-x}Mg_xO$  layer propagate through the film interface and induce switching across the film stack.

#### 10:40 AM

(ICC-SYM2-012-2024) Development of (K,Na)NbO<sub>3</sub>-based lead-free piezoceramics with high piezoelectricity and enhanced temperature stability (Invited)

J. Li\*1

1. Tsinghua University, School of Materials Science and Engineering, China

Piezoelectric materials enable the conversion between mechanical and electrical energy, and the development of high-performance lead-free piezoelectric ceramics has been one of the most active materials research topics in the last decades. (K,Na)NbO<sub>3</sub> (KNN) has been revealed as the most promising candidate for lead-free piezoelectric applications, and continuous efforts are still devoted to improve its comprehensive performance for extending its applications. This talk will review several approaches to develop high-performance KNN-based ceramics with temperature-insensitive high piezoelectricity, including diffused phase boundary engineering and gradient doping strategies. I will present our recent work demonstrating that the introduction of layered distributions of key dopants (Li and Sb) in monolithic ceramics is a facile approach to simultaneously enhance the piezoelectricity of KNN-based ceramics and its temperature stability, by which an outstanding d<sub>33</sub> of 508 pC/N and excellent temperature stability  $(d_{33} \text{ variation within 13\% over the temperature range of 25–150 °C})$ were achieved. Finally, I will introduce some application examples and share my perspectives about the future research and development of KNN-based piezoelectric ceramics.

#### 11:10 AM

## (ICC-SYM2-013-2024) In-situ TEM Observation of Domain Wall-Precipitate Interaction in (Ba<sub>0.7</sub>Ca<sub>0.3</sub>)TiO<sub>3</sub> Ceramics

- O. Taylor\*1; E. Chaffee1; B. Liu1; C. Zhao3; J. Rödel2; L. Zhou1; X. Tan1
- 1. Iowa State University, Materials Science and Engineering, USA
- 2. Technische Universität Darmstadt, Department of Materials and Earth Sciences, Germany
- 3. Xi'an Jiaotong University, State Key Laboratory of Electrical Insulation and Power Equipment, China

In-situ biasing with transmission electron microscopy (TEM) was used to observe the interaction between precipitates and domain walls in precipitation-hardened  $(Ba_{0,7}Ca_{0,3})TiO_3$  ferroelectric ceramics.  $(Ba_{0,7}Ca_{0,3})TiO_3$  ceramic pellets were fabricated by the solidstate reaction method. Two-step aging was employed to introduce Ca-rich precipitates in the Ba-rich ferroelectric matrix. TEM specimens were prepared with two gold pads as electrodes. DC voltages were delivered to the specimen in situ. Bright-field micrographs and electron diffraction patterns were recorded during ramping-up and

#### 22 **10th International Congress on Ceramics**

ramping-down of the voltage. It is observed that as voltage increases, the domains start to disappear in areas away from Ca-rich precipitates. Domains close to precipitates are persistent and disappear at higher voltages. During voltage ramping down, domains first appear at the precipitate/matrix interface and grow around the precipitates. Some domains are completely blocked by the precipitates, while others appear to cross over partially or fully. These real-time obser-vations suggest that the Ca-rich precipitates are acting as pinning points for the domain walls. The precipitate/matrix interface appears to be a preferred site for the nucleation of ferroelectric domains. The interactions between the Ca-rich precipitates and ferroelectric domains are responsible for the piezoelectric hardening effect.

#### 11:30 AM

#### (ICC-SYM2-014-2024) Simultaneous enhancement of piezoelectricity and mechanical quality factor in KNN-based lead-free ceramics via multivalent B-site codoping *WITHDRAWN*

T. Feng\*<sup>1</sup>; J. Li<sup>1</sup>

1. Tsinghua University, School of Materials Science and Engineering, China

Potassium sodium niobate (KNN) ceramics has been proved to be one of the most promising lead-free piezoelectric materials. However, for hard-type KNN piezoceramics with high quality factor ( $Q_m$ ), the piezoelectric coefficient ( $d_{33}$ ) usually decreases drasti-cally (<120pC/N), which limits its application in various electronic devices. Herein, a KNN-based ceramic with enhanced  $d_{33}$  and high  $Q_m$  is prepared by a conventional solidstate technique. The multi-valent B-site codoping induces various defects and defect dipoles, which disturbs the local stress field and electrostatic field, leading to the formation of hierarchical nanodomains and enhancement of  $Q_m$  and  $d_{33}$ . The best overall performance can reach  $d_{33}$ =132pC/N,  $Q_m$ =948 at room temperature and  $d_{33}$ =189pC/N,  $Q_m$ =916 at 100°C. This work provides a new insight for domain structure of hard-type KNNbased ceramics and offers potential for lead-free high-power piezoelectric applications.

### Symposium 4: Green ceramics for clean energy and sustainability

### Symposium 4: Design of Energy Ceramics

Room: Montreal 6

Session Chair: Flavio de Souza, Federal University of ABC

#### 9:50 AM

# (ICC-SYM4-014-2024) Microwave-Assisted Synthesis as New Route to Rare-Earth-Based Ceramic Materials (Invited)

E. Hemmer<sup>\*1</sup>

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

The remarkable optical properties of the rare-earth (RE) make RE-based materials ideal for applications ranging from biomedicine to optoelectronics and energy conversion technology. This is due the unique electronic properties of the RE elements allowing for upconversion, i.e., the emission of UV-visible light under near-infrared excitation. Upconverting nanoparticles (UCNPs) based on sodium rare-earth fluorides (NaREF<sub>4</sub>) are commonly synthesized by the thermal decomposition of metal precursors in high-boilingpoint solvents, based on convectional heating. Microwave reactors can improve reproducibility as offering better control over a reaction environment, more homogeneous heat distribution yields narrow size distributions, and rapid heating to the desired temperature shortens reaction times from hours to minutes. Targeting applica-tions in optoelectronics, photochemistry, and energy conversion, boosting the efficiency of upconverters is key. This presentation will shed light on the microwave-assisted synthesis of core/(multi) sheDerlotespresenter highlight pros and cons of this strategy towards the design of smallyet-bright upconverters.

#### 10:20 AM (ICC-SYM4-016-2024) Heteroatoms Co-doped Graphene for Supercapacitor Application

T. Tseng\*1

1. National Yang Ming Chiao Tung University, Institute, Taiwan

A simple, eco-friendly, cost-effective heteroatoms (nitrogen, phosphorus, and fluorine) co-doped graphene oxide (NPFG) is synthesized by hydrothermal functionalization and freeze-drying approach. The effect of different heteroatoms doping on the energy storage performance of synthesized reduced graphene oxide is investigated extensively. This study demonstrates that the nitrogen, phosphorous, and fluorine co-doped graphene (NPFG-0.3) synthesized with the optimum amount of pentafluoropyridine and phytic acid (PA) exhibits a notably enhanced specific capacitance (319 Fg-1 at 0.5 Ag-1), good rate capability, short relaxation time constant  $(\tau = 28.4 \text{ ms})$ , and higher diffusion coefficient of electrolytic cations  $(Dk + = 8.8261 \times 10-9 \text{ cm} 2 \text{ s} - 1)$  in 6 M KOH aqueous electrolyte. A symmetric coin cell supercapacitor device fabricated using NPFG-0.3 as the anode and cathode material with 6 M KOH aqueous electrolyte exhibit maximum specific energy of 38 W h kg-1, the maximum specific power of 716 W kg-1, and ~88.2% capacitance retention after 10000 cycles.

### Symposium 7: Novel Processing Techniques

#### Symposium 7: Innovations in processing and sintering Room: Montreal 8

Session Chairs: B.Reeja Jayan, Carnegie Mellon University; James Sitter, United States Naval Research Laboratory

#### 9:50 AM

(ICC-SYM7-012-2024) Unusual processing routes for the production of ceramic matrix composites (Invited)

J. Binner\*1

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

Ceramic matrix composites (CMCs) have shown considerable potential for use in a wide range of applications in sectors as diverse as aerospace, defence, energy and transport. Their significantly improved toughness compared to monolithic ceramics offers opportunities to take full advantage of the benefits offered by advanced ceramics, including low mass, high strengths and hardness, chemical inertness and high thermal resistance without having to suffer their major disadvantage of being brittle and hence susceptible to catastrophic failure. There are several different types of CMC, including those based on oxide, silicon carbide and carbon fibres with a diverse range of different ceramic matrices. This talk will examine a number of unusual processing routes, including the use of phosphate-based matrices for oxide-based CMCs, prepreg-based approaches more commonly found with polymer matrix composites, the use of polymer-derived ceramics and microwave- or RF-enhanced chemical vapour infiltration, as well as the additive manufacturing of CMCs. The advantages and disadvantages of each route will be discussed.

#### 10:20 AM

#### (ICC-SYM7-013-2024) Combining Flash Lamp Annealing (FLA) with Chemically Bound Ceramic (CBC) Chemistries to Enable Direct Additive Manufacturing of Ceramic Composites (Invited)

M. D. Losego\*1; E. Özmen1; N. Somers1

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

This talk will introduce a rapid, pressureless process to chemically bind ceramic parts via a combination of inorganic phosphate chemistries and electromagnetic irradiation. The ceramic components are synthesized from slurries of ceramic powders and  $Al(H_2PO_4)_3$ binders. In initial studies, short-waved IR irradiation is shown to be sufficient to drive phosphate condensation reactions, however under these low irradiation powers (~0.2 W/cm<sup>2</sup>), condensation and ceramization requires minutes to tens of minutes to complete. Transitioning these chemistries to a high power flash lamp annealing (FLA) system (~10 kW/cm<sup>2</sup>) enables rapid ceramization in under 1 min and denser overall microstructures. Interestingly, though, the total irradiation energy required to drive complete ceramization, as determined by diffraction, spectroscopy, and hygroscopicity, is found to be similar for the IR and FLA systems. Light absorbers also play a significant role in FLA processing and will be discussed. We have been able to generalize this approach to fabricate a range of ceramic composite compositions including Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub>, In<sub>2</sub>O<sub>3</sub> and others. As an exemplary model, a Cr<sub>2</sub>O<sub>3</sub>:Al<sub>2</sub>O<sub>3</sub> high temperature thermochromic ceramic will be presented and additively manufactured into a readable indicator that warns users of the temperature of an object.

#### 10:50 AM

#### (ICC-SYM7-014-2024) Contactless Flash Sintering: Enabling Rapid Densification of Ceramic Coatings

Z. Zhang<sup>\*1</sup>; C. Grimley<sup>1</sup>; A. Kupferberg<sup>1</sup>; C. Green<sup>1</sup>; G. Jones<sup>1</sup>; J. Minkiewicz<sup>1</sup>
 Lucideon, USA

Flash sintering (FS) offers accelerated kinetics and enhanced microstructural control in ceramic material processing. Lucideon's Contactless Flash Sintering (CFS) method innovatively replaces a contact point with plasma, enabling "contactless" current transfer and facilitating diverse sample geometries and coatings. A plasmamodulating algorithm enhances stability, and integration with a three-axis robot densifies electrolyte material sheets. However, the introduction of plasma introduces spatial, thermal, and mechanistic complexities. Understanding thermal distribution is crucial, especially with increasing sample thickness, decoupling localized plasma heat from Joule heating. CFS may interact differently with ceramics' conduction and dielectric breakdown mechanisms than traditional FS. This presentation explores the current understanding of CFS and ongoing efforts. Cross-sectional microstructural images of zirconia samples processed under various plasma conditions, along with in-situ infrared monitoring and finite element analysis, provide insights into thermal evolution. This aids in differentiating thermal and electrical effects and elucidating mechanistic variations between CFS and conventional FS. The study aims to offer valuable insights into the complexities and potential advantages of CFS in ceramic material processing.

#### Symposium 7: Reprocessing and Purification

Room: Montreal 8

Session Chair: Jeff Vervlied, Free Form Fibers, US

#### 11:10 AM

## (ICC-SYM7-015-2024) Effects of ore impurities on downstream purification processes of feldspar ore leachate

A. Caamino\*2; S. Shakibania1; J. Rosenkranz1

- 1. Luleå Tekniska Universitet, Department of Civil, Environmental and Natural Resources Engineering, Sweden
- 2. MSU-Iligan Institute of Technology, Department of Materials and Resources Engineering & Technology, Philippines

The abundance of feldspars as a silicate mineral has made them a potential source to extract potash (KCl) and alumina  $(Al_2O_3)$ , for agriculture and ceramic applications. However, like most tecto-silicate minerals, feldspars are difficult to decompose for the dissolution of their metallic components. This study, as part of the POTASSIAL-EU Project, has employed intensive grinding followed by HCl leaching approach to address the low dissolution

efficiency of K-feldspar components. Characterization of ground samples suggest that grinding model used resulted in particles less than 20 $\mu$ m. Regression and ANOVA results showed that among the three grinding parameters used in the study, ball-to-ore ratio was the most impactful parameter, followed by grinding time and the milling speed as the least impactful among them. Samples of varying Specific Surface Area (SSA) were subjected to HCl leaching where the recovery of the main elements – Potassium (K<sup>+</sup>) and Aluminum (Al<sup>3+</sup>) were consistent to increase with increasing time while Sodium (Na) and Calcium (Ca) were the major impurities recorded in the Pregnant Leach Solution (PLS). The results further recorded that while grinding significantly increases the concentration of K<sup>+</sup> and Al<sup>3+</sup>, the concentrations of the soluble Na and Ca impurities have remained low and incomparable to the recovery of the of main elements.

#### 11:30 AM

#### (ICC-SYM7-016-2024) Processing and Characterization of Model Cermet Waste Forms for Nuclear Applications

N. Marrero\*1; S. K. Sundaram1

1. Alfred University, Inamori School of Engineering, USA

The nuclear fuel cycle and various treatments for nuclear waste led to high-level waste (HLW) and low-level waste (LLW) streams, depending on the levels of radioactivity. Currently, glass and ceramic waste forms have been developed for HLWs and glass, cement-based waste forms for LLWs for repositories. Cermets or ceramic metal matrix composites show promise as versatile waste forms for a wide range of waste streams. Additionally, the combination of metals and ceramics aims to incorporate all the positive aspects of each material class including ductility, chemical durability, and overall strength of the waste forms. We have produced a series of model cermet samples by varying the ceramic-metal ratio (25 – 50 volume% of ceramics) and composition between stainless steel 316 combined with either zirconia or silicon carbide ceramics. The samples were densified using spark plasma sintering (SPS) and hot uniaxial pressing (HUP). We have characterized the cermet samples using scanning electron microscope and electron dispersive spectroscopy (SEM-EDS), the immersion method for density, mercury porosimetry, and x-ray diffraction (XRD). Density values of our samples are in the range of 69 – 99% with a porosity, in the range of 0.6 – 17%. We will present the preliminary microstructure development, density, and porosity of the waste forms along with processing limits and correlations.

### Symposium 8: Nanostructured Ceramics

#### Symposium 8: Synthesis of Nanostructured Ceramics Room: St. Laurent 4

Session Chair: Hidehiro Yoshida, The University of Tokyo

#### 9:50 AM

# (ICC-SYM8-009-2024) Fabrication and characterizations of some typical transparent ceramics from wet-chemical synthesized nano-powders (Invited)

J. Li\*1

1. Shanghai Institute of Ceramics, Chinese Academy of Sciences, Transparent Ceramics Research Center, China

Optical materials are the foundation and core component of cuttingedge scientific engineering and livelihood equipment. Transparent ceramics are polycrystalline optical materials that can transmit light with specific wavelengths and form clear images. According to the material characteristics, transparent ceramics can be divided into optical ceramics and opto-functional ceramics. The former includes transparent ceramics that transmit light in the ultraviolet, visible, and infrared wavelengths. The latter includes laser ceramics, ceramic scintillator, magneto-optical ceramics, electro-optical ceramics, ceramic phosphors. Compared with traditional optical crystals and glass, transparent ceramics have some incomparable advantages, which have important practical and potential applications in fields of infrared optical windows, solid-state lasers, optical isolators, nuclear medicine imaging, high-energy particle detection, and high brightness lighting/displays. In this paper, some typical transparent ceramics, such as garnets and sesquioxides materials, were prepared from wet-chemical synthesized nano-powders and their microstructures and properties were mainly discussed.

#### 10:20 AM

### (ICC-SYM8-010-2024) Synthesis of new Ba-rich perovskite oxide hydroxide materials by employing NSP

S. L. Kunz\*1; O. Clemens1; A. Shahzad1

1. Universitiy of Stuttgart, Institute for Material Science, Germany

Perovskite compounds ABO<sub>3</sub> exhibit a great compositional flexibility. Specific structure types can be precisely tailored by variations in both the cation sites A and B. Moreover, the anion sublattice can be substituted, for instance by fluorides or hydroxides. Wellknown examples for oxyhydroxides include Y-doped BaZrO3  $(BaZr_{1-x}Y_xO_{3-x/2})$  and  $BaInO_2(OH)$  derived from  $BaInO_{2.5}$ . Such mixed-anion materials are typically applied as proton conducting ceramics within ceramic fuel cells. When utilizing classical high temperature synthesis routes, the formation of oxide hydroxides is not expected. In our group, we observed that nebulized spray pyrolysis (NSP) can be utilized to stabilize water-rich perovskites directly, supported by the high water partial pressure during synthesis. The amount of incorporated water within the unit cell depends on the elemental composition of the cationic A and B site.  $BaFe_{1-x-y}Co_{x}Ti_{y}O_{3-d}$  show a strong tendency to crystallize within perovskite modifications with partial hcp stacking of the AO<sub>3-d</sub> layers. In this contribution, we investigate the structural changes associated with the hydration of these Ba-rich transition metal oxides by synthesis via NSP. We observe that the short reaction time in combination with the high water pressure tends to form nanoscopic compounds with a ccp-derived structure with structural deviations mainly originating from the ordering of vacancies.

#### 10:40 AM

# (ICC-SYM8-011-2024) Study of structural changes in SiVOC ceramic nanocomposites in relation to oxygen reduction electrocatalysis (Invited)

R. Kumar\*1; E. Ionescu2; S. Mathur3; K. Papakollu4

- 1. Indian Institute of Technology Madras, Metallurgical and Materials Engineering, India
- 2. Technical University Darmstadt, Materials Science, Germany
- 3. University of Cologne, Institute of Inorganic Chemistry, Germany
- 4. Indian Institute of Technology Madras, India

Precursor based nanocomposites are suitable for electrocatalysis owing to good electronic conductivity due the presence of free carbon, possibility to engineer nanostructural features and tailorable porosity. In this work, vanadium-based nanocomposites were synthesized with the objective of elucidating the effect of microstructure changes on catalytic performance for oxygen reduction reaction (ORR). For this purpose, a single-source precursor was synthesized using a polysiloxane precursor and vanadium acetylacetonate followed by pyrolysis at 1100 deg C. The resulting ceramic showed sparsely distributed vanadium carbide (VC) nanocrystallites precipitated within an amorphous SiOC matrix. Heat-treatment of the pyrolyzed samples beyond 1300 deg C led to the crystallization of  $\beta$ -SiC along with VC. This sample showed a high specific surface area of 239 m<sup>2</sup>/g. Also, the sample heat-treated at 1300 deg C gave the best performance for ORR with an onset potential and halfwave potential values of 0.81 V and 0.72 V, respectively. In addition, improved kinetics with a Tafel slope of 57 mV/dec was observed for

this sample. The superior performance of this sample was attributed to the optimal interfacial characteristics between VC and SiOC matrix along with the higher electronic conductivity caused by the free carbon effectively connecting metallic VC crystallites.

#### 11:10 AM

#### (ICC-SYM8-013-2024) Dual Loading MXene for High Sulfur Loading Cathodes for use in Lithium-Sulfur Batteries

M. Hassig\*2; V. Kalra1; M. Barsoum2

- 1. Cornell University, Chemical and Biomolecular Engineering, USA
- 2. Drexel University, Materials Science and Engineering, USA

Many countries are pledging to phase out carbon dioxide emitting vehicles. While policy makers seem keen to make the change, consumers are more hesitant. One reason is that electric vehicles (EVs) have driving ranges about 150 miles less than combustion-engine vehicles. To expand driving range, manufacturers are looking for more energy-dense batteries. Lithium Sulfur (Li-S) batteries are promising contenders as their theoretical energy density is ~2,600 Wh/kg, more than 7x that of a Li-ion battery, the current market standard. The work described herein focuses on a synthesis method combining the physical entrapment of S between Ti<sub>3</sub>C<sub>2</sub>T, multilayer MXene layers with the covalent bonding of S surface terminations. Combining chemical confinement, via termination exchange of halide-terminations, and physical confinement, via tuning of the interlayer gap with large cations, helps optimize S loading within the cathode. This increases the overall S loading to close in on the high-loading range and approaches the targeted DOE energy density value of 500 Wh/kg. The confinement also helps minimize creation of electrolyte soluble polysulfides (PS) and thus the well-known shuttle effect. Additionally, lacking PS to decompose the carbon species, we can use this technique to create Li-S cathodes compatible with carbonate-based electrolytes--the safer, EV battery industry standard electrolyte.

#### 11:30 AM

#### (ICC-SYM8-014-2024) Synthesis of Nanostructured Carbide and Nitrides Catalysts by Clusters Route or Laser Pyrolysis

G. Dubois<sup>2</sup>; F. Grasset<sup>4</sup>; C. LaGrost<sup>2</sup>; S. Surble<sup>3</sup>; P. Lonchambon<sup>3</sup>; S. Cordier<sup>2</sup>; T. Uchikoshi<sup>4</sup>; N. Herlin Boime<sup>3</sup>; F. Tessier<sup>2</sup>

- 1. CNRS, IRL3629, Japan
- 2. University of Rennes, UMR 6226 ISCR, France
- 3. CEA, NIMBE, France
- 4. National Institute for Materials Science, Japan

Transition metal carbides and nitrides demonstrate very interesting electrocatalytic properties, close to those of noble metals. Indeed, platinum being a scarce and expensive element, carbides and nitrides could be an interesting alternative to make this technology economically viable. Recently, different authors have reported promising electrocatalytic properties of molybdenum carbides for the HER reaction. For instance, when synthesized by the urea route, Mo<sub>2</sub>C shows a higher catalytic activity than other compounds (Mo<sub>2</sub>N and MoB) in acidic and alkaline aqueous media, nearby platinum. Herein we report the synthesis of Mo nitrides and carbides from original routes using transition metal cluster-based precursors or CO<sub>2</sub> laser pyrolysis. The resulting nitrides and carbides were characterized with several complementary techniques (XRD, SBET measurement, SEM, etc.). These innovative modes of synthesis afford nanostructured compounds that are evaluated as catalysts for the HER and WGS reaction.

# Symposium 11: Recent advances and the future of additive manufacturing

#### Symposium 11: Material Extrusion I

Room: Montreal 7

Session Chair: Paolo Colombo, University of Padova

#### 9:50 AM

## (ICC-SYM11-014-2024) Additive Manufacturing of Glass by Direct Ink Writing (Invited)

R. J. Dylla-Spears<sup>\*1</sup>; D. T. Nguyen<sup>1</sup>; N. Dudukovic<sup>1</sup>; T. Yee<sup>1</sup>; J. Ha<sup>1</sup>; R. Walton<sup>1</sup>; K. Sasan<sup>1</sup>; T. Fears<sup>1</sup>; D. Porcincula<sup>1</sup>; A. Browar<sup>1</sup>

1. Lawrence Livermore National Laboratory, USA

Direct ink writing (DIW), a 3D printing technique in which rheologically-tuned pastes are extruded through a nozzle, has been used to produce silica-containing preforms that can be consolidated to fully dense transparent glass. The approach offers design and production advantages for applications where simultaneous control of glass structure and composition is warranted. For example, using a mixer and multiple inks, DIW allows creation of multi-compositional glass optics, such as gradient refractive index lenses. This presentation will provide an overview of progress in 3D printing of transparent glass by DIW. The talk will highlight development of associated capabilities, current understanding, and challenges in feedstock synthesis and formulation, mass transport and mixing performance, glass consolidation, as well as materials and optical characterization. This work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344. LLNL-ABS-861170

#### 10:20 AM

### (ICC-SYM11-015-2024) ATZ/3Y-TZP Multimaterial Printing with $\mbox{DIW}$

B. Göksel\*1; E. Koos2; B. Van Meerbeek3; J. Vleugels1; A. Braem1

- 1. KULeuven, Materials Engineering, Belgium
- 2. KULeuven, Chemical Engineering, Belgium
- 3. KULeuven, Oral Health Science, Belgium

Direct ink writing (DIW) is a paste-based additive manufacturing technique where objects are built layer by layer by extruding a filament through a narrow nozzle under applied pressure. This method allows for the production of multimaterial parts, enabling compositional changes in both the xy and z directions, which are not possible with conventional processing routes. Producing an implant with an alumina-toughened zirconia (ATZ) surface and a zirconia core holds promise as it combines the LTD resistance of ATZ with the toughness of 3Y-TZP. The composition difference also induces residual stresses during cooling from the sintering temperature. Therefore, the positive influence of compressive residual surface stresses can increase strength and wear resistance. In this study, parts with ATZ surfaces and 3Y-TZP cores were produced using DIW, and the printing parameters were optimized accordingly. The design was selected based on the optimization of residual stresses with finite element analysis. Further characterization of the residual stresses was conducted using X-ray diffraction (XRD) and µRaman spectroscopy.

25

#### 10:40 AM

#### (ICC-SYM11-016-2024) Processing of water-based systems for UV-assisted direct ink writing: development and characterization of the photocurable ceramic pastes compositions

A. M. Wieclaw-Midor\*1; B. Seredynska1; P. Wiecinski1; P. Wiecinska1

1. Warsaw University of Technology, Faculty of Chemistry, Poland

The rapidly developing 3D printing technologies of recent years are increasingly used in ceramic processes. Proper adhesion between printed layers is crucial to obtaining a good quality ceramic product with a complex shape without defects. For this reason, there is a significant interest in developing the composition of such pastes dedicated to UV-DIW. The selection of appropriate organic compounds, such as monomers and dispersing agents, which allow the obtaining of pastes of high solid loading (above 50 vol%) and proper rheological behavior is necessary. In this research, the various water-based systems containing the monomer (e.g, acrylates, dimethacrylates), the photoinitiator, the ceramic powder (e.g.  $ZrO_2$ ,  $Al_2O_3$ ) and other organic additives (e.g. dispersing agent) have been prepared. The rheological behavior, stability, and cure depth have been measured. Based on this research, the best composition of ceramic paste and the curing conditions (time of exposure) have been selected. Complex-shaped samples were obtained using the UV-assisted direct ink writing method. The selected properties were examined for the sintered parts, and the microstructure observations were carried out. Acknowledgment Research was funded by the Warsaw University of Technology within the Excellence Initiative: Research University (IDUB) programme.

#### 11:00 AM

#### (ICC-SYM11-017-2024) Preparation and Characterization of 3D Printed Ceramic Electrolyte for Batteries

M. Faral\*1; A. Laventure1; M. Dollé1

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.

#### 11:20 AM

# (ICC-SYM11-018-2024) Comparison of rheology and printability between aqueous ceramic slurries for material extrusion additive manufacturing

J. Kaufman\*1; C. Wyckoff1; L. Rueschhoff2

- 1. UES, Inc., USA
- 2. Air Force Research Lab, USA

The additive manufacturing technique of material extrusion is of interest due to the ability to fabricate of robust near-net shaped ceramic parts with tailored microstructures. Here, we report a comparison of a variety of ceramic compositions of interest within the aerospace application field. This work will detail the process of modifying the rheological properties to reach the 'printability window' of silicon nitride, silicon carbide and zirconium diboride based aqueous slurries. The large variations in rheological properties of the different ceramics will be discussed and tied to printability and sintered microstructure. Ceramics components were fabricated from all three slurry compositions to densities ranging from 93%-96% relative density via pressureless sintering to maintain the complex shapes formed during printing. In addition to near-net shaping, microstructure control in the form of alignment of fibers added as a reinforcing phase is possible. We will report on the addition of fibers to the silicon carbide and zirconium diboride slurries and the resulting effect on rheology and fiber alignment from the printing process.

#### 11:40 AM

### (ICC-SYM11-019-2024) Eco-friendly ceramic feedstocks for additive manufacturing

L. Hu<sup>\*1</sup>; A. Anagri<sup>1</sup>; P. Vuillaume<sup>1</sup>

1. COALIA, Plastics, Canada

Owning to their excellent resistance to wear, heat and corrosion, technical ceramics find applications in various sectors including aerospace, chemistry and biomedicine. However, ceramics are more difficult to be processed than metals and plastics because of its high melting temperature. Additive manufacturing is considered as a promising avenue to create ceramic parts of any shape. Unlike conventional shaping processes (pressing, injection molding, extrusion), ceramic additive manufacturing consists of creating desired forms layer-by-layer from computer-aided design (CAD) models, follows by a series of discrete production steps: green body machining, debinding, sintering, final machining, polishing. Organic binders, mainly composed of petrochemical-based polymers, are essential for keeping the shape of additive manufactured parts. These binders must be mostly burned in a time and cost-consuming manner before sintering of ceramic particles, thus leading to energy efficiency and environmental concerns. This study addresses the potential of using organic bonders derived from renewable resources. Meanwhile, supercritical CO2 debinding and thermal debinding processes were compared. It is expected to improve the sustainability of current ceramic additive manufacturing.

# Symposium 1: Aerospace Ceramics and Composites

#### Symposium 1: Aerospace Ceramics and Composites Room: Montreal 1-3

Session Chair: Zbigniew Pedzich, AGH University of Krakow

#### 1:30 PM

# (ICC-SYM1-020-2024) Ceramics in Space: The role of advanced ceramics and ceramic matrix composites on the Dream Chaser Space Plane (Invited)

R. Fahsbender\*1

1. Sierra Space, Advanced Materials, Manufacturing, and processes, USA

Ceramics and ceramic matrix composites are enabling materials for spacecraft. With the emergence of commercial space, a renewed focus on accessibility and cost of these material is taking place. This push to lower cost is occurring while reusability in spacecraft is simultaneously driving a desired increase in material performance. Application of advanced composite structures, novel thermal protection systems, and ceramic matrix composites are all needed to deliver the next generation of reusable spacecraft. Sierra Space, a leading commercial space company is developing the Dream Chaser, a reusable space plane that will become operational this year. The primary mission of the first Dream Chaser is delivery of cargo to the International Space Station and return of cargo to earth under the NASA CRS2 program. To increase performance and lower operational costs, Sierra Space is actively applying the next generation of ceramic materials and structures on the Dream Chaser platform. Future Dream Chaser variants will leverage emerging trends and potential breakthroughs that will change spacecraft fundamental design and capabilities. The goal of this presentation is to present the critical role these materials play in the advancement of spacecraft design and operation and the challenges and opportunities that lie ahead.

#### 2:00 PM

#### (ICC-SYM1-021-2024) Carbon Nanotube - Polysiloxane Nanocomposite for Ablative Thermal Protection

- P. B. Patel\*<sup>1</sup>; S. Kim<sup>2</sup>; J. Dai<sup>1</sup>; J. Buffy<sup>3</sup>; V. L. Wiesner<sup>4</sup>; J. Koo<sup>2</sup>; B. Wardle<sup>1</sup>
- 1. MIT, USA
- 2. The University of Texas at Austin, USA
- 3. Techneglas LLC, USA
- 4. NASA Langley Research Center, Advanced Materials and Processing Branch, USA

Ultra high temperature resins (UHTR) such as polysiloxane possess excellent characteristics for ablation including low thermal conductivity, lightweight composition, high thermal resistance, and its ability to form a durable char layer. To enhance its mechanical strength, the aerospace industry utilizes fiber reinforcement, such as carbon fiber reinforced polysiloxane (CF-polysiloxane), due to its high strength-to-weight ratio. Carbon nanotubes (CNTs) have advantages over microfibers including higher specific strength, lower density, and customizable thermal conductivity. Their nanoscale dimensions allow for a more homogenous material compared to traditional microfibers. Conventional methods of incorporating CNTs into resins often have structural defects such as voids, CNT agglomerations, and randomly dispersed CNTs, resulting in suboptimal improvements in properties. A novel approach known as bulk nanocomposite laminating (BNL) has been introduced. involving CNT densification, resin polymer infiltration, and pressurized curing to achieve a high packing density exceeding 35% by volume. Characterization and testing to simulate re-entry conditions is performed with a sample of CNT-polysiloxane nanocomposite cured onto a CF-polysiloxane backing. These samples are subjected to ablation by an oxyacetylene flame while collecting temperature data to evaluate thermal protection properties.

#### 2:20 PM

## (ICC-SYM1-022-2024) Synthesis and Characterization of High Entropy Carbonitrides

L. Backman<sup>\*1</sup>; H. Keshmiri<sup>1</sup>; S. C. Mills<sup>2</sup>; E. Patterson<sup>2</sup>; H. Ryou<sup>2</sup>;

J. Wollmershauser<sup>2</sup>; E. Gorzkowski<sup>2</sup>; J. Maxwell<sup>1</sup>

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

entropy systems, for performance in high temperature environments. Material system candidates will be reviewed, with a special focus on carbonitride high entropy ceramics. Experimental work on the synthesis and characterization of high entropy carbonitrides will also be presented.

#### 2:40 PM

#### (ICC-SYM1-023-2024) Investigation of O-SiAlON Materials for Oxidation Barrier on Silicon Nitride-Based Ignition-Assisted Devices in Small Aircraft Engines

P. Numkiatsakul\*1; T. Lee2; K. Kim3; C. Kweon3; W. M. Kriven4

- 1. University of Illinois at Urbana-Champaign, Materials Science and Engineering, USA
- 2. University of Illinois at Urbana-Champaign, Mechanical Science and Engineering, USA
- 3. Combat Capabilities Development Command Army Research Laboratory, USA
- 4. University of Illinois at Urbana-Champaign, USA

Silicon nitride-based ignition-assisted devices, such as glow plugs, experience significant degradation above 1000°C, rendering them unsuitable for next-generation small jet-fuel aircraft engines designed for lower-quality alternative fuels. The increased demand for continuous operation in combustion environments highlights the critical need for improved oxidation resistance in  $Si_3N_4$ . This study centers on O-SiAlON, a solid solution SiAlON ceramic with the highest oxygen content phase, aiming to improve the oxidation resistance of Si<sub>3</sub>N<sub>4</sub>. The investigation explores the intrinsic and processing properties of O-SiAlON materials as potential candidates for an oxidation barrier coating on bulk Si<sub>3</sub>N<sub>4</sub>, with a particular focus on the effects of reaction-sintered O-SiAlON composition and sintering parameters on density, final microstructures, and oxidation resistance. Samples, fabricated through pressure-less sintering with a Y<sub>2</sub>O<sub>3</sub> sintering aid, underwent isothermal oxidation coupon testing and were analyzed using XRD, SEM, and EDS. The oxidation weight gain curve indicates superior oxidation resistance in O-SiAlON compared to Si<sub>3</sub>N<sub>4</sub> when oxidized at 1300°C for 50 hours.

# Symposium 2: Electronic Ceramics and Devices

#### Symposium 2: Electronic Ceramics and Devices

Room: St. Laurent 3 Session Chair: Yukio Sato, Kumamoto University

#### 1:30 PM

## (ICC-SYM2-015-2024) Millimeter wave dielectric materials derived from ZSM-5 zeolite (Invited)

X. Chen\*<sup>1</sup>; J. Shi<sup>1</sup>; T. Sun<sup>2</sup>; F. Zhou<sup>1</sup>; X. Zhu<sup>1</sup>; L. Li<sup>1</sup>

- 1. Zhejiang University, School of Materials Science and Engineering, China
- Zhejiang University of Technology, China

In order to search low-k dielectric materials, ZSM-5 zeolite powder compacts were sintered at temperatures ranged 1200~1300°C, and the microwave dielectric characteristics were investigated together with the structure evolution. It was failed to obtain ZSM-5 ceramics by high temperature sintering because the densification could never occur below the tolerable temperature and the ordered porous structure completely collapsed above the tolerable temperature, The structure changed from ZSM-5 zeolite to SiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub> glass at 1240°C, and finally to SiO<sub>2</sub>-based cristobalite ceramics above 1275°C. A low dielectric constant (2.63-2.78) combined with low dielectric loss (<0.0005 at 18GHz) and small temperature coefficient of resonant frequency (varied from -18.6 ppm/ °C to -8.1 ppm/ °C) were achieved in the present materials, and the value of dielectric loss was the lowest one achieved so far in the dielectric materials with a dielectric constant lower than 3. With the structure evolution from

ZSM-5 zeolite to  $SiO_2$ -Al<sub>2</sub>O<sub>3</sub> glass and finally to  $SiO_2$ -based cristobalite ceramics, the dielectric constant increased from 2.27 to 2.78, while the Qf value indicated a complicated variation tendency, and the higher Qf values were achieved in amorphous and crystalline states, while significant drop was determined in partial amorphous state. The present materials could be expected as promising candidate for millimeter wave dielectrics.

#### 2:00 PM

#### (ICC-SYM2-016-2024) Phase Manipulation of Epitaxial Thin Films by Thermal Technique Based on Sol-Gel Deposition

Y. Cheng\*<sup>1</sup>; L. Liu<sup>2</sup>; J. Luo<sup>2</sup>; Y. Huang<sup>1</sup>; L. Shu<sup>1</sup>; X. Zhang<sup>1</sup>; Z. Yang<sup>1</sup>; J. Li<sup>1</sup>

1. Tsinghua University, School of Materials Science and Engineering, China

2. Nanjing university of science and technology, China

Thin films with good piezoresponse and large electric field induced stain is highly desired for actuators and Micro-Electro-Mechanical Systems. However, many techniques that have been applied in constructing multiphase boundary in bulks is inaccessible for epitaxial thin films, given the challenge in fabricating epitaxial thin films with complex component. In this work, Li, Ta, Ca, Zr, Mn co-doped epitaxial thin films with good epitaxy are grown by sol-gel deposition. Furthermore, by tuning the thermal technique during the chemical deposition process, KNN-based epitaxial thin films with single or multiple phases can be obtained. Great increments in piezoresponse and strain are exhibited for the multiphase coexistent thin films. In addition, excellent stability in large range of frequency (0.5 kHz - 500 kHz) and temperature have also been demonstrated, exhibiting great potential in actuators for harsh environment. This work provides perspectives in growth of epitaxial thin films by sol-gel deposition and phase manipulation by thermal techniques.

#### 2:20 PM

## (ICC-SYM2-017-2024) High-Entropy Electroceramics: $A_6B_2O_{17}$ (A = Zr, Hf; B = Nb, Ta) Phases

R. Spurling\*1; J. Maria1

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

We report on dielectric properties of novel, disordered A<sub>6</sub>B<sub>2</sub>O<sub>17</sub> (A = Zr, Hf; B = Nb, Ta) phases across the bulk and thin film regimes. We provide an experimental procedure for thin film sputter deposition from densified bulk ceramic targets. We note sensitivities of the crystallinity, density, roughness, and electrical properties of as-deposited films to deposition conditions, particularly total pressure and oxygen partial pressure. We outline relationships between the structure (as characterized by X-ray diffraction, atomic force microscopy, scanning electron microscopy, X-ray photoelectron spectroscopy, and related techniques) and the electronic properties of thin films. We measure dielectric permittivities of ~60 and loss tangents in the 10<sup>-3</sup> range across microwave frequencies in ternary permutations of  $A_6B_2O_{17}$ . We also probe the temperature dependence of this electronic behavior. Furthermore, we explore high-entropy and extended solubility effects in these systems using additional cation species, paying particular attention to trends in electronic behavior. We confirm the polar structure of these phases and probe candidate ferroelectricity at high fields.

#### 2:40 PM

## (ICC-SYM2-018-2024) Effect on Nickel on structural characterizations and properties of $PaBaFe_2O_{5+\delta}$

A. Akbarifakhrabadi\*1; G. Escobar1

1. University of Chile, Mechanical Engineering, Chile

Mixed ionic and electronic conducting oxides with perovskiterelated structures have attracted significant attention due to extensive applications for solid oxide cells and oxygen separation membrane reactors as highly efficient electrochemical energy conversion devices. Among all MIEC oxides, double perovskites  $PaBaFe_2O_{5+\delta}$ (PBFO) as potential cobalt-free, iron-rich and cost-effective mixed ionic and electronic conductive perovskite oxides for air electrode in SOCs with improved long-term stability and high compatibility with other cell components as well as favorable electrochemical performance at intermediate temperatures is becoming a new research trend. Numerous efforts have been directed toward enhancing its performance through B-site doping and partial substitution of Fe with transition metals such as Nickel. Additionally, as NiO has been identified as a sintering aid for oxides with refractory properties, effectively improving their sinterability, in this work,  $PaBaFe_{2-x}Ni_{x}O_{5+\delta}$  (x: 0-0.15) synthesize via sol-gel method. Furthermore, NiO powders are also used as sintering aid for calcined  $PaBaFe_{2-y}O_{5+\delta}$  (y: 0-0.1) powders to perform densification studies. Structural characterizations and Rietveld refinement analyses are performed using XRD techniques. After fabrication, comprehensive characterizations of their mechanical, electrical, and thermal expansion properties will be conducted.

#### 3:30 PM

#### (ICC-SYM2-019-2024) Ceramics for Electrostatic Chucks (ESC): Plasma Etch & High Volume Resistivity

J. Santillan\*1

1. Watlow, Engineering, USA

Transient liquid-phase (TLP) sintering of inorganic salts as additives on the densification behaviors and microstructural development of AlN ceramics are investigated. It is found that <1 wt% additive (s) of CaF<sub>2</sub> in AlN-Al<sub>2</sub>O<sub>3</sub>-TiO<sub>2</sub> can effectively promote densification process. Increasing content of the additive results in finer grain size and slower densification during intermediate sintering stage. XRD results show that grain-boundary phase is formed at low temperatures ~ 1150C from reactions of AlN. With further temperature increasing, the grain-boundary phases of are formed from the solid state diffusion at higher temperature. Literature results show that formed grain-boundary phases can evaporate from sintering bodies during further soaking, leaving clean grain boundaries with segregated Spinel structures. The efficiency of TLP sintering mechanism is further manifested by the processing and process preparation of AlN ceramics with good combination properties.

#### 3:50 PM

### (ICC-SYM2-020-2024) Effect of binder chemistry on the transfer of vibration energy

- O. Kwon\*1; S. Park1; J. Lee1; H. Kim2; K. Chung2; S. Bae2; C. Bae1
- 1. Korea Institute of Materials Science, Republic of Korea

2. Kyungwon Industry, Republic of Korea

Haptic sensors delivery the sense of touch by combinations of force, vibration and motion, experiencing fully immersive virtual reality. In order to transmit strong, noise-free vibration signals from piezo-ceramics to metal boosters, selecting a proper binder system is one of the most important steps. Here, we have investigated ways how to enhance the propagation of vibration energy according to the chemical structure and electric and vibration behaviors of binders. Chemical structure of matrix was tailored based on the consideration of epoxy-based binders, associating to flexibility or hardness and higher curing temperature by aliphatic or aromatic curing agents, respectively. The electrical interconnections and percolation were secured by adding low-specific gravity graphite into epoxy matrix, achieving large surface area and percolation threshold. Finally, vibration behaviors of binder chemistry have been represented as functions of crosslinking density and rigidity, resulting in lower damping factors and propagating higher vibration energy.

#### 4:10 PM

#### (ICC-SYM2-021-2024) Dry Aerosol Deposition of Nanostructured Barium Rare Earth Titanate Dielectric Ceramics

P. A. Fuierer<sup>\*1</sup>; A. Valdez<sup>1</sup>

1. New Mexico Tech, Materials Engineering, USA

The purpose of this study was to demonstrate the viability of Dry Aerosol Deposition (DAD) for agile and on-demand manufacturing of electronics (ODME). DAD is an emerging additive manufacturing spray process for building fully dense, nanostructured ceramic coatings and low profile 3D structures directly from dry powder without the need for binder or liquid medium. DAD relies on kinetic energy of particle impact rather than high temperature sintering for densification; thus, functional ceramics can be deposited directly on a variety of substrate materials. This study focused on barium neodymium samarium titanate, a high-K microwave dielectric ceramic of interest for low loss resonators in RF/microwave communication modules. Using a custom built deposition system and commercial feedstock powder, very thick, fully dense and robust films were successfully deposited onto glass, copper and printed circuit board substrates. Dielectric constant (K) and loss were measured over a wide frequency range to 1 GHz via impedance bridge method. Results suggest a thickness dependence for K, with thicker films approaching a value of about 40, roughly half that for conventionally sintered (1340°C) bulk ceramic. Copper metallization with high conductivity was also demonstrated in the same apparatus, suggesting that DAD is a promising method for ODME.

### Symposium 4: Green ceramics for clean energy and sustainability

#### Symposium 4: Energy Ceramics and Green Processes Room: Montreal 6

Session Chair: Eva Hemmer, University of Ottawa

#### 1:30 PM

#### (ICC-SYM4-019-2024) Carbon Sequestration Using Enhanced Mineralization in Ceramics (Invited)

B. P. Gorman<sup>\*1</sup>

1. Net Negative, LLC, USA

In addition to reducing anthropogenic carbon emissions, carbon sequestration is anticipated to also be needed in order to meet carbon change goals. Sequestration of carbon through enhanced mineralization is a potential route to meeting these goals, however, the CO2 uptake is rate limited by surface area effects. In this work we show that highly reactive minerals can be incorporated into bricks, tiles, and other products with carbonate precipitates being formed within microstructural porosity. Commercial production of bricks incorporating particulates of these sequestration materials is possible without any process modifications and improved extrusion. Welldispersed microstructures with frequent pore - CO2 mineralization interfaces are produced while meeting all applicable ASTM standards for water absorption, compressive strength, and freeze-thaw cycling. Exceptionally large surface areas are achieved through adaptation of these products in enviroment-facing construction materials and could result in >10 Mtons of CO2 sequestered per year in the US alone. Globally, CO2 sequestration in ceramics could be greater than 1 Btons per year using materials widely available in the earth's crust.

#### 2:00 PM

# (ICC-SYM4-020-2024) Structure Property Relations in Hexagonal Perovskite Proton Conductors of the form $Ba_5Er_2Al_2ZrO_{13}$ (BEAZ) and $Ba_5In_2Al_2ZrO_{13}$ (BIAZ)

K. Brinkman\*1

1. Clemson University, Materials Science and Engineering, USA

Development of novel systems with the ability of high proton conduction is essential for the advancement of hydrogen-based energy technologies such as protonic fuel cells, electrolyzers, and hydrogen membranes. The  $Ba_5Er_2Al_2ZrO_{13}$  (BEAZ) system was first reported by Murakami et al. in 2020. The system was found to form the  $P_3$ /mmc space group, as was seen in previous structural studies in layered systems. Water uptake, and therefore proton incorporation is assumed to occur in the intrinsic oxygen deficient layer in this system resulting in high level of proton conductivity. This work will examine synthesis and structural studies over a fixed compositional range in  $Ba_5Er_2Al_2ZrO_{13}$  (BEAZ) and related  $Ba_5In_2Al_2ZrO_{13}$ (BIAZ) systems followed by electrochemical characterization aimed at understanding the relative electron and ionic transport properties in these systems.

#### 2:20 PM

#### (ICC-SYM4-021-2024) Perovskite Ceramic Honeycombs for High-Temperature Thermal/Thermochemical Heat Storage

- G. Karagiannakis\*1; C. Pagkoura1; V. Mitrousis2; L. Chasapidis1; K. Fotiadis1
- 1. Centre for Research & Technnology Hellas (CERTH), Chemical Process & Energy Resources Insitute (CPERI), Greece
- 2. Aristotle University of Thessaloniki (AUTH), Chemical Engineering Department, Greece

The study relates to experimental results on preparation of extruded honeycomb-like structures and their ability to function as effective means for thermal/thermochemical heat storage. The latter occurs via a cyclic reduction-oxidation (redox) scheme and can facilitate storage of high temperature (>1000°C) heat, in-principle applicable to various industrial processes. Stable cylindrical honeycombs (OD=25 mm) of 2 different geometries, were prepared entirely from calcium manganite (CaMnO3). Structures were experimentally validated for their thermochemical heat storage functionality upon cyclic electrical heating up to 1100°C under air flow, followed by cooling in nitrogen and isothermal oxidation in air at 750°C. As the structure is heated up, CaMnO3 is reduced in a quasi-continuous/nonstoichiometric manner to obtain the form of CaMnO3- $\delta$  ( $\delta$  indicates the reduction extent). Reduction is documented by oxygen evolution as measured by an oxygen analyzer. This step is endothermic and stores heat which is subsequently released by the exothermic oxidation. Experiments demonstrated the ability of such structures to store and release heat defined by the enthalpy of redox reaction and thermochemical storage density was calculated at 150 J/g. To the authors' knowledge, this is the first time that the ability of a perovskite to generate repeatable heat effects upon cyclic redox operation is demonstrated.

### Symposium 7: Novel Processing Techniques

### Symposium 7: Additive, near net shaping and related

precursor engineering Room: Montreal 8

Session Chairs: Babak Anasori, Purdue University; Carolina Tallon, Virginia Tech

#### 1:30 PM

## (ICC-SYM7-017-2024) Additive manufacturing of Ceramics from Preceramic Polymers (Invited)

P. Colombo\*1

1. University of Padova, Industrial Engineering, Italy

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

#### 2:00 PM

## (ICC-SYM7-018-2024) Volumetric additive manufacturing of SiOC by xolography

K. Huang<sup>1</sup>; G. Franchin<sup>1</sup>; P. Colombo<sup>\*1</sup>

1. University of Padova, Industrial Engineering, Italy

Additive manufacturing (AM) of ceramics suffers from defects induced by the layer-by-layer approach on which traditional AM techniques are based. This study presents volumetric AM of a SiOC ceramic from a preceramic polymer using xolography, a linear volumetric AM process that allows to avoid the staircase effect. Besides optimizing the trade-off between preceramic polymer content and transmittance, a pore generator was introduced to create transient channels for gas release before decomposition of the organic constituents and moieties, resulting in crack-free solid ceramic structures even at low ceramic yield. The viscosity of the resin formulation was optimized without compromising transmittance, alleviating sinking of printed parts during printing and preventing shape distortion. With the optimization of printing parameters, including UV irradiance and moving speed, solid and porous ceramic structures with a smooth surface and sharp features were fabricated. This work provides a new method for the AM of ceramics at µm/mm scale with high surface quality and large geometry variety in an efficient way, opening the possibility for applications in fields such as micromechanical systems and microelectronic components.

#### 2:20 PM

# (ICC-SYM7-019-2024) The weight loss mechanism and thermal deformation behavior of the SiC precursor obtained via photolithography

Y. Lee\*1; D. Shin1; J. Kim1

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

The development of AM for ceramic materials has been less rapid than for other materials, and has mainly been concentrated on apatite, alumina, and stabilized zirconia. The AM of SiC ceramic, a material with high development demand in the industry, is being developed through printing methods such as filament extrusion process, binder jetting, and laser sintering. The difficult sintering properties of this material are a significant obstacle to AM development, which is why photolithographic printing methods using silicon-based polymers as precursors has been suggested as a remedy. Even in this technology, it is necessary to understand the conversion behavior of polymers into ceramics. While mass loss is commonly believed to be the sole cause of thermal deformation in ceramic precursors, polymer precursors have thermal expansion behavior and different mass loss mechanisms depending on temperature. The objective of this study is to investigate the shape deformation that occurs when printed materials are subjected to heat treatment. We confirmed that the geometric model and gas generation conditions are the main causes of shape deformation and defects, and that curing conditions, heat treatment temperature, and pressure changes can control thermal deformation of the printed shape.

#### 2:40 PM

#### (ICC-SYM7-020-2024) In situ formation of iron-nickel alloys and silicides in a polymer-derived Si-C-O-N matrix to promote electrocatalytic water oxidation *WITHDRAWN*

M. Fradin<sup>\*3</sup>; G. Soldi De Souza<sup>1</sup>; M. Ben Miled<sup>3</sup>; A. Habrioux<sup>2</sup>; S. Celerier<sup>2</sup>; O. Masson<sup>3</sup>; S. Bernard<sup>3</sup>

- 1. Federal University of Santa Catarina, Brazil
- 2. Institut de Chimie des Milieux et Matériaux de Poitiers IC2MP UMR7285, France
- 3. Institute of Research for Ceramics (IRCER) UMR7315, France

The production of hydrogen through electric water splitting is considered as a promising green energy alternative. Due to multiple electron transfers in alkaline media occurring during the Oxygen Evolution Reaction (OER), the kinetics of the whole process are low. Noble metals (NM) have been so far considered as excellent electrocatalysts. However, their high costs, low abundance and low stability in several media require today to find alternative efficient materials. Recently, transition metals (TM) (Ni, Fe, Co, Zr) and nanostructured TM (carbides, silicides, nitrides) have gained attention in this field, competing each drawback of NM. Yet, their synthesis at nanoscale is challenging. This work utilizes the Polymer-Derived Ceramic (PDC) route to grow Ni<sub>x</sub>Fe<sub>y</sub> alloys and Ni<sub>x</sub>Fe<sub>y</sub>Si<sub>z</sub> silicides nanoparticles inside a ceramic Si-C-O-N matrix. PDCs provide a new approach to produce micro/mesoporous ceramics with a high surface area that can be used as electrode materials. The highly reactive Si-H and/or N-H bond(s) in polymers' backbone is(are) prone to link and/or coordinate with TM complexes in solution, forming a TM-containing pre-ceramic polymer (PCP). After heat treatment at low temperatures, the PCP becomes a porous ceramic containing nanoparticles of NiFe alloys and silicides. All of these elements combined are expected to boost OER performance.

#### 3:30 PM

### (ICC-SYM7-021-2024) Polysilazane-derived SiON coating on stainless steel weld for corrosion resistance

K. Lu\*1; H. Choi2

- 1. University of Alabama at Birminghngham, USA
- 2. Virginia Tech, USA

Dry storage systems used for storing spent nuclear fuel (SNF) is vulnerable to stress corrosion cracking (SCC) caused by corrosive environments and physical stress. This is especially the case for the weld region with inherent physical stress. Protective coatings can be applied to mitigate stress corrosion cracking. Perhydropolysilazane (PHPS), a polymer-derived ceramic, is a promising material for corrosion prevention coatings. In this study, SiON coatings were created using PHPS on stainless steel substrates with non-uniform microstructure and composition from welding. The influences of substrate composition and microstructure on coating formation, mechanical properties, composition, and corrosion resistance are discussed.

#### 3:50 PM

# (ICC-SYM7-022-2024) An investigation into the impact of $Al_2O_3$ particulate morphology on the rheological flow properties of backbone polymers

E. Valenzuela-Heeger\*<sup>1</sup>; J. Binner<sup>2</sup>

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

The design and creation of ceramic slurries is often considered a balanced art of powder content, dispersants, and binders (known as backbone polymers) within an often-aqueous suspension. It has been well established that particle size has a specific influence on the flow properties of a slurry; however, the physical properties of a particle's morphology play an equally important role. The aim of this research was to assess Al<sub>2</sub>O<sub>3</sub> powders for their physical descriptors via particle shape and SEM analysis to highlight the physical differences between the tested powders. The powders were combined with different backbone polymers and subjected to varying shear rates prior to conducting comparable tests on complete slips with the same backbone polymers. Samples were frozen in situ and analysed via microstructural imaging to formulate statistical trends in particulate movement, orientation, and agglomeration. The results were analysed to investigate trends and factors to assess the most suitable particulate morphology for industrial applications where high-pressure movement is required for ceramic processing.

#### 4:10 PM

## (ICC-SYM7-023-2024) Role of the Monomer in Gelcast Green and Sintered Alumina Parts

J. Heintz\*1; L. Gauzere3; C. Besnard2; S. Couillaud2

- 1. ENSMAC-Bordeaux INP, ICMCB, France
- 2. Galtenco Solutions, France
- 3. ICMCB, France

In recent years, several powder shaping techniques have been developed to facilitate the production of net-shaped ceramics. Gelcasting suspensions, in particular, have the advantage of being highly charged with ceramic powder (>50 %vol.) while remaining sufficiently fluid to be cast into molds of any shape and size, resulting in the production of large, complex ceramic parts. Our study examines the impact of the chemical nature of the monomer on the quality of granular packing in green alumina samples and their subsequent sintering behavior. Three monomers from the same chemical family were studied: acrylamide (AM), methacrylamide (MAM), and dimethylacrylamide (DMAA). The results indicate that, under the same suspension preparation conditions, the relative green densities were close. But, X-ray tomography revealed slight packing heterogeneities, which were confirmed by mercury intrusion porosimetry.

### Symposium 8: Nanostructured Ceramics

# Symposium 8: Synthesis and Properties of Nanostructured Ceramics

Room: St. Laurent 4

Session Chairs: Jiang Li, Shanghai Institute of Ceramics, Chinese Academy of Sciences; Christian Moreau, Concordia University

#### 1:30 PM (ICC-SYM8-015-2024) Thermodynamics of Nanostructured Ceramics (Invited)

#### A. Navrotsky\*1

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

Many modern ceramics are produced through nanophase intermediates, with small grain size remaining even in agglomerated or sintered materials. The formation of such ceramics and their physical and catalytic properties depend, in both thermodynamic and kinetic contests, on particle size, with surface and grain boundary energies main factors determining stability and persistence in use. This talk summarizes the use of calorimetry in determining these energetic parameters and highlights systematics in the variation of surface and grain boundary energies with composition and structure. Differences in these parameters can cause crossovers in phase stability at the nanoscale. The interface energy of a given material is typically 30-50 % of its surface energy. It is noteworthy that spinels often have low surface and interface energies while values for complex silicate structures are relatively high.

#### 2:00 PM

### (ICC-SYM8-016-2024) Nano-Phase Diagrams for Controlling Nanocrystalline Ceramics

#### R. Castro\*<sup>1</sup>

1. Lehigh University, Material Science & Engineering, USA

One of the greatest challenges in the field of nanostructured ceramics is the design of structures with predictable and stable features. Despite various synthesis methods available, these ceramics often exhibit poor stability, especially in high-temperature applications. Traditional phase diagrams fall short in guiding the design of stable nanoceramics due to their failure in accounting for crucial factors like interfaces. The interface energies—spanning surfaces and grain boundaries—are pivotal in defining the metastability of nanostructured compounds. These energies, influenced by composition, segregation tendencies, and temperature, play a crucial role. In our presentation, we share recent pertinent data on nanoceramic phase diagrams, highlighting the critical role of interfacial energies and chemical heterogeneities in nano-oxides. This discussion aims to draw attention to this burgeoning field, crucial for controlling the properties of nanostructured ceramics.

#### 2:20 PM

## (ICC-SYM8-017-2024) Using Vapor Phase Infiltration to Form Nanoceramic Clusters Inside of Polymers (Invited)

M. D. Losego\*1

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

Vapor phase infiltration (VPI) exposes polymers to inorganic vapors that sorb and diffuse into the polymer to transform it into an organic-inorganic hybrid material. This talk will discuss our current understanding of the VPI process kinetics which require a combination of sorption, diffusion, and reaction, and the chemical, physical, and electronic structure of the infiltrated inorganic species. Interestingly, for certain chemistries these inorganics form primary chemical bonds to the polymer, cross-linking the polymer and affecting its chemical and thermophysical properties; in other cases, the inorganics become unbound from the polymer and can effectively mineralize with themselves to form larger inorganic clusters. These structural changes have been studied with advanced spectroscopies including EXAFS and XANES and by probing changes in physical properties such as thermal expansion and chemical dissolution. Recently, we have also demonstrated that photoexcited electrons can be injected from the conducting polymer P3HT into infiltrated TiO<sub>2</sub> nanoclusters and be used for photocatalysis. These results demonstrate an opportunity to engineer the electronic structure in these hybrid materials as well.

#### 3:20 PM

## (ICC-SYM8-018-2024) Tailoring MXene Synthesis for Enhanced Performance (Invited)

Y. Baek<sup>2</sup>; K. Pakravan<sup>2</sup>; M. Beidaghi<sup>\*1</sup>

- 1. University of Arizona, Aerospace and Mechanical Engineering, USA
- 2. Auburn University, Materials Engineering, USA

Two-dimensional (2D) MXenes have garnered significant attention in recent years due to their exceptional properties and wide-ranging potential applications. This presentation showcases our group's recent research on understanding how the synthesis process can be tuned to enhance MXenes' electrochemical, mechanical, and membrane performance. Our studies show that the synthesis process has a profound impact on MXene morphology, defect concentrations, and surface chemistry, ultimately influencing their electrical, electrochemical, and mechanical properties. Understanding the impact of synthesis conditions on the properties of MXene can help in improving their performance for various applications. Also, given the substantial impact of synthesis on MXene properties, the development of standardized synthesis protocols is imperative, as is the establishment of consistent performance reporting protocols across various fields of application. This presentation illustrates our research findings pertaining to the formulation of strategies aimed at enhancing MXene-based device performance through innovations in MXene synthesis.

#### 3:50 PM

#### (ICC-SYM8-019-2024) Observing The Effects Of Co-dopants and Energy Mediating Middle Shells on The Emission Spectra Of Upconverting Nanoparticles

A. Dutta\*1; M. Liu1; F. Vetrone1

1. Institut National de la Recherche Scientifique, Canada

The unique ability of rare earth doped nanoparticles to convert low energy light into higher energy light by a process known as upconversion have made them ideal candidates for theranostic (therapy + diagnostic) applications in the biomedical field. Previously, optically triggered drug release in deep tissue was severely hindered. Specifically, the high energy Ultraviolet (UV) light needed to trigger photochemical reactions lacked the general penetration depth, whilst low energy Near-Infrared (NIR) light has the penetration depth but not the energy. When upconverting nanoparticles (UCNPs) are irradiated with near infra-red (NIR) light they can convert the incident NIR light into the higher energy UV light needed to trigger the photochemical processes for drug release. Here we report on two different methods used to create UCNPs tuned to have more intense peaks in the UV region. We use a standard LiYbF<sub>4</sub> (0.5% Tm<sup>3+</sup>) as a base matrix and manipulate co-dopant concentrations and shell compositions to enhance and increase the peaks of the UV regions. After co-doping with all other rare earth elements, two ions, namely,  $Eu^{3+}$  and Tb<sup>3+</sup> showed higher intensity UV emission. Furthermore, after utilising a middle shell that acted as an energy mediating shell between the light absorbing outer shell, and the light emitting inner shell, the emission was also augmented.

# Symposium 11: Recent advances and the future of additive manufacturing

#### Symposium 11: Material Extrusion II

Room: Montreal 7

Session Chair: Rebecca Dylla-Spears, Lawrence Livermore National Laboratory

#### 1:30 PM

## (ICC-SYM11-021-2024) Additive manufacturing of geopolymers with biomimetic structures (Invited)

P. He\*1

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

3D printing of geopolymers with desirable patterns, compositions, and properties holds great promise for sustainable construction materials, porous adsorbent, and high-temperature ceramics. Current research mainly focuses on the rheological behavior of geopolymer slurry, 3D printing of geopolymer with high resolution, and hierarchical structures. In this paper, we reported a universal method to realize direct ink writing of geopolymer of bionic structures were prepared and strengthening and toughening mechanisms were investigated. Current results proved that 3D printing together with bionic-structure designing provide a novel method for the composites of lightweight, high strength & toughness, and superior impact resistance, which would lead to a resurgence of interest in new lightweight and reliable structure design strategies.

#### 2:00 PM

#### (ICC-SYM11-022-2024) Synthesis and Robocasting of Hydroxyapatitte, Tricalcium phosphate and Wollastonite based composites

G. Rajan\*1

1. Anna University, India

Tissue engineering (TE) plays a pivotal role in addressing organ regeneration, particularly in the context of bone reconstruction, which is often necessitated by trauma, neoplasms, and aging. Our research is centered on pioneering biomaterials in bone tissue engineering (BTE), specifically tricalcium phosphate (TCP), hydroxyapatite (HAp), and wollastonite (WS). The synthesis of HAp involves repurposing waste biomaterials, namely orange peels and sugarcane bagasse, augmenting ceramic powders to enhance tissue regeneration. The utilization of direct ink writing (DIW) facilitates the creation of 3D structures at room temperature. Meticulous heat treatment ensures sintering structural integrity while minimizing cracks. Subsequent evaluations comprehensively cover physiochemical, surface, and mechanical properties, guiding both in vitro and in vivo testing phases. Our research initiative, "GREEN PRINT HEALTH," serves as a bridge between materials science and healthcare, placing a strong emphasis on sustainability and medical innovation. Collaboration extends across academia, medical

institutions, and industry. Beyond BTE, our investigations extend to exploring the application of hydroxyapatite nanoparticles (HAp NPs) in cancer therapy and diagnosis. Our overarching vision is to shape a greener, healthier future by uniting science and humanity.

#### 2:20 PM

## (ICC-SYM11-023-2024) Solution-based feedstock preparation for fused filament fabrication

O. Yucel\*1; J. Binner<sup>2</sup>

- 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, metals and, more recently, ceramics. Interest in this approach is rapidly increasing due to the potential for reduced costs and providing design freedom without expensive tools. Fused Filament Fabrication (FFF) is an up-and-coming ceramic AM method in which ceramic powder is mixed with a polymer binder system. This mixture is then used to produce filaments that are the feedstock for 3DP; a printer driven by specialist software uses the feedstock to produce pre-designed components. ZrSiO<sub>4</sub> is the ceramic powder used in this work to produce a ceramic matrix composite with polymers being dissolved in a solvent for mixing rather than via melting. There are several advantages of this approach, including low tool costs, low energy usage and simplicity of the method. The desired homogeneity has been confirmed and filament production was successful. Characterization of the mixtures and filaments will be presented.

#### 2:40 PM

#### (ICC-SYM11-024-2024) Binder Burnout Behavior of 3D Printed Ceramic Green Body

- B. Gao\*1; Y. Li1; S. Jang1; H. Son1; S. Park1; C. Bae1
- 1. Korea Institute of Materials Science, Department of 3D printing materials, Republic of Korea

Binder burnout (BBO) is a critical and extremely delicate process during ceramic manufacturing especially for the growing ceramic additive manufacturing technology. However, an in-depth understanding of the thermal degradation behavior of BBO is still lacking. Here, we investigated the BBO process via combining theoretical modeling and experimental analysis. The evolution of key factors such as the binder distribution, and the mass transport were simulated, demonstrating the inhomogeneity of binder removal in a planar receding mode within a larger body under a faster heating rate. The decomposition kinetics were found to be changed during BBO process due to the alteration of degradation mechanism from initial rate limited reaction of generating volatile flammable products to later diffusion of small molecules. The fundamental study provides valuable preliminary exploration for programing of high-efficient and defect-free thermal debinding.

# Symposium 1: Aerospace Ceramics and Composites

#### Symposium 1: Aerospace Ceramics and Composites

Room: Montreal 1-3 Session Chair: Ryan Fahsbender, Sierra Space

#### 3:30 PM

## (ICC-SYM1-024-2024) Laser Ablative Patterning of $B_4C$ and MoAlB Ceramics for Hydrophobic Surfaces (Invited)

B. Cui\*<sup>1</sup>; S. Ruiz<sup>1</sup>; R. Wall<sup>1</sup>; Y. Yoo<sup>1</sup>; L. Wadle<sup>1</sup>; Y. Lu<sup>1</sup>; C. Wohl<sup>2</sup>; V. L. Wiesner<sup>2</sup>

- 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<sub>4</sub>C) 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 ( $\theta < 90^\circ$ ) to hydrophobic ( $\theta > 90^\circ$ ), 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<sub>4</sub>C and MoAlB surfaces.

#### 4:00 PM

### (ICC-SYM1-025-2024) Innovative Ceramic Design: Multilayered Architectures for Enhanced Multi-Hit Tolerance

- H. Yazdani Sarvestani\*1; D. Backman1; M. Genest1; B. Ashrafi1
- 1. National Research Council, Canada, Canada

Ceramics boast exceptional properties such as low density, high compressive strength, and impressive thermal stability. However, their brittleness restricts their applications, especially in scenarios demanding high toughness. Drawing inspiration from nature's biomechanical marvels, we present multilayered ceramic systems with bioinspired architectures that exhibit enhanced toughness and multi-hit tolerance. Our designs include helicoidal ceramics inspired by beetles, architectured ceramics inspired by nacre and conch shells, and stochastic ceramics inspired by the American white pelican feather and dragonfly wing. Manufactured through laserengraved/cut tiles and Surlyn, these ceramics underwent rigorous testing under quasi-static and impact loads. We utilized various techniques, including digital image correlation, computed radiography, micro-CT scanning, and 3D laser scanning microscopy for damage assessment. Finite element analysis using ANSYS LS-DYNA or ABAQUS modeled the responses, revealing the impact of architectural parameters on energy absorption and multi-hit capabilities in architectured ceramics.

#### 4:20 PM

#### (ICC-SYM1-027-2024) Alumina-Based Ceramic Coatings Obtained by the SHS Process for High Temperature Corrosion Protection of Steel Tubulars (Invited)

E. Medvedovski\*1; G. Mendoza2

- 1. Consultant, Canada
- 2. Endurance Technologies Inc., Canada

For steel tubulars' protection against high temperature corrosion of gases, often containing abrasive solid particles, and to enhance their integrity, ceramic coatings can be employed. Combining self-propagating high-temperature synthesis (SHS) with high-speed centrifugal process, alumina-based coatings were produced onto the inner surface of tubulars. According to the developed SHS batch formulation and the designed centrifugal device and process, wellconsolidated composite ceramic layers with a thickness of 1.5-3 mm have been obtained. The developed SHS process does not create hazardous gases occurrence, and it takes ~30 sec for the ceramic phase formation. A high-level compaction due to centrifugal forces and a liquid phase sintering approach provided dense gradient ceramic structure formed by oxide grains cemented by a glassy phase and bonded with a steel substrate through a thin iron layer. The coatings onto the inner surface of carbon and stainless steels' tubes of up to 12 ft.-lengths and standard dimensions from 2-3/8″ to 7.5″ OD were successfully produced. They can be employed in high temperature (over 1000°C) corrosive environments and erosive flows due to high contents of Al2O3 and some other oxides and consolidated ceramic structure.

### Symposium 4: Green ceramics for clean energy and sustainability

#### Symposium 4: Energy Ceramics and Green Processes II Room: Montreal 6

Session Chair: Claire Dancer, University of Warwick

#### 3:30 PM

### (ICC-SYM4-023-2024) A Review on Recent Trends on Green Ceramics and Sustainability

O. Taiwo\*1; A. Adediran2

1. Nnamdi Azikwe University, Metallurgical and Materials Engineering, Nigeria

2. Landmark University, Nigeria

The demand for clean energy technologies continues to grow as societies worldwide seek to mitigate environmental impact and promote sustainable development. In this context, ceramic materials play a pivotal role due to their unique properties, such as high thermal stability, chemical inertness, and electrical insulation. This paper reviews the recent trends of using green ceramics in advancing clean energy solutions and promoting sustainability. Green ceramics, characterized by environmentally friendly production processes and recyclable components, offer significant promise in various clean energy applications. From photovoltaics to fuel cells and energy storage devices, ceramics contribute to improving energy efficiency, reducing greenhouse gas emissions, and enhancing overall system performance. Key areas of focus include the development of novel ceramic materials with enhanced electrochemical properties for solid oxide fuel cells, sustainable fabrication techniques for ceramic-based photovoltaic devices, and advancements in ceramic membranes for clean water production and purification. Furthermore, the paper discusses challenges and opportunities in scaling up green ceramic production, addressing issues such as material purity, cost-effectiveness, and manufacturing scalability. Finally, this paper underscores the importance of materials science in driving progress towards a cleaner, more sustainable world.

#### 3:50 PM

# $(ICC-SYM4-024-2024)\ Recycling\ secondary\ raw\ materials\ in\ the\ ceramic\ tile\ industry:\ how\ to\ overcome\ technological\ hindrances$

S. Conte<sup>\*1</sup>; C. Molinari<sup>1</sup>; m. dondi<sup>1</sup>; C. Zanelli<sup>1</sup>

#### 1. CNR ISSMC, Italy

After the decline in the industrial tiles production recorded during the pandemic, the European industry has returned to growth with a consequent general increase in the demand for raw materials, which represents an open challenge for turning ceramic manufacturing fully sustainable in the long run. The transition towards a Circular Economy requires to enhance the resource efficiency also by reducing the consumption of strategic raw materials by waste recycling. Although the tile-making industry proved to be able to recycle its own processing residues into cannibalistic loops, the use of waste from further sources is at present quite limited. Waste glass, at variance of others waste, is quite studied, especially the silica-limesoda one (SLS). It has been mainly employed as flux in substitution of feldspars in porcelain stoneware batches, pointing out technological problems of fired tiles, mostly related to a mismatch between maximum densification and water absorption ≤0.5%, as well as a lower bulk density with respect to the waste-free bodies. This research aims to overcome this technological hindrances acting on the starting formulation of the batches: next to the well-known substitution glass in place of feldspars, a chemical balance of the batch has been adopted. Good technological results were obtained for porcelain stoneware bodies containing 5 and 10 wt% of SLS glass.

#### 4:10 PM

## (ICC-SYM4-025-2024) Oxide-carbide duplex ceramics with recycled tungsten carbide for greener cutting tools

P. Klimczyk\*<sup>1</sup>; M. Podsiadlo<sup>1</sup>; K. Chat-Wilk<sup>1</sup>; J. Laszkiewicz-Lukasik<sup>1</sup>;

- K. Momot<sup>1</sup>; Y. Rumiantseva<sup>1</sup>; A. Gubernat<sup>2</sup>
- 1. Lukasiewicz Research Network Krakow Institute of Technology, Poland
- 2. AGH University of Krakow, Faculty of Materials Science and Ceramics, Poland

Sintered carbides (WC-Co) have found extensive applications in various industries, particularly for cutting tools and wear-resistant parts. However, the classification of both tungsten and cobalt as critical raw materials (CRM) raises concerns about their long-term availability and environmental impact. Our research focused on the development of ceramic matrix composites (CMCs) in which oxide (Al<sub>2</sub>O<sub>3</sub>, ZrO<sub>2</sub>) and carbide (WC) phases form an interpenetrating "duplex microstructure". Such CMCs may be a promising tool material utilizing WC recovered from WC-Co scrap. The elimination of Co, the partial replacement of WC with oxide phases and the use of recycled WC can reduce the need for CRM and have a positive impact on the environment. The two variants of CMC, both containing oxides and 42 vol.% of WC were obtained by Spark Plasma Sintering. The "fresh" commercial WC powder was used for the first variant and the recycled one for the second variant. Composites had relative density of 98-100% and a Young's modulus of 400-470 GPa, depending of WC used and sintering parameters. SEM observations showed good homogeneity of both composites, but the first variant had a microstructure more similar to an ideal duplex. Best material had the hardness of 21.5 GPa, fracture toughness of 5.8 MPa\*m<sup>1/2</sup> and cutting performance (during turning of Inconel 718 alloy) comparable to leading commercial ceramics.

#### 4:30 PM

### (ICC-SYM4-026-2024) Residues from beneficiation of granite in porcelain stoneware: effects on technological properties

C. Molinari\*1; A. Sima3; M. Cavina2; G. Guarini1; S. Conte1; S. Albonetti2;

- E. Sanchez3; m. dondi1; C. Zanelli1
- 1. CNR ISSMC, Italy
- 2. Alma Mater Studiorum Bologna University, Department of Industrial Chemistry "Toso Montanari", Italy
- 3. Instituto Universitario de Tecnología Cerámica, Universidad Jaume I, Spain

Granite extraction waste represents an interesting secondary material for ceramic production. Its influence on the porcelain stoneware formulations is not well defined. For this reason, residues from granite extraction for flux production were studied. Two different waste were selected: an iron-rich material, from magnetic separation, and one derived from abatement systems with bag filters. Batches were formulated by partial substitution of feldspar, from 3 to 9 wt%. The technological behaviour was assessed by simulating the industrial tile manufacture on a laboratory scale. Tiles of 10 cm  $\times$  5 cm  $\times$ 0.5 cm were shaped by uniaxial pressure. The industrial process was simulated by fast firing in electric roller kiln at maximum temperature in the 1180-1220 C range (60 min cold-to-cold). The effect of waste addition was evaluated during the whole production process. Fired samples were characterized in terms of technological properties, colour, mineralogical composition, and microstructure evolution. A progressive reduction of the maximum sintering temperature with waste is observed. The formulation optimization led to reduce firing temperature, respecting the technological constraints. A further increase of finer waste content worsened compaction during the shaping process and mechanical strength. Furthermore, the presence of micaceous particles may act as cracks initiation.

#### 4:50 PM

## (ICC-SYM4-027-2024) Valorization of granite plant sludge and recycled glass fines into slip casted porcelain-like products

- F. Delobel\*1; A. Gelinas2
- 1. Centre de transfert technologique en écologie industrielle, Canada
- 2. Studio Mineral, Canada

Several studies have shown the potential of including residual inorganic waste in the production of dry-pressed ceramics. However, this research explores waste incorporation specifically via slip casting production. The granite fines characterizations were followed by a triaxial study to determine the maximum addition of granite fines and recycled glass dust to obtain a porcelain-like material. This study produced 18 samples, each fired at 2,232 degrees Fahrenheit in an oxidized atmosphere. Workability, plasticity and deformations were evaluated on each sample. This case study suggests the possibility of including waste in a large-scale production context in low-tech productions such as slip casting. On a larger scale, this also opens the door to establish processes to effectively assess and adapt to the variations in the composition of residual inorganic materials.

# Symposium 11: Recent advances and the future of additive manufacturing

# Symposium 11: Material Extrusion, Binder Jetting, and Novel Methods

Room: Montreal 7

Session Chairs: Xuan Song, University of Iowa; Rodney Trice, Purdue University

#### 3:30 PM

### (ICC-SYM11-025-2024) Gradient Ceramic Structures Produced via Multi-Material Additive Manufacturing (Invited)

N. Ku\*<sup>1</sup>; M. Guziewski<sup>1</sup>; F. Kellogg<sup>2</sup>; M. Golt<sup>1</sup>; C. Mock<sup>1</sup>; S. G. Hirsch<sup>1</sup>; P. Goins<sup>1</sup>; L. Vargas-Gonzalez<sup>1</sup>

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

Multi-material additive manufacturing is an enabling process for creating heterogeneous dense ceramics with mesoscale tailoring and compositional control. Mesoscale structures, such as functional gradients, have been shown to exhibit unique extrinsic behaviors in the bulk that are not inherent to the intrinsic behaviors of the constituent materials. This presentation will discuss the ongoing investigation into utilizing additive manufacturing techniques to enable mesoscale tailoring in structural ceramics. Non-oxide ceramics, such as silicon carbide, boron carbide, and ultrahigh temperature ceramics (i.e. example here) will be discussed. Utilization of direct-ink-write to co-deposit multiple highly-loaded particulate suspensions will be presented, as well as post-processing and sintering challenges of the printed heterogeneous parts and challenges associated with mitigation of residual stresses from thermal expansion mismatch. Furthermore, reactive laser sintering methods to enable co-deposition of dissimilar material systems (ceramics and metals) will be shown.

#### 4:00 PM

#### (ICC-SYM11-026-2024) Shape stability of additively manufactured carbon fiber reinforced PEEK-based ceramic matrix composites (CMCs) through FFF technology

W. Freudenberg\*1; J. Best1; F. Wich1; N. Langhof1; S. Schafföner1

1. University of Bayreuth, Ceramic Materials Engineering, Germany

The use of additive manufacturing in the production of advanced materials, particularly carbon fibre reinforced ceramic matrix composites (CFRCMCs), represents a significant advance. To enable the additive manufacturing of CFRCMCs via fused filament fabrication (FFF) techniques using the liquid silicon infiltration (LSI) process, it is necessary to incorporate an additional step to ensure shape stability during pyrolysis at 1000°C in a nitrogen atmosphere. This stabilisation step involves thermally induced crosslinking of the additive manufactured samples below the melting temperature of 343°C. Differential scanning calorimetry (DSC) and rheological tests were carried out to investigate the effect of annealing time and temperature. These analyses provide valuable insight into the thermal and flow properties of the material throughout the stabilisation process. In addition, a visual inspection was carried out to confirm the accuracy of the stabilisation process, ensuring that the manufactured CFRCMCs retain their intended shape and structural integrity. This research highlights the innovation of additive manufacturing techniques and the importance of careful control and understanding of post-processing steps, such as stabilisation, to achieve the desired material properties for advanced applications.

#### 4:20 PM

#### (ICC-SYM11-027-2024) Hydrothermal-assisted Jet Fusion: A Selective Cold Sintering Approach (Invited)

X. Song $^{*1}$ 

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

Ceramic additive manufacturing (AM) typically uses a high fraction of organic binders to form pre-sintered green parts that require a post de-binding process to remove. The de-binding process inevitably results in severe gas expansion and residual chars, leading to structural defects, accumulated stress, and compromised material properties in the final parts. Here we report a binder-free additive manufacturing process that utilizes a hydrothermal method to create geometrically and compositionally complex ceramics under mild temperatures. The hydrothermal method utilizes a selectively deposited volatile dissolving ink, high pressure, and milt heat to strategically fuse a ceramic powder bed into complex geometries. Compared to traditional AM methods for ceramics, the HJF process eliminates the need for organic binders in green part fabrication and offers the potential to directly co-print ceramics with other dissimilar materials, such as polymers and metals, enabling the development of novel multi-functional ceramic composites.

#### 4:50 PM

#### (ICC-SYM11-028-2024) The Influence of Zirconia Additives on Processing and Properties in Coupled Laser Powder Bed Fusion and Reaction Bonding Methods

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- 1. California Institute of Technology, USA
- 2. Jet Propulsion Laboratory, USA

In striving to expand additive manufacturing (AM) to high-temperature materials, laser powder bed fusion (LPBF) has been combined with reaction bonding (RB), bringing net-shape forming benefits to ceramic structures. Historically, yttria-stabilized zirconia (YSZ) has been a common additive in RB of alumina-based ceramics, but its role in this synergized process is less certain. This study investigates YSZ's influence in the combined printing and reaction-bonding technique by scrutinizing its impact on densification and thermal and mechanical properties of alumina-based materials for high-temperature applications. By incorporating YSZ into aluminum-alumina mixtures, YSZ's

effect on powder flowability, oxidation, and densification is explored. Detailed flow experiments investigate YSZ's role in enhancing powder bed printing, while thermal analysis aims to determine YSZ's contribution to the oxidation character of mixtures. Morphological analysis through scanning electron microscopy and phase assessment via X-ray diffraction elucidate the microstructural enhancements attributed to YSZ, including grain refinement and thermal shock mitigation under printing and sintering conditions. By probing YSZ's role in processing and performance of ceramics via the AM+RB method, we aim to provide insights into the optimization of material properties.

#### 5:10 PM

#### (ICC-SYM11-029-2024) Layer-by-Layer Ceramization of Chemically Bonded Oxide Ceramics Using Flash Lamp Annealing: A New Approach to Additive Manufacturing

- E. Özmen<sup>\*1</sup>; M. D. Losego<sup>1</sup>
- 1. Georgia Institute of Technology, School of Materials Science and Engineering, USA

The use of flash lamp annealing (that can deliver up to 4.3 kW/cm<sup>2</sup> in a single pulse) to bind and/or sinter oxide ceramic layers (Al<sub>2</sub>O<sub>3</sub> and ZnO) containing an alumina phosphate binder was studied. We also investigated the light-matter interaction during photonic curing, and the improvement of the light absorbance using enhancer additions with graphite, black iron oxide, and organic dyes. Aqueous slurries of Al<sub>2</sub>O<sub>3</sub> and ZnO powders blended with aluminum dihydrogen phosphate (ADP,  $Al(H_2PO_4)_3$ ) were spray cast onto glass slides, and then heat treated with FLA. As sprayed layers are about 100 µm thick but consolidate to as thin as 45 µm upon heat treatment. Using x-ray diffraction, we can track the transformation of ADP to the aluminum metaphosphate (AlPO<sub>4</sub>) phase. Minimum FLA energy densities of 450-500 J/cm<sup>2</sup>(less than 1 minute to deliver) are sufficient to drive the ADP to AlPO<sub>4</sub> transformation. Additionally, coatings did not show any degradation behavior in room atmosphere. For samples with multiple layers, the interaction between consecutive layers, and the effect of light absorbance on the microstructure were investigated using SEM. This study demonstrates that FLA can be offered as an alternative to laser-based methods for producing stable oxide ceramic coatings with a potential for scaling to other additive manufacturing approaches.

### **Poster Session**

Room: St. Laurent 1 and 2

5:30 PM

(ICC-P001-2024) Impact of Thickness, Refractive Index and Minority Carriers Diffusion Length on AR Materials: Study on the Spectral Response of Monocrystalline Si Cell

A. Diaw $^{*1}$ 

1. Cheikh Anta Diop University, Physics, Senegal

In this work, the following materials were chosen as anti-reflective layer namely magnesium fluoride (MgF2); silicon oxynitrides (SiOxNy), silicon oxides (SiOx), silicon nitride (Si3N4) and hydrogenated silicon nitride (SiNx: H). The calculations were made on the basis of layer thickness values and refractive indexes which allow the phase and amplitude conditions to be respected. Numerical simulations have shown that weak reflectivities on the surface of the plane cell coated with a single layer can be obtained. For example, for simple coatings of materials based on Si3N4 and SiOx, a value of the reflectivity is obtained rotating around 3 and 2% respectively. Structures with multilayer type coatings such as SiOx/SiNx:H/Si, give a very low reflectivity around 1%. Thus, the refraction index of the coating is an important parameter which plays a major role in the optical properties of the materials. The closer the refractive index is to the index of the substrate or the layer above the substrate, the greater reflectivity.

# (ICC-P002-2024) Investigations of binder burnout behavior of KNN-based tapes for the fabrication of piezoelectric multilayer actuators with Ni electrodes

B. Capraro\*1; D. Eckardt2; J. Topfer2; M. W. Alkanj2

- 1. Fraunhofer IKTS, Germany
- 2. Ernst-Abbe-Hochschule Jena, Germany

Piezoceramic multilayer actuators are not only important for the precise metering of diesel injection in vehicles but are also used for high-precision actuators or sensors. The main cost driver for production of such multilayer actuators are the precious metallization pastes used for printing electrodes. To reduce fabrication costs, base metals are used as electrodes. Cofiring and reoxidation processes of lead-free, KNN-based multilayer systems with Ni electrodes have to be performed at low p<sub>02</sub> conditions to avoid oxidation of electrodes. The preceding binder burnout of green multilayer actuators with printed Ni electrodes is a critical step. If debindering is performed in air, oxidation of Ni particles may occur. The binder burnout out in reducing conditions may lead to incomplete decomposition and residual carbon. We present results of a study of the binder burnout behavior of KNN-based tapes with new binders with allow burnout in reducing atmospheres. After selecting possible binders we studied the thermal decomposition behavior of these in different oxygen partial pressures. The rheology of the slurries based on the new binder systems was studied and piezoelectric green tapes were fabricated. The debindering behavior of the tapes was examined and is discussed in comparison with that of the pure binders.

## (ICC-P004-2024) All-solid-state lithium batteries (ASSLBs) with Ta-doped lithium lanthanum zirconium oxide electrolyte

D. K. Panda\*1; S. Creager2; R. Bordia1

- 1. Clemson University, Materials Science and Engineering, USA
- 2. Clemson University, Department of Chemistry, USA

First, a thin fully doped dense LLZTO layer of ~20µm thickness is prepared by tape casting and subsequent sintering. To reduce Li-loss during sintering, a suitable sintering aid has been used. Secondly, a directional freeze casting, followed by sublimation of the ice technique was used to make the engineered porosity LLZTO electrodes with hierarchical porosity. The microstructural goal is to have large, aligned pores with the walls of the aligned pores being themselves porous with small pores. This design will maximize the interface between electrode and electrolyte after the scaffolds have been infiltrated with the electrodes and integrated with the electrolyte. Research is continuing further optimization of the process, especially to make thin engineered porosity scaffolds. LLZTO films and pellets have been characterized using microscopy, spectroscopic and electrochemical techniques. To study the effect of thickness on energy density, an analytical model was developed to analyze the effect of thickness on anode, cathode, and current collector. Acknowledgment: This work was supported by Clemson University's Virtual Prototyping of Autonomy Enabled Ground Systems (VIPR-GS), under Cooperative Agreement W56HZV-21-2-0001 with the US Army DEVCOM Ground Vehicle Systems Center (GVSC). DISTRIBUTION STATEMENT A. Approved for public release; distribution is unlimited. OPSEC8246.

#### (ICC-P005-2024) Simple method for La<sub>2</sub>Zr<sub>2</sub>O<sub>7</sub> synthesis

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- 1. University of Chemistry and Technology, Prague, Department of Glass and Ceramics, Czechia
- 2. Czech Academy of Sciences, Institute of Photonics and Electronics, Czechia

The pyrochlores  $(A_2B_2O_7)$  can be prepared by methods of solid state synthesis, sol-gel, combustion or molten salt synthesis. The solid state method is simple but requires relatively high temperatures (~1600 °C) and subsequent attrition milling to produce fine powders. The sol-gel and combustion method are suitable for

synthesis of pure fine powders with precise stoichiometry at relatively low calcination temperature (~900 °C) but consists of multiple steps and are costly. The molten salt synthesis allows preparation of fine powder but it is demanding when it comes to the amount of salt (the amount of salt exceeds the amount of reactants by several times) which limits the yields. In this contribution a simple method for La<sub>2</sub>Zr<sub>2</sub>O<sub>7</sub> synthesis from La<sub>2</sub>O<sub>3</sub> and ZrO<sub>2</sub> by combination of solid state and molten salt synthesis is presented. The formation of La<sub>2</sub>Zr<sub>2</sub>O<sub>7</sub> starts ~850 °C with the help of LiF which is added in very low amount (5 wt.%). Phase purity as well as morphology of the powder was investigated. 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).

### (ICC-P006-2024) Characterization of Microwave-Synthesized Bioactive Glass 58S: Structural and Bioactivity Insights

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- 2. Universitat Jaume I, Instituto Universitario de Tecnología Cerámica, Spain

The synthesis of bioactive glasses by a hydrothermal chemical route using microwaves is a novel and efficient approach. This approach offers several advantages over traditional methods, including rapid and uniform heating, energy efficiency, high yield, short preparation time, low processing cost, narrow particle size distribution, and high purity. The bioactive glass 58S (58% SiO<sub>2</sub>, 33% CaO, and 9% P<sub>2</sub>O<sub>5</sub> in wt%) was selected as the working composition. Aqueous-based solutions were prepared using various alkoxides and salts. The sols were then subjected to microwave heating at different temperatures and pressures using a microwave digestion system. The resulting material was ground into powder and stabilized by thermal treatment in a lab-made microwave sintering furnace. All glasses were immersed in simulated body fluid (SBF) for up to 14 days following a standardized protocol. Scanning electron microscopy, X-ray diffraction, energy-dispersive X-ray spectroscopy, and Fourier-transform infrared spectroscopy were used to characterize the glasses before and after immersion. The authors thank the Spanish Ministry of Universities the support provided by the Margarita Salas postdoctoral contract MGS/2022/20 (European Union - NextGenerationEU); by the grant PID2021-128548OB-C21 and C22 (MCIN/AEI/10.13039/501100011033), and by the grant CIGRIS/2022/077 (Generalitat Valenciana).

### (ICC-P007-2024) Characterization of High Purity Alumina for Advanced Ceramic Applications

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- 2. Ecole Polytechnque de Montreal, Genie Chimique, Canada

With the recent progress in the semiconductors, there is demand for ceramic parts that has premium dielectric properties, which implies to have very low level of impurities. Despite the growing demand for High Purity Alumina (HPA), its sintering properties is not well investigated. It is expected to observe a different sintering behaviour for HPA compared to typical 99.8% alumina. In the ceramics, prepared from standard alumina, the impurities participate in the structure of ceramics and it contribute to the sinterability of the material. In this work, HPA with the purity of 99.999% is used. The alumina has been obtained through a a mineral acid process, thus producing alumina with low sodium and silicon impurities, which are major contaminants in the commercially available alumina from Bayer process. The alumina powder has alpha crystallinity, primary particles size of D50 = 0.3 microns and BET surface area of 4.5 m<sup>2</sup>/gr. Sintering curve and dilatometry behaviour of the HPA are studied. For sintering curve, the alumina powder

is compacted using uniaxial press up to 100 MPa. The obtained green density is about 2.2 gr/cm<sup>3</sup>, which is considered acceptable. Thereafter, the green bodies are fired up to four hours at different temperatures 1300 – 1600 °C. The fired density is measured and it is compared with the skeleton density of alumina, which is 3.96 gr/cm<sup>3</sup>.

### (ICC-P008-2024) Effects of $Ho^{3+}$ codoping on the properties of the $(Pb_{0.91}La_{0.09})(Zr_{0.65}Ti_{0.35})0_{.9775}O_3$ ceramic materials

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Lead lanthanum zirconate titanate (PLZT) is one of the luminescent ferroelectric ceramics that are very interesting materials for many electro-optical devices. Although the chemistry of such materials has been investigated for years, the impact of lanthanide dopants on PLZT remains largely unexplored. Consequently, there is a growing interest in lanthanide-codoped PLZT ceramics due to their promising properties and potential applications in electronics and optics. In this study, we synthesized ceramic materials of  $(Pb_{\scriptscriptstyle 0.91}La_{\scriptscriptstyle 0.09})(Zr_{\scriptscriptstyle 0.65}Ti_{\scriptscriptstyle 0.35})_{\scriptscriptstyle 0.9775}O_3$  composition, incorporating different concentrations of holmium ions (ranging from 0 to 1 wt.%). We employed a gel-combustion route to synthesize ceramic nanopowders using high-purity raw materials (> 99.9%), then sintering using the hot uniaxial pressing method. The resulting ceramics underwent analysis using techniques such as X-ray powder diffraction (XRD), scanning electron microscopy (SEM) with energy-dispersive X-ray spectroscopy (EDS), and examination of dielectric, ferroelectric, and optical properties. The obtained results give us a detailed account of the influences of the holmium ions on the structural, microstructural, dielectric, ferroelectric, and luminescent properties.

# (ICC-P009-2024) Verification of silicon nitride materials properties supported by image analysis by the means of two-point correlation method

- Z. Pedzich\*<sup>1</sup>; M. Grabowy<sup>2</sup>; A. Kluczowska<sup>2</sup>; G. Grabowski<sup>1</sup>; A. Wojteczko<sup>1</sup>; D. Kozien<sup>3</sup>
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- 2. Intitute of Power Engineering, Ceramic Division Cerel, Poland
- 3. AGH University of Science and Technology, Poland

Work concerns of the use of the two-point correlation method, a numerical image analysis method that can be used if microstructure images do not allow precise detection of grain boundaries. Experiments were performed on different silicon nitride materials dedicated used for fabrication of elements used in form for casting of complex parts for aircraft industry. Six types of sintered silicon nitride materials were used for testing. They differed in process conditions and chemical composition of sintering additives. The determined microstructural parameters were compared with physical, mechanical and thermal properties. It was concluded that the use of image analysis utylized two-point correlation allows for relatively easy, fast and precise characterization of the microstructure of Si<sub>3</sub>N<sub>4</sub> sinters. Some parameters determined during the analysis gave a quantitative view of the homogeneity of the microstructure. On their base, materials with very similar microstructures could be effectively classified in terms of mechanical properties. Acknowledgments: The research was financed by the Polish National Centre for Research and Development (NCBR) within a framework of the project POIR.01.01.01-00-0102/21-00 : Innovative silicon nitride based materials characterized by extremely high resistance to thermal shock, mechanical strength and corrosion resistance.

# (ICC-P010-2024) Silica nanoparticles: from rice husk and rice straw by fungal mediated biotransformation using Pleurotus florida

- D. Kaur<sup>\*1</sup>; M. Reddy<sup>2</sup>; O. Pandey<sup>2</sup>
- 1. University of Trento, Industrial Engineering, Italy
- 2. Thapar Institute of Engineering and Technology, School of Physics and Materials Science, India

For a balanced ecosystem, agriculture waste needs proper management. The agriculture waste can be processed to produce bioenergy, conserving nonrenewable resources and producing nanoparticles. This research reports the utilization of rice husk and straw in an innovative bio-transformative route to isolate silica nanoparticles (SiNPs). The rice husk and straw were subjected to mushroom cultivation, followed by the extraction of SiNPs from the spent substrates. These SiNPs were characterized by using X-ray diffaction (XRD), field emission scanning electron microscopy (FESEM), Fourier transforms infrared spectroscopy (FTIR), and energy dispersive X-ray spectroscopy (EDS). FESEM results showed that the particle size of SiNPs lies between 15 and 30 nm and 25 and 30 nm for rice husk and straw, respectively. The mushroom yield of Pleurotus forida was 26.9% and 53.1% for rice husk and rice straw, respectively. The total yield of SiNPs from spent husk and straw was 67.5% and 26.2%, respectively. The nanoparticles derived from rice husk and straw inhibited the growth of Staphylococcus aureus (59.9±1.3% and 65.3±2.4%, respectively) and Escherichia coli (68.5±2.8% and 70.1±2.5%) at a 100 mg/ml SiO2 concentration. Present study results suggest that raw husk and straw and spent husk and straw could serve as a potential source for the production of SiNPs.

### (ICC-P011-2024) Enhancing Epoxy Resin Biocomposite using bamboo fiber and Mangifera indica particulate

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- 1. Nnamdi Azikiwe University, Awka, Metallurgical and Materials Engineering, Nigeria

The demand for an eco-friendly environment has led to the development of green plant fibre/polymer matrix composites. The water absorption resistance and impact strength performance of bamboo fibre/mangifera indica particulate/epoxy biocomposites were examined. The surface morphology of the developed composites was analyzed using field emission scanning electron microscopy (SEM). Taguchi's statistical method was used to analyze the results of the tensile strength test. The epoxy resin was incorporated with bamboo fibre/mangifera indica particulate in different proportions using the hand lay-up technique. The hybrids of bamboo fibre/Mangifera indica particulate were added in equal ratios. The absorption curve shows that the water absorption rate of specimens with high reinforcement materials shows a higher rate of water absorption after extended immersion. The diffusion coefficient and maximum water content values increase as the reinforcement content increases. The impact results show that composite samples with higher reinforcement materials ranging from 15% to 20% recorded the maximum impact strength over other samples. This indicates excellent interfacial bonding of molecules of epoxy, Mangifera indica particulates, and bamboo fibre.

# (ICC-P012-2024) Effects of temperature, aging time, and method of introducing stabilizer oxide into solid solution on properties of Mg-PSZ materials

- D. Kozien\*1; M. Grabowy<sup>2</sup>; A. Kluczowska<sup>2</sup>; K. Sieron<sup>2</sup>; A. Wojteczko<sup>1</sup>; Z. Pedzich<sup>3</sup>
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Part of the system in the technology of casting of precise parts of aircraft engines is a ceramic sealing element, which is also a fragment of the liquid metal supply system, taking on itself the impact of the metal stream, as well as it calms and separates the stream of the flowing melt. Partially stabilized zirconia (PSZ) was used due to its properties which meet demanded requirements. The properties of such material strongly depend on the manufacturing conditions. The proper selection of stabilizing oxide precursor and aging conditions system allowed us to design the phase composition and microstructure and made possible to obtain a material with good mechanical properties and resistance to thermal shock under rapid heating conditions (low CTE). Significant improvement was achieved when comparing CTEs of the standard and a new material - 5.35 vs 4.5 x10<sup>-6</sup> 1/°C. It has a key impact on thermal shock resistance under sudden heating. It was an effect of combination of the phase composition and microstructure which are a consequence of the specific chemical heterogeneity of the system. Acknowledgments: This work was financed by the Polish National Centre for Research and Development (NCBR) within a framework of the project POIR.01.01.01-00-1094/19 : Advanced structural ceramic materials used in investment casting of aircraft engine elements.

### (ICC-P013-2024) Effect of composition of CaO-B $_2O_3$ -SiO $_2$ system glass fiber on wound healing

- S. Yamaguchi\*1; T. Takeuchi1; M. Ito1
- 1. Chubu University, Department of Biomedical Sciences, Japan

The management of injuries, including cuts, burns, and ulcers, is crucial, especially for individuals with chronic wounds. Jung and Day introduced a novel wound dressing made of borate glass, known as 13-93B3, containing K<sub>2</sub>O, CaO, and B<sub>2</sub>O<sub>3</sub>, which was FDA-approved in 2016 for treating acute and chronic wounds. Recent discussions have emphasized the potential of bioactive glasses in soft tissue repair. However, there's still a need for systematic research to understand the mechanisms behind accelerating wound healing processes. In this study, glass fibers with different compositions in the CaO-B2O3-SiO2 system were examined both in vitro and in vivo to understand their specific roles in wound healing. Effect of exudate, ion release, and formation of silica gel and apatite on wound healing was investigated step by step. As a result, it was observed that glass fibers containing moderate  ${\rm \tilde{SiO}}_2$  levels effectively controlled exudate and facilitated wound healing by releasing ions like Si, Ca, and B. The formation of silica gel and apatite was believed to maintain a moist environment, aiding early-stage healing, though they might hinder wound surface closure at later-stage healing.

### (ICC-P014-2024) Thermal Shock Resistance of EBC/EBC+crack healing agent Layered Material

#### K. Lee\*1; J. Shin1; M. Suh1

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The engine parts applied with environmental barrier coatings (EBCs) on SiC/SiC composites are being studied as an alternative to alloy to improve high-temperature resistance of aircraft engine. The EBCs not only provides heat resistance but also solves the issues of mass loss at hot water vapor condition. On the other hand, there is not much studied on the thermal shock resistance of EBCs that takes into account the repeated exposure to thermal cycling. When

the EBC coated on the SiC/SiC is repeatedly exposed to high and room temperature, cracks and delamination can occur due to the mismatch in the properties between the two layers. In this study, research was conducted to introduce a layer containing crack healing agents underneath the EBC to heal the cracks that occur during thermal shock cycles. A study was conducted to control the thickness ratio of the two layers and the content of crack healing agents to minimize stress generation due to the difference in properties between the two layers. First, optimization studies were conducted through thermal shock tests on the EBC/EBC+crack healing agent bilayered materials. Finally, the EBC bilayer was fabricated using APS (Atmospheric Plasma Spray) method with optimized conditions.

### (ICC-P015-2024) Investigating Structural, Magnetic and Dielectric Properties of Sr<sub>2</sub>Co<sub>2</sub>Fe<sub>12</sub>O<sub>22</sub>/CoFe<sub>2</sub>O<sub>4</sub> Composites

C. K. Patel<sup>\*1</sup>

1. Nirma University, Gujarat, India, Institute of science, India

Y-type Sr<sub>2</sub>Co<sub>2</sub>Fe<sub>12</sub>O<sub>22</sub> and spinel CoFe<sub>2</sub>O<sub>4</sub> ferrites were prepared separately using chemical co-precipitation technique. The prepared precursors were preheated at 500 C followed by final heating at 1000 C for 5 hrs. The prepared powder then mixed in different weight ratio (Y:S- 7:3, 6:4, 5:5, 4:6 and 3:7) and heated at 1150 C for 5 hrs. in a muffle furnace and slowly cooled to room temperature in order to get composite. The prepared composite powders were characterized using different instrumental techniques like FTIR, XRD, VSM, and dielectric measurements. FTIR spectra of all composites show two bands between 600-430 cm<sup>-1</sup> of Fe-O stretching vibrations. XRD analysis of pure Sr<sub>2</sub>Co<sub>2</sub>Fe<sub>12</sub>O<sub>22</sub> ferrite shows the presence of M and Y-type hexagonal phases, whereas composites samples consist of both phases of spinel and hexagonal ferrites. Coercivity of all composites was found < 800 Oe revealed soft magnetic nature of prepared composites. The squareness ratios of pure and composites were found to be less than 0.5, confirmed formation of multidomain structure. dielectric measurement was carried out at room temperature in a frequency range of 20Hz to 2MHz and the dielectric behavior is explained using Maxwell-Wegner type interfacial polarization.

### (ICC-P016-2024) Properties of FAp derived from DCPD and fluoride ions applied for fluoride-contaminated soil

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- 2. National Institute of Technology, Toyama College, Japan

The authors have previously found that dicalcium phosphate dihydrate (DCPD) can mineralize fluoride ions in fluorine-contaminated soil to form fluorine apatite (FAp), a stable mineral. Release of fluoride in the soil after the treatment was suppressed for the long term. However, detailed information on FAp from DCPD is not clear. In this study, we investigated various properties of the FAp derived from DCPD under various fluoride content in an aqueous solution. Reagent DCPD was made treatment with aqueous solutions containing various sodium fluoride under various solid/liquid ratios. The obtained solid phase was separated and characterized by SEM, surface area measurement, and XRD. The FAp particles were needle shape nano-particle and aggregated in the original DCPD particle shape. The particle shape of the FAp nano-particles was changed by fluoride content and solid/liquid ratio. Specifically, FAp particle size was decreased by smaller solid/liquid ratios, and surface area was increased. In this presentation, we summarized the experiment results and discussed future perspectives.

### (ICC-P017-2024) Correlation Between Ore Mineralogy and the Dissolution Behavior of K-feldspar

- A. Caamino\*1; S. Shakibania2; J. Rosenkranz2
- 1. MSU-Iligan Institute of Technology, Department of Materials and Resources Engineering & Technology, Philippines
- 2. Luleå Tekniska Universitet, Department of Civil, Environmental and Natural Resources Engineering, Sweden

The utilization of feldspar as an alternative source to produce potash (KCl) and alumina (Al<sub>2</sub>O<sub>3</sub>) has grown for agriculture and silica applications. However, its low dissolving efficiency has been a frequent problem encountered in extracting its metallic components. This study, as part of the POTASSIAL-EU Project has utilized intensive milling varying ball-to-ore ratio; milling speed; and grinding time to assess the correlation between the ore mineralogy and the dissolution behavior of K-feldspar using HCl leaching. Characterization of ground samples suggest that grinding conditions employed resulted to ultrafine particles which led to agglomeration. Acid leaching was performed to samples with intact mineral phases and samples where agglomeration at varying degrees persisted. Results showed that agglomeration was not a considerable factor that affected the dissolution, but the Specific Surface Area (SSA) together with structural disordering from the grinding process contributed to the increasing recovery of metallic components of the mineral. Sample with the finest particles recorded the highest metallic recoveries due its high SSA which allowed the particles to be more reactive to the leaching process, while coarse particles, even with distinct mineral liberation, have recorded a lower SSA and metallic recoveries, respectively.

### (ICC-P018-2024) Towards hybrid PIP and CVI processing of UHTCMCs at the Naval Research Laboratory

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- 2. US Naval Research Laboratory, Chemistry Division, USA

Ultra-high temperature ceramic matrix composites (UHTCMCs) are of interest for extreme aerothermal environments such as those experienced by hypersonic vehicles. There are several fabrication methods available to fabricate UHTCMCs. Of these methods, chemical vapor infiltration (CVI) stands out due to its ability to produce dense, near-net-shape structures. CVI involves the reaction of gaseous spices with a fibrous preform at elevated temperatures to decompose the gases and create a solid matrix. A disadvantage of the CVI process is the long infiltration times needed to produce high densities. Polymer impregnation and pyrolysis (PIP) is procedurally simpler than CVI and introduces a liquid preceramic polymer precursor into a fiber preform. The sample is then pyrolyzed to densify the UHTCMC. Typically several PIP cycles are needed to densify the UHTCMC and even then, densities achieved are lower than those achievable by CVI. To shorten processing times while maintaining high densities, hybrid processing of UHTCMCs can be done by coupling CVI with PIP. The Naval Research Laboratory has recently acquired a CVI system capable of producing large-scale, near-net-shape UHTCMCs. This poster will review the best design practices for CVI, PIP, and hybrid processing of UHTCMCs and some preliminary PIP results will be shown.

# (ICC-P020-2024) Unique oriented-nanocrystalline structure in $BaZrO_3$ prepared by acid-base chemical densification at near-room-temperature

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We developed a novel low-temperature fabrication process for complex oxides which is named as acid-base chemical densification (ABCD) process. This process can fabricate dense BaZrO<sub>3</sub> structure using the synthesis reaction between zirconia hydrous gel and barium hydroxide octahydrate. It was found that BaZrO<sub>3</sub> prepared at 100°C consisted of agglomerated grains. Particularly, it was revealed that these grains were consisted of aligned nano crystals by electron diffraction with transmission electron microscopy and the electron backscattered diffraction with scanning electron microscopy. In this study, crystallization behavior of BaZrO<sub>3</sub> in ABCD process is investigated. Particularly, effect of local structure around Zr ions in the zirconia hydrous gel used as precursor on crystallization to BaZrO<sub>3</sub> is revealed in detail.

### (ICC-P023-2024) Synthesis of Zr-, HfC fibers with Tunable Aspect Ratio

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To meet the demanding requirements on materials for extreme aerospace environments, new methods to produce ultra-high temperature ceramic (UHTC) fibers in high yield must be developed due to limited commercial availability. Group IV transition metal (Zr, Hf, etc.) carbides are of particular interest as fiber supports because of their robust thermal and mechanical properties. While chemical vapor deposition is a popular, well-developed synthesis method to produce these fibers, low yields and high expense limit its adoption for industrial application. Carbothermal reduction is a viable synthesis technique for producing high yields, however methods that successfully tune aspect ratio while achieving size- and shape-control need more development. For future use in additive manufacturing (AM) techniques as discrete reinforcements, control over both fiber aspect ratio and size dispersion is valuable. This work reports the progress on developing an aspect ratio-tunable synthesis for Zr-, and HfC fibers through carbothermal reduction, utilizing the AM method of material extrusion as an example use case.

#### (ICC-P024-2024) Physicochemical Characterization of Acid Treated Philippine Coal Fly Ash for Non-Fired Microporous Ceramics

- E. A. Limbaga<sup>1</sup>; M. Fuji<sup>2</sup>; V. T. Resabal<sup>1</sup>; I. B. Arugay<sup>1</sup>; L. M. Jabile<sup>1</sup>;
- L. I. Cabalo<sup>1</sup>; A. Caamino<sup>1</sup>; C. V. Cahimtong<sup>1</sup>; S. E. Manlupig<sup>1</sup>;
- R. V. Virtudazo\*1
- Mindanao State University-Iligan Institute of Technology, Department of Materials and Resources Engineering & Technology (DMRET), Philippines
- 2. Nagoya Institute of Technology, Japan

Fly ash from coal fired power stations is a potential raw material for the production of ceramic tiles, bricks and blocks. Previous study has demonstrated that by treating fly ash with acid solution it can change its microstructural morphology making it suitable for microporous ceramics. In this study, acid treated coal fly ash was investigated for non-firing ceramics bodies. The fly ash samples came from a coalfired power plant and was acid-treated. The fabrication process involves mixing thoroughly the fly ash and alkali activator. After mixing the materials, it was casted in a mold welded in a steel frame, cured, aged and tested for its physicochemical characteristics. Observed results demonstrated that the non-firing ceramic bodies of acid-treated fly ash (FADE) resulted to a changed in its physical, chemical, and morphological properties that implores a novel process for coal fly ash for microporous ceramic applications.

#### (ICC-P025-2024) Investigating Residual Stresses in Environmental Barrier Coatings for CMCs: A Multifaceted Approach Using Synchrotron X-Ray Techniques

S. Nauriyal\*1

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This investigation scrutinizes the nuanced residual stress dynamics within environmental barrier coatings (EBCs) for ceramic matrix composites in gas turbine engines. Employing synchrotron X-ray diffraction, radiography, and tomography, the experiment authenticates strain measurements in the bond coat, pivotal for prognosticating coating durability. Hole drilling coupled with radiography furnished a comprehensive perspective and substantiation of residual strains. Anticipated outcomes are poised to augment comprehension of EBC comportment, steering refined manufacturing processes and protracting component longevity. Future ramifications encompass innovative coating qualification methods and insights for high-temperature investigations.

### (ICC-P027-2024) Near-infrared lifetime of rare-earth doped nanoparticles and potential applications

M. Liu<sup>\*1</sup>; F. Vetrone<sup>1</sup>; J. Liang<sup>1</sup>

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Luminescent nanomaterials-which can be excited and emit in the near-infrared (NIR) range-have attracted great attention in a plethora of applications, including nanomedicine, biosensing, bioimaging, solar cells, photocatalysis and information storage. Especially in biomedical applications, NIR light mitigates the drawbacks associated with ultraviolet and/or visible light (e.g. photodamage, background autofluorescence, and limited penetration depth). At the forefront of NIR-excited nanomaterials are rare-earth doped nanoparticles (RENPs) that possess exceptional optical properties, such as multiple emission wavelengths and long luminescence lifetime. However, the NIR lifetime property of RENPs and their potential application are rarely explored. One of the limitations is the instruments since the current techniques, such as time-correlated single-photon counting, have limited measurement efficiency. Here, we present the synthesis of various NIR excited (and emitting) RENPs, the NIR lifetime is characterized subsequently by using the developed ultrahigh-speed line-scanning microscopy. The potential use of NIR lifetime in anticounterfeiting and nanothermometry will also be demonstrated.

#### (ICC-P028-2024) Monitoring phase evolution and temperature dependence of elastic properties and damping of porous anorthite-mullite ceramics by impulse excitation

P. Simonova\*1; E. Gregorova1; W. Pabst1

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Porous ceramics have low density and thermal conductivity, they are fire-resistant with good chemical stability and corrosion resistance even at high temperatures. Therefore, they are potential candidates for thermal protection applications in the aerospace area, for which a thorough knowledge of the mechanical behavior at high temperatures is indispensable. The impulse excitation technique (IET) is an ideal tool because it characterizes the high-temperature adiabatic elastic constants directly from the resonant frequency of vibrations, i.e. under conditions that are close to real conditions during application, while also assessing anelastic behavior (damping). In this contribution we present IET results for porous anorthite-mullite ceramics, in particular the temperature dependence of Young's modulus and damping of sintered ceramics, and also the evolution of elastic properties and damping during heating of unsintered ceramics, which is correlated with its phase evolution as determined by XRD. Samples were prepared from kaolin and calcite by uniaxial pressing and conventional sintering up to 1300 °C, resulting in bulk densities 1.70-1.77 g/cm<sup>3</sup> and open porosities 33-38 %. The phase composition was determined by XRD and compared with the main reactions as monitored by in-situ IET measurements of elastic properties (up to 1200 °C).

### (ICC-P029-2024) CO $_2$ Curable Akali-activated Geocement using Powder Bed Binder jetting

- T. Kim<sup>\*1</sup>; B. Jeong<sup>1</sup>; M. Lee<sup>1</sup>; H. Kim<sup>1</sup>; H. Lee<sup>2</sup>; B. Ye<sup>1</sup>
- 1. Korea Institute of Industrial Technology, Republic of Korea
- 2. Pusan National University, Republic of Korea

In this study, Al, Si, and Ca geocement-based green body was manufactured using powder bed binder jet 3d printing, and carbonation reaction was investigated. We focused on successfully 3D printed a CO2 curable geocement material system designed based on Ca, Al, and Si by jetting an alkali activator, and studied how to effectively immobilize CO2 into geocement parts through an accelerated carbonation reaction test. To investigate the carbonation of geocement, a pressurized carbonation reactor with a simple structure was designed, and the effects of carbonation were analyzed in relation to phase analysis, microstructural changes, pore size distribution, and the amount of carbon dioxide immobilized. Carbonation of geocement was confirmed to have the ability to immobilize up to 7.31% of 99.9%, 4 bar CO2, ambient temperature, RH over 60%, and 24-hour carbonation. Thermogravimetric analysis was performed to investigate the geopolymerization due to carbonation. It was confirmed that the characteristic peaks occurring in the geopolymerization are quickly terminated through carbonation. Additionally, the changes in pore distribution structure due to the evolution of carbonate, a reaction product, in the geocement matrix were investigated in detail using x-ray computed tomography (x-CT) and mercury intrusion porosimetry (MIP). This is expected to provide greater insight into the carbonation of geocement.

# (ICC-P030-2024) Template assisted synthesis of antibacterial SnO<sub>2</sub> nanofibers via electrospinning techniques for microreactor based photocatalytic dye degradation

- V. Katoch\*1
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In the present work, Tin oxide  $(SnO_2)$  nanofibers were synthesized using the electrospinning of polymer and metal oxide mixture followed by the calcination process. Polyvinyl pyrrolidone (PVP) with two distinct molecular weights (Mw = 1,300,000 and Mw = 40,000) served as a template, and the evaporation process of PVP was conducive to the creation of nanofibers composed of SnO<sub>2</sub> grains. An improved surface area of 43.75 mg was calculated via BET analysis and the optical bandgap of the hollow nanofibers was determined to be 3.78 eV. In contrast, the nanofibers exhibited a surface area of 19.50 mg and a bandgap of 4.45 eV. Due to the superior surface area, the SnO<sub>2</sub> hollow nanofibers demonstrated a prominent inline photocatalytic dye degradation efficiency of 82% evaluated for rhodamine B dye. The photocatalytic dye degradation efficiency of regular nanofibers showed a 65% reduction, which can be attributed to the decreased surface area of the SnO<sub>2</sub> nanofibers. Furthermore, the hollow nanofibers demonstrated superior antibacterial efficacy against Escherichia Coli bacteria when compared to the regular nanofibers.

### (ICC-P032-2024) The Potential of Rare-Earth, Metal Oxide Nanoparticles for Space applications

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Erbium and metal oxide nanoparticles have been synthesized as functional materials for integration into optical claddings, protective layers, and fiber coatings to be used in space. Erbium nanoparticles (Er NPs) were produced using pulsed laser ablation in liquid (PLAL), followed by advanced characterizations using X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS), scanning electron microscopy (SEM), Raman spectroscopy, and ultraviolet-visible-near infrared (UV-Vis-NIR) spectroscopy. The nature of the produced nanoparticles can be influenced by the fabrication conditions, such as the presence of solvents in the host liquid, the laser power and pulse frequency. In our study, we examined the effects of these parameters on the size, size-distribution, oxidation level, and stability of Er NPs to evaluate their impact. SEM investigations showed that the nanoparticles had a spherical shape with average diameters varying between 80 and 200 nm, depending on the laser line intensity. The oxidation level of Er NPs and their inner composition also resulted in various levels of stability and crystal orientations. The monitoring of the preparation conditions over the feasibility parameters of the PLAL technique showed that they can be set to deliver nanostructured systems with morphologies and properties compatible with the technological constraints met in space.

#### (ICC-P035-2024) Structural Development of Preceramic Polymers

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The pursuit of safe and reliable space travel requires the relentless research of next generation aircraft materials. In particular, materials that can withstand extreme high temperatures are in high demand. Polymer-derived ceramics (PDCs) are a compelling material capable of withstanding harsh operating conditions and hostile environments. PDCs are formed by the pyrolysis of an organosilicon polymeric precursor such as polysiloxanes or polysilazanes and exhibit excellent thermal resistance. Preceramic polymers form complex microstructures upon their thermal decomposition that modify the macroscopic thermal and mechanical properties. PDCs begin as amorphous covalent ceramics upon pyrolysis at 1000 °C but can crystallize when annealed at higher temperatures. The goal of this research is to study the transition from unbound preceramic polymer chains to crosslinked network to high temperature resistant PDC. The elemental composition and its subsequent changes during pyrolysis, the functional group progression, and the thermal transitions and mass loss events will all be studied. In summary, PDCs are an exciting class of high temperature resistant ceramics and understanding their structural development and subsequent performance is an essential step in exploring next generation aircraft materials.

#### (ICC-P036-2024) Firing Ceramics with Inpatients with Mental Health Conditions Using the Yoyang Refractory Brick Stove and Microwave Oven **WITHDRAWN**

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1. Abubakar Tafawa Balewa University Bauchi, Department of Industrial Design, Nigeria

Ceramics practice at mental health facilities in Nigeria is a novel activity. Nok relief modelling reproduction was introduced to reduce boredom and increase the self-efficacy of inpatients who signed the informed consent forms. The absence of a kiln would have brought a setback but the Yoyang Refractory Brick Stove and Microwave oven were alternative firing sources that turned the four weeks into ceramics care. Three females and ten males with bipolar I or bipolar II, as well as depression at Substance Abuse Treatment and Rehabilitation Centre, Vom Christian Hospital were part of the study. Hospital studio practice, interviews and observations using the exploratory sequential mixed method were used to assess the firing with the active participation of inpatients from preheating to full firing. Offloading the Yoyang stove was an engaging moment in the studio, and inpatients asked probing questions, some showed surprise at the outcome, in excitement, and some whistled and screamed. The final phase was the burnishing and the enamelling of wares with the microwave oven. The result of the bisque firing revealed complete and incomplete combustion respectively using the Yoyang stove. The studio environment changed with 90% reporting they had never seen a firing before, while 70% said it was an exciting activity that increased their enthusiasm for ceramics.

#### (ICC-P037-2024) Beyond Transparency: Unraveling the Four Aspects of Nd<sup>3+</sup> Doped Glass-Physical, Optical, Structural, and Thermal Analysis

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In the present study, Nd<sup>3+</sup> rare earth ion-doped telluroborate glass samples were synthesized using melt quenching techniques. The glass compositions were (50-x)B<sub>2</sub>O<sub>3</sub> 20TeO<sub>2</sub> 15Mg<sub>2</sub>CO<sub>3</sub> 15K<sub>2</sub>CO<sub>3</sub> xNd<sub>2</sub>O<sub>3</sub> (where x = 0, 0.5, 1, 1.5, 2 and 2.5 mol %). The study involved characterizing samples through various techniques, including XRD, DTA, FTIR, Raman, optical absorption, photoluminescence, and decay curve analysis. All samples showed an amorphous nature in XRD spectra and DTA thermograms revealed thermal behavior. FTIR and Raman spectroscopy confirmed the coexistence of trigonal BO<sub>3</sub> and tetrahedral BO<sub>4</sub> units. Optical absorption spectra indicated an increase in band gap energy with dopant concentration. Judd-Ofelt) intensity parameters were determined and spectroscopic parameters like spontaneous radiative transition probability, fluorescence branching ratio and emission cross-section were calculated. The study also explored the impact of Nd<sub>2</sub>O<sub>3</sub> concentration on physical parameters, revealing increased density, molar volume, and refractive index with higher Nd<sub>2</sub>O<sub>3</sub> concentration. In conclusion, this study provides insights into the potential applications of Nd<sup>3+</sup> doped telluroborate glass in advanced optical and photonic devices, contributing to the field of glass science and materials engineering.

#### (ICC-P038-2024) Geomechanical characterization of high organic matter lateritic clays and termite mound materials for sustainable building materials: Adamawa-Cameroon

- M. Ngono Mbenti Emvana<sup>1</sup>; R. Ntouala<sup>\*2</sup>; R. Kaze<sup>3</sup>; A. Ngo'o Ze<sup>1</sup>; V. Onana<sup>1</sup>
- 1. University of Yaounde 1, Department of Earth Sciences, Cameroon
- 2. University of Maroua, Department of Mining Engineering and Mineral Processing, National Advanced School of Mines and Petroleum Industries, Cameroon
- 3. University of Ngaoundere, Department of Mineral Engineering, School of Chemical Engineering and Minerals Industries, Cameroon

The Meiganga high organic matter lateritic clays and termite mounds were characterized for their use as sustainable construction materials. Mineralogy and geochemistry of major elements were determined by X-Ray Diffraction and X-Ray Fluorescence, respectively. Subsequently physical and mechanical properties of unfired and fired bricks at 850, 950 and 1050°C were evaluated. They are composed of quartz, kaolinite, hematite, goethite, gibbsite, muscovite, and anatase. After firing at 1050°C, mullite is formed at the expense of kaolinite, while quartz, muscovite, hematite, and anatase remain present. The studied SiO<sub>2</sub>- and Al<sub>2</sub>O<sub>3</sub>-rich materials show low values of SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> ratio, indicating their relatively abundance of kaolinite. The high organic matter content induces the development of secondary porosity favorable to produce sustainable building materials as their particle size distribution. This high organic matter content has effects on the overall physical and mechanical characteristics of the Meiganga materials and hides the influence of the parent rock or their provenance. Considering these characteristics, four clay materials are suitable to produce sustainable bricks at all the studied temperatures whereas two others are adequate only after firing at 1050°C.

### (ICC-P039-2024) Challenges associated with the processing of barium titanate nanoparticles in aqueous environments

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- 1. Paris Lodron University Salzburg, Chemistry and Physics of Materials, Austria

Barium titanate, BaTiO<sub>3</sub>, is an important dielectric material for research and industry. Due to its outstanding ferroelectric properties, it is widely used in multi-layer ceramic capacitors, sensors or random-access memories. To engineer materials with novel properties, nanocrystalline BaTiO<sub>3</sub> can be used. Aiming at 1-dimensional

BaTiO<sub>3</sub> composite nanostructures, we formulated BaTiO<sub>3</sub> nanoparticles for electrospinning and observed significant structural and compositional changes. Instead of observing polymer-nanofibers with homogenously distributed nanoparticles, those fibers contained large and well-defined micrometer sized rod-shaped structures. Systematic experiments using X-ray diffraction and electron microscopy revealed that those materials transformations originate in the high reactivity of the gas-phase grown BaTiO<sub>3</sub> nanoparticles in aqueous environments. This mechanism involves the incongruent dissolution of barium ions and the subsequent reaction with dissolved CO<sub>2</sub>. The influence of different materials properties like particle size, surface area and crystallinity were determined, as well as the influence of the polymer needed for electrospinning. This study points out the importance of the understanding and precise control of materials properties during synthesis, storage and processing, since unexpected physical and chemical interactions can occur at the particle interfaces.

#### (ICC-P040-2024) Investigation of Agricultural Waste-Derived Amorphous Silicate Materials for Thermal Management Applications

S. E. Manlupig\*2; L. Jabile2; M. Fuji1; I. B. Arugay2; A. Caamino2;

- C. V. Cahimtong<sup>2</sup>; E. A. Limbaga<sup>2</sup>; R. V. Virtudazo<sup>2</sup>
- 1. Nagoya Institute of Technology, Japan
- 2. Mindanao State University Iligan Institute of Technology, Department of Materials and Resources Engineering and Technology, Philippines

The study presents the investigation of amorphous silicate materials (ASM) from agricultural wastes sourced from South-Central Mindanao, Philippines. Studies reveal the properties of ASP including Particle Size Analysis, Adsorption-Desorption, TG DTA, FTIR, XRD, XRF, SEM and Thermal Conductivity Analysis. Results reveal that the properties of ASP could potentially contribute in developing functional materials using novel techniques intended for thermal management applications.

#### (ICC-P041-2024) Electronic and photocatalytic properties of colloidal one-dimensional titanium oxide lepidocrocite nanofilaments

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One-dimensional filaments with a titanium oxide lepidocrocite structure (1DL) have proven to be active in photocatalytic applications. As a material with a base unit of just 3x2 titanium octahedra in cross section, they exhibit quantum confinement in two dimensions. Their unique electronic properties will be presented. In the context of application, as a highly negatively charged surface, the colloidal 1DLs destabilize to form flocs upon addition of organic cations. They exhibit exceptional affinity for cationic dyes – with uptakes of nearly 0.8 g dye per g of 1DL. This high adsorption capacity enables sensitization behavior, in which dyes can be decolorized by more than 70% in 30 min, using only the visible spectrum.

#### (ICC-P042-2024) Activation and use geopolymer as porous support for FeOx deposition via glidarc plasma hydrolyticprecipitation: Application for catalytic oxidation *WITHDRAWN*

- M. Pitap Mbowou<sup>\*1</sup>; F. Boyom Tatchemo<sup>1</sup>; S. Laminsi<sup>1</sup>
- 1. Université de Yaoundé I, Chimie inorganique, Cameroon

In this study, we investigated the possibility of dissolving the basic phases and impurities contained in volcanic ash to make it porous and further use as a framework to support, through glidarc plasma route, iron oxides as a good catalyst to effectively degrade Rhodamine 6G in aqueous solution. The different samples obtained were characterized. The results of Nitrogen Physisorption show that sulfuric acid activation of volcanic ashes increases the total pore volume from 0.0026 cm<sup>-1</sup> to 0.264 cm<sup>-1</sup> as well as its specific surface area, which ranges from 1.5 to 287 m<sup>2</sup>/g. In addition, the insertion of iron nanoparticles by hydrolytic precipitation assisted by glidarc

plasma process increases the specific surface area of the activated material from 287 m<sup>2</sup>/g to 330 m<sup>2</sup>/g. Ageing of the plasma-supported material in a water bath reduces the specific surface area of the material (306 m<sup>2</sup>/g). XRD analyses confirm that iron oxides confined in these different materials are contained respectively within Augite for the supported material, within sodium Diopside for the aged material and within the goethite for both materials. Catalytic activity of different materials shows that, the ageing material give the best decolorization efficiency of Rh 6G (98%) compared to the supported material 94% and the raw material (75%) in the same condition.

### (ICC-P043-2024) Compression strength of soil-cement admixtures with addition of activated alumine waste

M. E. Mota Garboggini Guerra<sup>\*1</sup>; R. Pacheco Evangelista<sup>1</sup>; I. B. Gusmão Santos<sup>1</sup>; C. A. França<sup>1</sup>; C. T. de Oliveira Lima<sup>1</sup>; A. J. Rocha Palma<sup>1</sup>; M. Antunes Leão<sup>1</sup>

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Soil cement is a building material that can be used as a destination for solid wastes such as activated alumina, used to purify poliolefins. This study evaluates how soil-cement's compressive strength reacts to the addition of this waste to assess whether this admixture can be used as a material to make construction bricks or not. Four soil-cement admixtures were tested: one with no addition of alumina, which was used as a control, and the other three contained 5%, 10%, and 15% of alumina waste. Four specimens were made for each admixture and tested for compression strength following the NBR 12025 standard. After this test, the specimens' microstructures were analyzed using scanning electron microscopy. The results showed that the addition of alumina residue gradually reduces the avarage compressive strength to below 2 MPa, the minimum strength required by the NBR 8191 standard, which determines the requisites to validate soil-cement bricks. The microscopies showed poor adhesion of the alumina grains to the soil-cement matrix, which can be the major factor contributing to the reduction. Among the admixtures with alumina waste, the one with 5% of alumina showed the best result with an average of 1,7 MPa of compresive strength, with some individual specimens reaching 2 MPa. For this reason, it's worthy to continue the study with further experiments using lower alumina waste content.

### (ICC-P044-2024) Effect of activated alumina waste on water absorption rates of soil cement admixtures

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- 2. Federal Institute of Science, Education and Technology of Bahia, Academic Department of Mechanical Technology, Brazil

Activated alumina is used as an adsorbent for the purification of poliolefins, and it becomes a solid waste in the chemical industry after its usage. Many researchers investigate if some building materials can work with the addition of solid waste to give these pollutants an environmentally friendly destiny. This research tested soil cement admixtures with the addition of activated alumina waste in relation to their water absorption rates to see whether they were suitable to manufacture soil cement bricks or not. To achieve this goal, four admixtures were made, one with no waste to be used as a control and the other three with the following proportions of alumina: 5%, 10%, and 15%. The specimens were produced and tested for water absorption according to standards NBR 12024 and NBR 13555, respectively. The specimens also had their microstructure analyzed using scanning electron microscopy. The addition of activated alumina waste reduced the water absorption rates for all admixtures, and all of them were below the 20% threshold fixed by NBR 8191 standard, which is the maximum average water absorption rate of soil cement bricks. Therefore, activated alumina waste is being used as a partial substitute in soil cement admixtures used in construction bricks.

### (ICC-P045-2024) Novel Cermet Waste Forms for Nuclear Applications

- A. S. Piechota\*1; N. R. Sponenberg1; N. Marrero1; S. K. Sundaram2
- 1. Alfred University, Engineering, USA
- 2. Alfred University, Inamori School of Engineering, USA

Cermets or ceramic metal matrix composites show promise as versatile waste forms for a wide range of nuclear waste streams. Combination of metals and ceramics in cermets can potentially enhance the ductility, chemical durability, and overall strength of the waste forms. We have produced two series of cermet samples containing sodalite-stainless steel 316 and silicon carbide-Hastelloy. We varied the ceramic-metal ratio and composition, 25 - 50 volume% of ceramics and used hot uniaxial pressing (HUP) to densify these samples. We have characterized the cermet samples using scanning electron microscope and electron dispersive spectroscopy (SEM-EDS), the immersion method for density, mercury porosimetry, and x-ray diffraction (XRD). We will present the preliminary microstructure development, density, and porosity of these waste forms demonstrating viability of these novel waste forms for nuclear applications.

### (ICC-P046-2024) Smart nanocomposite in the TiC-BN-SiC-B<sub>4</sub>C-SiAlON-Al<sub>2</sub>O<sub>3</sub> system

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Our task was to study the phase composition of received consolidated materials in the TiC-TiB<sub>2</sub>-BN-SiC-B4C-β-SiAlON-Al<sub>2</sub>O<sub>3</sub> system. The obtained mass was grounded in an attritor and the consolidated composite was obtained by hot pressing at 1620°C during 40 minutes, with glass perlite dope 2mass%, delaying at final temperature for 8min, under 30MPa pressure and vacuum 10<sup>-3</sup>Pa. Perlite from Aragatc contained 96 mas% glass. In TiC-TiB<sub>2</sub>-BN-SiC-B<sub>4</sub>C- $\beta$ -SiAlON-Al<sub>2</sub>O<sub>3</sub> system we obtained nanocomposites with high mechanical properties. The compounds, which are newly formed thanks to interaction going on at thermal treatment: Si<sub>3</sub>N<sub>4</sub>, Si,AlN are active, which contributes to β-SiALON formation at relatively low temperature, at 1300-1350°C. It is evident that inculcation of ALN in crystal skeleton of B-Si<sub>3</sub>N<sub>4</sub> is easier since at this temperature interval crystal skeleton of Si<sub>3</sub>N<sub>4</sub> is still in the process of formation. ß-SiAlON was formed at 1450°C. Part of boron carbide was transformed into boron nitride in nitrogen environment and in titanium diboride, The phase composition of the obtained composite provides high physical-technical and performance properties of these nanocomposites. Compression strength-2198MPa, Bending strength-271MPa. Acknowledgment We express our gratitude to Shota Rustaveli Georgian National Science Foundation. The work is done with the grant of the Foundation FR-21-1413 Grant 2022.

#### (ICC-P047-2024) Evaluation of the Thermodynamic and adsorption properties of strangler fig leaves extracts as a bio corrosion inhibitor of mild steel in acidic medium

H. C. Olisakwe\*1; D. T. Ipilakyaa1; J. L. Chukwuneke1

1. Nnamdi Azikiwe University, Mechanical Engineering, Nigeria

The strangler fig leaves extracts possess -OH, -COOH, -COOR, -NO, C-Halogen which are corrosion inhibitive functional group, when subjected to Fourier Transform Infrared spectroscopic analysis, the corrosion rate and inhibition efficiency results after two hours of exposure at various extract concentration and temperatures shows that strangler fig leave extract has potent corrosion inhibition property. It also exhibited some thermodynamic properties which shows that there was physical adsorption mechanism between the extract and the surface of the mild steel. The activation energy values were between 9.573-11.488KJ/Mol, lower than the threshold value of 80KJ/Mol required for chemical adsorption to take place, hence physical adsorption occurred, the Gibbs free energy values were all less than -20KJ/Mol, this affirms that the adsorption mechanism is spontaneous and exothermic in nature, hence physical adsorption took place on the surface of the mild steel. The heat of adsorption values are also negatives, also signifying exothermic reaction, hence physical adsorption. The adsorption equilibrium constant values reduce as extract concentration increases. The enthalpy of adsorption values are quite lower than 100KJ/Mil which indicates a physical adsorption.

#### Wednesday, July 17, 2024

#### **Plenary Session**

#### Plenary Session: Diletta Sciti

Room: Montreal 1-3

#### 8:30 AM

### (ICC-PLE-004-2024) Could UHTCs and UHTCMCs be enabling solutions for extreme environments?

D. Sciti<sup>\*1</sup>

1. Consiglio Nazionale delle Ricerche, ISSMC (former ISTEC), Italy

New space exploration trends require materials capable of withstanding extreme universe conditions. 'Hostile environments' cover thermochemical, thermophysical, and thermomechanical challenges that may occur simultaneously. Ultra-high temperature ceramics (UHTCs), like transition metal carbides, nitrides, and diborides, meet these demands with melting points exceeding 3000°C and high thermal conductivity (>100 W m-1 K-1). They form stable, high melting temperature oxides and boast remarkable mechanical strength. More recently, the severe requirements of hypersonic travel have led to the creation of composites known as ultra-high temperature ceramic matrix composites (UHTCMCs), made of UHTC matrices incorporating reinforcing fibrous phases. These composites represent an advancement over UHTCs due to their superior damage tolerance and thermal shock resistance, and can be considered a novel subfield of CMCs for their ability to operate at temperatures much higher than 1600°C. This lecture will outline key research on ultra-high temperature ceramics (UHTCs) and composites (UHTCMCs). The covered topics range from understanding process, microstructure and properties relationship, to functionalizing materials through appropriate methodologies, and to creating demonstrators for technology validation in relevant environments up to 2500°C. Scale-up issues and technological challenges will also be addressed.

# Symposium 1: Aerospace Ceramics and Composites

#### Symposium 1: Aerospace Ceramics and Composites Room: Montreal 1-3

Session Chair: Christopher Weinberger, Colorado State University

#### 10:10 AM

#### (ICC-SYM1-029-2024) Mechanical Testing of Carbon Fiber-Reinforced Silicon Carbide Composites (C/C-SiC): Size effect of bending and tensile strength

S. Flauder<sup>\*1</sup>; N. Langhof<sup>1</sup>; S. Schafföner<sup>1</sup>

1. University of Bayreuth, Chair of Ceramic Materials Engineering, Germany

Ceramic matrix composites (CMC) overcome the brittleness of monolithic ceramics and combine high thermo-mechanical resistance with low density and damage tolerant material behavior. The study reports the dependency of strength from the tested size of the sample for carbon fiber-reinforced silicon carbide composites (C/C-SiC). For this purpose, different sample sizes and geometries were tested under bending and tensile loads. The C/C-SiC samples were fabricated by the liquid silicon infiltration (LSI) process with a plain weave fabric reinforcement. Furthermore, a test device for self-alignment and centering of tensile samples was developed to minimize errors in tensile testing. The statistical behavior and the influence of the sample size and testing method on the strength of C/C-SiC was evaluated. A decrease of the bending strength with increased tested specimen size was determined. It was further found that the Weibull modulus was not a material constant. The Weibull modulus depended on the load condition and the tested volume. The size effect of strength was discussed regarding the different concepts of statistical and quasi-brittle fracture mechanics. The classical probabilistic aspects of brittle fracture with the application of Weibull statistics could not properly describe the size effect of strength.

#### 10:30 AM

# (ICC-SYM1-030-2024) Fatigue of a SiC/SiC Ceramic Composite with an Ytterbium-Disilicate Environmental Barrier Coating at Elevated Temperature

T. Wiliams<sup>1</sup>; M. Ruggles-Wrenn<sup>\*1</sup>

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<sup>™</sup> 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<sub>2</sub>Si<sub>2</sub>O<sub>7</sub> top coat applied via air plasma spraying. Tensile properties were evaluated at 1200°C. Tensiontension 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.

#### 10:50 AM

#### (ICC-SYM1-031-2024) Characterisation and Testing of 3D-printed ceramic and PM-parts for Aerospace application with novel Thermo-Optical-Measuring technique

A. Diegeler\*1; M. Kilo1

1. Fraunhofer ISC, Center of Device Development, Germany

Thermo-optical measurement will be used to test and inspect additive manufactured ceramic and PM-parts during the whole temperature cycle inside the furnace to evaluate full functionality. This method allows to follow changes of sample shape (up to 50mm in diameter) with an accuracy of nearly 0.3 micron by contactless measurement. Due to the purely optical detection samples of any shape can be monitored inside the furnace. A sophisticated algorithm detects the contour of samples - and thus dimensional changes. Possible applications include simply measuring the shrinkage or shape distortions. By a computer-controlled program we realized full atmospheric control, i.e.changing the gas atmosphere inside the furnace (inert gas at different pressures, forming gas or vacuum) during the sintering process. Main focus according additive manufactured ceramic and PM-parts lies on quality and quality control during production process, and on characterization of behavior under thermal or other stress application. Additional modules as load-, balance- or gas-analysis - module are appliable to the TOM system to detect in-situ creep, crack or degasing character of the parts during heat treatment. This data is highly needed for the optimization of production process and only available with innovative TOM-Technique.

#### 11:10 AM

### (ICC-SYM1-032-2024) R-Curve Determination of highly densified Si3N4

M. Mazaheri\*1

1. SKF, Research & Technology Development, Netherlands

This study focuses on the R-curve measurements of bending samples composed of five distinct highly densified Silicon Nitride materials. Testing was conducted during stable crack growth using the SEVNB sample and compliance method on an exceptionally rigid bending machine. The machine's high stiffness was crucial to absorb minimal elastic energy during loading, facilitating stable crack advancement. The initial two samples, featuring a relative notch depth of 57%, experienced unstable fractures. This was attributed not only to the inherent brittleness of the material but also to the higher force required to initiate crack propagation from the notch tip compared to achieving stable crack advancement. Addressing this challenge, new samples were designed with a relative notch depth of 68%, effectively resolving the issue. This underscores the dependency of R-curve validity on the size of the notch. Evaluation of load versus displacement measurements during fracture revealed a steep R-curve. The study demonstrated that the "length of R-curve," representing the range where the R-curve reaches 90% of its saturation value, is notably short for fine-grained silicon nitride material, approximately 10 µm. While all tested silicon nitrides specimens exhibit R-curve behaviour, the plateau value is reached at distinctly different crack length increments, dependent on the microstructure, specifically the grain size.

# Symposium 3: Educational Trends in Ceramics and Glass

### Symposium 3: Educational Trends in Ceramics and Glass

Room: Montreal 6 Session Chair: Brian Gorman, Colorado School of Mines

#### 9:50 AM

(ICC-SYM3-001-2024) Creating Engaging Learning Environments for Special Learners Through Multisensory Exploration with Clay in the Art Ability Project, Nigeria (Invited) *WITHDRAWN* 

E. Dokyoung\*<sup>1</sup>; E. Abubakar<sup>1</sup>; N. Gomwalk<sup>2</sup>

- 1. Modibbo Adama University of Technology, Department of Industrial Design, Nigeria
- 2. University of Jos, Department of Special Education and Rehabilitation Sciences, Nigeria

The art knowledge gap among teachers and lack of art exposure to learners with learning disabilities (LD) and intellectual disabilities in the Federal Capital Territory, Plateau, Ondo, Nassarawa, and Adamawa States of Nigeria, contributes to a lack of inclusivity and inadequate art education, which hinders creativity, self-expression and multisensory learning. Art Ability Project at Lutheran Deaf School, Jimeta, REMI Educational Foundation, Special School, Yola, and FOMWAN School for the Deaf, Song, exposed the need for qualified art teachers and parents who know how to transform Africa's rich artistic cultural heritage and abundant natural resources to make up for scarce resources. To compensate for the special education students' subpar academic performance, poor visual-motor coordination, and lack of art supplies, volunteer professional artists simplified indigenous arts through classroom engagements, workshops and exhibitions and taught simple clay modelling techniques, alternative art tools/ materials from waste and found objects. Results of the activities show that art education was more accessible, inclusive, and attractive to learners. Schools got art supplies, explored circular economy and exposed teachers to Design Thinking for active engagements, as well as improving the well-being of learners through self-expression.

#### 10:20 AM

#### (ICC-SYM3-003-2024) Enriched Active Learning Through Design of Experiments & Option-Based Parameters Lab Courses

K. Scott\*

1. Colorado School of Mines, Metallurgical & Materials Engineering Dept, USA

Traditional lecture-lab courses often struggle to engage students in meaningful learning experiences. To address this challenge, educators are increasingly incorporating active learning methods to promote critical thinking and deeper engagement. Herein the incorporation of design of experiments (DOE) and option-based parameters lab courses is discussed for the Ceramic Engineering Program at Colorado School of Mines. DOE methodology offers students the opportunity to develop critical thinking skills and experimental design expertise. By guiding students through the process of planning experiments, manipulating variables, and analyzing results, these labs facilitate connections between fundamental concepts and practical implications. Moreover, this approach encourages students to think beyond prescribed procedures, fostering creativity and innovation in problem-solving. In addition, option-based parameter experiments provide students with autonomy and flexibility in their learning journey. It enhances student engagement and encourages exploration and discovery, thereby fostering a sense of ownership over their learning process. The combination of lecturebased instruction with hands-on lab experiences creates an enriched learning environment that promotes interdisciplinary understanding, better preparing students to become engineers.

#### 10:40 AM

# (ICC-SYM3-004-2024) The challenge of preserving and the transmission of traditional techniques in a contemporary factory: Realities of the field

H. Panhard Lathoud\*1

1. University of Jean Monnet Saint Etienne, Art History and Design, France

In an art factory, the hand gestures are essential, and the work is specialized by trade (Adamson, 2013). The transmission and preservation of craft trades and their traditional techniques has always been central. However, the role of traditional skills and their transmission now needs to evolve, to enable them to co-exist with an extremely advanced industrial function (Branzi, 1985), allowing large production volumes. Based on a case study of the French Revol Porcelaine factory, we propose to study the challenges and realities of preserving traditional techniques in the manufacturing process of a tableware factory. Revol Porcelaine exists for over 255 years and has preserved the specific know-how of its trades. The corpus of this study consists of observation sessions of the manufacturing process within the different units of the factory, semi-directive interviews (with different workshops and the management team) and the archives of the factory. The findings and data were then analyzed using an empirical qualitative method, with a view to understanding and interpreting them. This analysis reveals that the cohabitation with industrial processes renews and enhances the role for traditional techniques. It also shows that transmission in the field through training and education encourages a fruitful dialogue between tradition and innovation.

45

#### Symposium 5: Global Ceramics and Glass Industry Trends

### Symposium 5: Global Ceramics and Glass Industry Trends

Room: Montreal 7 Session Chair: Kristin Breder, Saint Gobain Crystals

#### 9:50 AM

### (ICC-SYM5-001-2024) A private equity perspective on clean energy investment in the ceramics industry (Invited)

A. Ku\*1; M. Fransson1; S. Hobart1

1. Mercator Partners, USA

The transition to decarbonized economies is expected to require trillions of dollars in annual investment globally through mid-century. Governments around the world are currently offering substantial public support, but the large-scale transformation of energy, industrial, and transportation ecosystems will not occur without massive mobilization of investment from the private sector. This presentation will discuss the intersection of private equity investment, the energy transition, and the ceramics industry. Specific examples will be used to illustrate the role of private equity can play in supporting the development and deployment of ceramics-enabled clean energy technologies.

#### 10:20 AM

#### (ICC-SYM5-002-2024) An Overview of Industrial Demonstration Activities within the U.S Department of Energy's Office of Clean Energy Demonstrations (OCED) (Invited)

K. Hoesch\*<sup>1</sup>

1. Department of Energy, Office of Clean Energy Demonstrations, USA

Industry is a backbone of the U.S. economy, producing the goods critical to everyday life, employing millions of Americans in highquality jobs, and providing an economic anchor for thousands of communities. Yet the energy- and carbon-intensity of the sector, which contributes nearly one third of the nation's primary energyrelated carbon dioxide emissions, poses a significant challenge as the economy transitions towards net-zero. The U.S. Department of Energy (DOE) established OCED to deliver clean energy demonstration projects at scale in partnership with the private sector to launch or accelerate market adoption and deployment of technologies as part of an equitable transition to a decarbonized energy system. To further these efforts, the DOE released a Funding Opportunity Announcement (FOA) in 2023. When combined with private sector cost share, this FOA represents a more than \$12 billion opportunity to catalyze high-impact, large-scale, transformational advanced industrial facilities to significantly reduce greenhouse gas (GHG) emissions in energy-intensive industrial subsectors. This presentation will cover the DOE's latest assessment of the opportunity space for the Industrial Demonstrations Program to catalyze first- or earlyof-a-kind decarbonization approaches across heavy industry, with a focus on ceramics and glass.

#### 10:50 AM

#### (ICC-SYM5-003-2024) Global Supply Chain Issues as Barriers to Advanced Materials Development, Growth and Implementation (Invited)

J. A. King\*1

1. Golden Bear LLC, USA

Continuous access to sufficient quantities of critical elements is essential for the growth of advanced materials and supporting technology development. Currently, the U.S. government lists 50 elements (or ores) as critical materials for economic growth and national security. Most of the key materials are from imports into the U.S. where some imports make-up the bulk or sole source of the material; the U.S. is 100% reliant on imports for at least 12 key elements/minerals deemed critical by the government, with China as the primary supplier and an additional 18 have import percentages ranging from 50% to 96%. The talk will cover which strategic elements are most central to existing and emerging advanced ceramics, their suppliers and some of the geopolitical issues associated with sufficient material access.

#### 11:20 AM

### (ICC-SYM5-004-2024) Process digitization of battery-grade ceramics using laser-induced breakdown spectroscopy

M. Michaud Paradis\*1; S. Rousselot1; K. Rifai2; M. Bertrand1;

- A. Hernández-García3; F. Doucet2; L. Özcan2; N. Azami2; M. Dollé1
- 1. Université de Montréal, Chemistry, Canada
- 2. ELEMISSION, Inc., Canada
- 3. Mila Université de Montréal, Canada

Recent Canadian investments in energy storage contribute to the planning of modern raw materials, fabrication, and recycling plants in the next decade. Although digitization is a hot topic in order to improve process efficiency and safety, its introduction to modern infrastructure designs is limited by the lack of fast-acquisition sensors sensitive to battery material contents. Lack of sensitivity to light elements is especially constraining in the context of lithium-ion battery materials. Laser-induced breakdown spectroscopy (LIBS) is a new type of sensor for elemental content analysis requiring little to no maintenance and sample preparation and has made its proof in automated fast-acquisition of raw material process analysis. Furthermore, its sensitivity to battery materials in the context of exact composition analysis has been demonstrated in our recent work. In the current study, we investigate LIBS composition and outlier metrics augmented by GPU integration and artificial intelligence in the context of real-time sensing (< 1s per sample) applied to lithium-bearing ceramics. We also report new real-time sensing metrics such as the quantity of information acquired and processed per second.

#### 11:40 AM

# (ICC-SYM5-005-2024) How did the versatile borosilicate float glass support various industry trends during the last 30 years and how will it continue to do so?

J. Brandt-Slowik\*1

1. SCHOTT Technical Glass Solutions GmbH, Germany

Borosilicate float glass is used in a large variety of applications among others in products like fire resistant glazing, heat resistant doors in pyrolytic ovens, impact-resistant glazing, glass used in diagnostics or as carrier wafers in semiconductor production. Myriad applications are enabled by the special properties of borosilicate glass. Borosilicate glass has been successfully utilized for decades in the laboratory where chemical resistance as well as low coefficient of thermal expansion, leads to its outstanding thermal shock and thermal gradient resistance. Critical functions in this aggressive environment. Additional features such as high optical transmission and a low autofluorescence, enable optical applications as well as in diagnostics. Mechanical properties, like superior stone impact resistance and enhanced scratch resistance open up other fields of application. Many unique properties are based on the special structure of borosilicate glass, which is presented in comparison to the one of the widely used standard soda lime glass. The differences in the glass structures will be explained, as the structure of a material determines its properties and the properties enable the use in certain applications.

#### 12:00 PM (ICC-SYM5-006-2024) Using Ceramic Society authority to develop standards for the public good WITHDRAWN

R. Bowman\*1

1. Intertile Research, Australia

While Ceramic Societies commonly provide a range of services to assist students, members and industry development, they can also deliver significant public benefits by utilising their resources, particularly intellectual capital, to help develop normative and informative Standards. This paper provides examples of how the Australian Ceramic Society has contributed to improving the quality of standards for ceramic tiles, the performance of tiling systems, the development and improvement of test methods, the selection of products based on expected service demands, the identification of compatible maintenance products and processes, as well as the minimisation of occupational diseases and slip-induced accidents. Such an approach is based on increasing the life cycle performance of systems at economically reasonable costs, while also contributing to the fulfilment of some of the UN Sustainable Development Goals.

# Symposium 6: Informatics and data analytics in ceramics and glass

### Symposium 6: Informatics and data analytics in ceramics and glass

Room: Montreal 8

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

#### 9:50 AM

### (ICC-SYM6-001-2024) Towards an End-to-end Framework using Artificial Intelligence for Materials Discovery (Invited)

N. Krishnan\*1

1. Indian Institute of Technology Delhi, Civil Engineering, India

Traditional materials discovery relies on trial and error approaches, leading to a design-to-deploy period of 20-30 years. To address this challenge, in this talk, we will discuss the application of artificial intelligence (AI) in accelerating materials modeling and discovery. Specifically, three aspects, namely (i) natural language processing (NLP) for information extraction, (ii) data-driven materials modeling, and (iii) physics-informed machine learning for materials modeling, will be discussed. The first focuses on extracting information on glasses and other materials from literature to answer specific queries. We will also discuss how MatSciBERT, the first materials-aware language model, can remove information regarding composition properties from the glass literature. Second, we focus on developing interpretable ML models for predicting properties based on the information extracted from the literature. This work covers nearly the entire periodic table for glass-forming elements. Third, we will discuss accelerating molecular simulations using physics-informed ML (PIML).

#### 10:20 AM

#### (ICC-SYM6-002-2024) Combining first-principles calculations and machine-learning to predict the synthesizability and properties of high-entropy materials (Invited)

C. Toher\*

1. The University of Texas at Dallas, Materials Science and Engineering, USA

The successful development and manufacturing of new materials, for applications ranging from batteries, catalysts and electronics to thermal protection barriers in aerospace engineering, requires extending computational approaches beyond those required for ordered materials to incorporate the effects of disorder and entropy. Descriptors and thermodynamic models have been developed to

#### 10:50 AM

### (ICC-SYM6-003-2024) Experimentalist Approach to Materials Discovery from Data Aggregation and Analytics (Invited)

V. Taufour<sup>\*1</sup>

1. UC Davis, Physics and Astronomy, USA

Despite advances in high throughput calculations, artificial intelligence, and automated synthesis, the rate of material discovery remains the same since 1960s, hindering progress in addressing the global challenges in energy, sustainability and healthcare. One of the major bottlenecks remains the identification of new structural families with targeted properties, as opposed to variation in composition of well-established structure types. Another challenge is the discovery of materials with rare or unconventional properties, for which only small datasets and limited theoretical understanding exist. Additionally, the lack of an efficient feedback loop between failed experiments and theoretical predictions impedes progress. Databases containing experimental results are much rarer and typically smaller than theoretical datasets. We aggregated a database of ~4000 Curie temperatures, and analyzed data trends to identify and then experimentally discover new ferromagnetic materials. I will discuss how natural language processing can rapidly expand the dataset and improve machine learning prediction of the Curie temperature. I will also illustrate how candidate materials with a rare property such as spin-triplet superconductivity can be identified despite the limited number of examples. Finally, I will provide a quantitative example of experimental verification of high-throughput calculations.

#### 11:20 AM

### (ICC-SYM6-004-2024) From BIG-data to HOT-properties of high-entropy carbides and carbo-nitrides (Invited)

S. Curtarolo<sup>\*1</sup>

1. Duke University, Center for Extreme Materials, USA

The need for improved functionalities in extreme environments is fueling interest in high-entropy ceramics. Except for the computational discovery of high-entropy carbides, performed with the entropy-forming-ability descriptor, most innovation has been slowly driven by experimental means. Hence, advancement in the field needs more theoretical contributions. Here we introduce disordered enthalpy-entropy descriptor (DEED), a descriptor that captures the balance between entropy gains and enthalpy costs, allowing the correct classification of functional synthesizability of multicomponent ceramics, regardless of chemistry and structure. To make our calculations possible, we have developed a convolutional algorithm that drastically reduces computational resources. Moreover, DEED guides the experimental discovery of new single-phase high entropy carbonitrides and borides. This work, integrated into the AFLOW computational ecosystem, provides an array of potential new candidates, ripe for experimental discoveries.

#### 11:50 AM

#### (ICC-SYM6-005-2024) New Superhard Boride Ceramics from a Combined Computational and Experimental Study (Invited) N Obradovic<sup>\*1</sup>

N. Obradovic<sup>\*\*</sup>

1. Institute of Technical Sciences of SASA, Serbia

A new super-hard (Hf,Mo,Ti,V,W)B<sub>2</sub> was identified by machine learning, predicted by be stable by thermodynamic simulations, and then synthesized by boro-carbothermal reduction. This composition

was produced for the first time as a single-phase ceramic in the present research. The optimized ceramic had a single hexagonal AlB<sub>2</sub>-type crystalline phase with a grain size of 3.8 µm and homogeneous distribution of the constituent metals. The Vickers hardness exhibited the indentation size effect, increasing from 27 GPa at a load of 9.8 N to as high as 66 GPa at a load of 0.49 N. This is the highest hardness reported to date for high entropy boride ceramics. The presentation will discuss computational methods for identification of new compositions with superior properties.

#### 12:20 PM

#### (ICC-SYM6-006-2024) Oxygen vacancy formation energetics in MgO-based high entropy oxides from DFT and experimental validation

O. Opetubo<sup>1</sup>; T. Shen<sup>1</sup>; R. Bordia<sup>1</sup>; D. Aidhy<sup>\*1</sup>

1. Clemson University, Materials Science and Engineering, USA

In contrast to single or double cation based oxides, high entropy oxides (HEOs) consist of equi- or near equimolar concentation of multiple cations randomly distributed on a crystal lattice. The random distribution causes a wide diversity of nearest-neigbor environments around oxygen atoms. Consequently, a range of bonding environments and oxygen vacancy formation energies are observed. We investigate oxygen vacancy formation energies in Mg(ZnCoNiCu)O-based HEO using density functional theory (DFT). We find that vacancy formation energy trends are controlled by cation valence charge. We explicitly determine the effect of all cations on formation energies. Experimentally, a series of binary, ternary, quaternary and quinary HEOs are synthesized using a simple and scalable polymeric steric entrapment method. By annealing in inert environment, oxygen vacancies are analyzed by XPS and TGA. The experimental obseravtions agree with our DFT calcilations. This understanding contributes to oxygen vacancy transport useful in microstructure evolution and electrochemical applications.

#### Symposium 10: Optical and Magnetic **Ceramics Materials and Devices**

#### Symposium 10: Optical and Magnetic Ceramics Materials and Devices

Room: St. Laurent 4

Session Chairs: Victoria Blair, US Army Research Laboratory; Robert Pullar, Ca' Foscari University of Venice

#### 9:50 AM

#### (ICC-SYM10-001-2024) Wet chemical synthesis of II-VI powders for sintering transparent (Invited)

Y. W11\*

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

II-VI compounds (TM: II-VI, TM=Cr<sup>2+</sup>, Fe<sup>2+</sup>, II-VI=ZnS, ZnSe) exhibit high transparency across a wide wavelength range of 0.5-18.0µm, which makes such materials attractive for numerous scientific and technological applications. The CVD process for developing transparent ZnS and ZnSe ceramics is effective, but it is very expensive and time-consuming. In addition, post-processing of hot isostatic pressure causes a decrease in mechanical strength and hardness due to recrystallization and grain growth. Researchers have attempted to synthesize ZnS and ZnSe powders using wet chemistry methods, followed by consolidation sintering, which is a cost-effective method to fabricate optical ceramics. In our research work,  $Fe_xZn_{1-x}Se$  powders were synthesized by a liquid-phase co-precipitation method. XRD analysis of the calcined Fe<sub>x</sub>Zn<sub>1-x</sub>Se powders revealed that the powders were single-phased cubic ZnSe. Meanwhile, the synthesis of ZnS powder was performed through

a colloidal processing method. Thioacetamide  $Zn(NO_3)_2$  were dissolved in DI water, respectively. The two solutions were then mixed by stirring in a hot water bath. Finally, the as-synthesized ZnS powders were heat-treated in flowing argon to remove residual polymers. The phase composition of the ZnS powders was determined using XRD. The morphological and structural features of the ZnS powder were investigated by SEM and BET.

#### 10:20 AM

#### (ICC-SYM10-002-2024) Manufacture and properties of transparent AlON ceramics WITHDRAWN

#### X. Mao\*1

1. Shanghai Institute of Ceramics, Chinese Academy of Sciences, China

Transparent AlON ceramics are considered as promising materials for transparent armours, infrared windows, and domes. In this study, transparent AlON ceramics were sintered using AlON powders. The hardness, fracture toughness, and refractive index of the ceramics increase with an increase in the AlN content. High transmission is obtained when the AlN content was 20.8-28.2 mol%. we propose an approach to prepare pore-free and low residual stress transparent AlON ceramics doped with SiO<sub>2</sub>. Then SiO together with N<sub>2</sub> in the pores would be condensed and dissolved into AlON matrix to form Si:AlON during HIP process, making the ceramic more dense. The obtained transparent AlON ceramics kept high transmittance 85.8% and low loss coefficient 0.005 cm<sup>-1</sup> at 2000 nm after annealing. At elevated temperatures between 800 °C and 1300 °C, the N atoms was oxidized and intermediate  $\gamma'$ -AlON phase was formed with weight increasing. Based on this oxidation behavior, transparent AlON ceramics bars were treated at elevated temperature to form a compressive stress layer on its surface. The three point bending strength increase from 390 MPa to 650 MPa, by heating the 3mm\*4mm\*36mm bars at 1100 °C. The biaxial flexural strength of  $\Phi$ 11mm×0.5mm AlON ceramic sheet after heating treatment at 900°C reaches 1.5 GPa, which is far higher than the conventional strength of AlON transparent ceramic.

#### 10:40 AM

#### (ICC-SYM10-003-2024) Transparent La<sub>2</sub>Zr<sub>2</sub>O<sub>7</sub> ceramics doped with Eu<sup>3+</sup>/Ho<sup>3+</sup>

- V. Nečina\*<sup>1</sup>; J. Mrázek<sup>2</sup>; W. Pabst<sup>1</sup>
   University of Chemistry and Technology, Prague, Department of Glass and Ceramics, Czechia
- 2. Czech Academy of Sciences, Institute of Photonics and Electronics, Czechia

The research of pyrochlores  $(A_2B_2O_7)$  branches into two main fields: thermal barrier coatings and luminophores. The latter field of luminophores offers promising and relatively unexplored applications in metrology, tracking systems, optical sensors and biomedicine due to the easy substitution of A<sup>3+</sup> ion by active media (i.e.: Eu<sup>3+</sup> or Ho<sup>3+</sup>). In this contribution, the preparation of transparent La<sub>2</sub>Zr<sub>2</sub>O<sub>7</sub> ceramics doped with either Eu<sup>3+</sup> or Ho<sup>3+</sup> is presented. The complete densification was achieved at relatively low temperature ~1500 °C, pressure of 80 MPa and short processing time ~5 h by spark plasma sintering with the help of sintering additive LiF. After annealing, the prepared samples were characterized in terms of the optical properties (transmittance, emission spectrum and lifetime), thermal properties (thermal conductivity) as well as microstructure (grain size). 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).

#### 11:00 AM

### (ICC-SYM10-004-2024) Non-resonant directional random scattering laser with porous Nd:YAG ceramics

H. Ma\*1; K. Lee2; Y. Park2; D. Kim2

- 1. Korea Institute of Materials Science, Republic of Korea
- 2. Korea Advanced Institute of Science and Engineering (KAIST), Republic of Korea

Non-resonant random lasers are advantageous because they are inexpensive to fabricate and may be used with a variety of gain materials in the spectral range where conventional lasers are not available. However, because to large threshold powers, poor power efficiency, and challenges with light gathering, their practical usefulness is constrained. Herein, we use a scattering cavity in a porous Nd:YAG gain material to demonstrate a power-efficient, simple-to-fabricate non-resonant laser. Strong diffuse reflections on the inner surface of the cavity provided non-resonant feedback that allowed the laser to operate in this counterintuitive cavity. Moreover, cavities made on a porous ceramic produced notable improvements in slope efficiency, threshold power, and directionality.

#### 11:20 AM

### (ICC-SYM10-005-2024) Oxide Window and Radome Materials for Extreme Environments

R. Hay\*1

1. Air Force Research Laboratory, USA

Refractory cubic oxides are comprehensively screened for use as windows and radomes in extreme environments. Empirical and density functional theory (DFT) based modeling, along with extensive datamining of literature and on-line databases are used to screen for transparency range, dielectric constant, melting point, hardness, CTE, thermal conductivity, and other properties. Extensive digitized compilations of powder IR transmission spectra are also used Cubic oxides that have been screened include garnets, spinels, pyrochlores, fluorites, bixbyites, and beta-cristobalites. Materials with an optimal balance of optical, structural, and thermochemical properties are identified. Promising materials are processed and characterized for optical transparency at room and high temperature by highthroughput methods to validate the predicted properties. An example of the screening and characterization process is presented for garnets.

#### 11:40 AM

### (ICC-SYM10-006-2024) Synthesis and Properties of Nanostructured Glass (Invited)

S. Singh\*1

1. International Advanced Research Center for Powder Metallurgy and New Materials (ARCI), India, Center for Curanic Processing, India

In recent times, glass has attracted substantial attention in the field of nanotechnology. Reservings are exploring various nanostructured forms of glass, such as grassy nanoparticles, nanofibers, and thin films, to discover novel properties and functionalities. These nanostructured glasses are promising materials for several applications in areasily photonics, sensing, drug delivery, and nanoelectronics. Their exceptional optical, mechanical, and chemical properties make them valuable materials for advancing technology in the nanoscale regime. Here, the discussion comprehends two main types of nanostructured glass: nano-metal doped glass nanocomposites (NGNC) and nanoglass (NG). NGNC exhibits intriguing optical properties, including surface plasmon resonance (SPR) resulting from various nano-metals dispersed within the glass matrix. This phenomenon holds promise for photonic applications and enhances the luminescence of rare earths in the glass due to the local field effect of the nano-metals. Another nanostructured glassy material known as nanoglass is characterized by the presence of nano-sized glassy regions and glass-glass interfaces. Recent studies have revealed fascinating mechanical, magnetic, catalytic, and structural properties of nanoglass compared to bulk glasses of the same compositions. These developments signify the potential of nanostructured glasses to lead the next generation of nanotechnology revolutions.

# Symposium 1: Aerospace Ceramics and Composites

#### Symposium 1: Aerospace Ceramics and Composites Room: Montreal 1-3

Session Chair: Marina Ruggles-Wrenn, Air Force Institute of Technology

#### 1:30 PM

#### (ICC-SYM1-033-2024) Ceramic Protective Surfaces and Coatings: Studies under Operational Conditions (Invited)

K. Faber\*1

1. California Institute of Technology, USA

In the development and characterization of coatings or protective surfaces an essential step is the evaluation of such systems under conditions which imitate or replicate operating conditions. Such examinations not only provide a go-no go assessment, but also offer an opportunity to study the science associated with potential reactions or sources of degradation. We explore two systems as illustrations. The first, a graphite-hexagonal boron nitride (h-BN) bimaterial, is of interest to the electric propulsion community for Hall-effect thrusters since graphite and h-BN are isostructural and have comparable coefficients of thermal expansion. The bimaterial is produced by converting the graphite surface to h-BN via a carbothermic reaction, providing a composite with a conductive graphite core adhering to the dielectric and emissive h-BN surface. An essential test of the bimaterial is plasma exposure. A second example is an environmental barrier coating designed for the protection of silicon-based ceramics in gas-burning turbine engines. Among the promising environmental barriers are rare-earth silicates due to their low silica volatility. However, with water as a combustion product in such engines, enhanced oxidation due to steam poses a serious challenge. This example focuses on the use of synchrotron methods to evaluate environmental barrier coatings through ex-situ and in-situ steam-based studies.

#### 2:00 PM

### (ICC-SYM1-034-2024) Multilevel design of environmental barrier coatings for SiCf/SiC composite (Invited)

J. Wang\*1

1. Institute of Metal Research, Chinese Academy of Sciences, Advanced Ceramics and Composites Division, China

Emerging engine hot-section components are focused on SiC<sub>i</sub>/SiC composite with environmental barier coating (EBC) that can withstand harsh thermal archerence to a straight the stand harsh thermal archerence of the straight the stand attacks against combustion environment. Typical EC provide reliable protections to SiC<sub>i</sub>/SiC components below the surface temperature of 1300°C. However, the present EBCs doministrate limited capability of corrosion resistances to hot straight molten CaO-MgO-Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub> (CMAS) deposit at higher temperatures, as well as the phase instability and amorphization during coating fabrication. To address these critical challenges, new strategies are adopted to design 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<sub>i</sub>/SiC composite engine components.

49

### Abstracts

#### 2:30 PM

#### (ICC-SYM1-035-2024) Tracking mechanical and thermal cycling damage evolution in 7YSZ thermal barriers by small scale testing of free-standing cantilevers (Invited)

D. Lal<sup>2</sup>; V. Ramanadham<sup>1</sup>; P. Kumar<sup>1</sup>; S. Sampath<sup>3</sup>; V. Jayaram<sup>\*1</sup>

- 1. Indian Institute of Science, Materials Engineering, India
- 2. IIT Jammu, Materials Engineering, India
- 3. Stony Brook University, Center for Thermal Spray Research, USA

The present study explores the progressive degradation in a 7YSZ air-plasma sprayed thermal barrier coating (TBC) upon mechanical and thermal cycling. Service environments in engines introduce many complexities in the interpretation of failure given temperature gradients and long term microstructural changes such as the thickening of the thermally grown oxide and densification / partitioning of the TBC. The present study explores a narrower range of conditions in which ~ 0.3 mm, free standing cantilevers are subject to cyclic bending at room temperature before and after multiple thermal cycles between RT and 700 C. Sensitive measurements of bending stiffness in a depth-sensing nanoindentation system allow the progression of damage to be tracked as a function of applied mechanical load as well as of prior thermal cycling. Coatings undergo a slow softening accompanied by ratchetting, followed by sudden failure and the number of cycles to failure at a given maximum stress drops substantially with the number of prior thermal cycles. A model, derived from cementitious materials, is developed based on the partially irreversible, hysteretic sliding at splat boundaries that lead to the linking of pre-existing micro-cracks leading to the final propagation of a flaw of critical size.

#### Symposium 1: Aerospace Ceramics and Composites

Room: Montreal 1-3

Session Chair: Katherine Faber, California Institute of Technology

#### 3:30 PM

### (ICC-SYM1-036-2024) High-Temperature Slurry Environmental Barrier Coatings (Invited)

R. Webster<sup>\*1</sup>; K. Lee<sup>1</sup>; B. J. Harder<sup>1</sup>; B. Puleo<sup>1</sup>

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 are comprised of a rare earth silicate top coat and a silicon bond coat. The upper use temperature of these EBCs is limited by the melting point of silicon (1414°C). The replacement of silicon with an oxide bond coat should allow CMCs to reach higher temperatures, which can result in reduced emissions and improved performance. A mullitebased bond coat produced by slurry processing and capable of withstanding temperatures of up to 1482°C has been developed at NASA Glenn Research Center. Various EBC architectures with this bond coat have been developed and tested; different top coat compositions were evaluated to further maximize performance. Hafnia (HfO<sub>2</sub>) is an attractive EBC topcoat material due to its stability at high temperatures and in steam; however, it has a large and highly anisotropic coefficient of thermal expansion (CTE), making it difficult to achieve long lives in cyclic environments. In this work, a graded slurry HfO<sub>2</sub>-HfSiO<sub>4</sub> topcoat was deposited on the mullitebased bond coat. The cyclic oxidation behavior of this EBC system was evaluated at 1482°C in a 90%  $H_2O/O_2$  steam environment.

#### 4:00 PM

#### (ICC-SYM1-037-2024) Highly Reliable TBCs for Industrial Gas Turbine and AeroEngine Applications

Y. Okajima\*1; K. Takeno1; H. Komuro1; T. Torigoe1

1. Mitsubishi Heavy Industries, LTD., Research & Innovation Center, Japan

Mitsubishi Heavy Industries, Ltd. had developed the world's highest turbine inlet temperature, 1650°C-class JAC gas turbine in 2020. This gas turbine successfully achieves a higher turbine inlet temperature by further increasing the thickness of the highly reliable Thermal Barrier Coatings (TBCs) utilized for the 1600°C-Class J-Series gas turbine to improve the thermal barrier capability. This advanced TBCs has been released to the market after various component tests and verification tests in real operation in JAC gas turbine. JAC-Series gas turbine has more than 110 orders and exceeds 2,000,000 accumulated operation hours, which shows sound operation results. MHI is currently developing Suspension High Velocity Oxy-Fuel spraying (S-HVOF) process to apply for aeroengines which requires extreme reliability. TBCs by S-HVOF shows higher reliability than those by Air Plasma Spray (APS) though S-HVOF's is higher thermal conductivity than APS's. This process is promising to alternate to EB-PVD, which is a standard process for aeroengine TBCs.

#### 4:20 PM

#### (ICC-SYM1-038-2024) Development of multicomponent stabilized zirconia as ultrahigh temperature thermal barrier materials

S. Ramachandran\*1; G. Ashutosh Suresh1

1. IIT Bombay, Metallurgical Engineering and Materials Science, India

8 mol% yttria-stabilized zirconia (8YSZ) has been the conventionally used thermal barrier coating (TBC) material. It undergoes phase transformations during service, which limits its service temperature to less than ~1200 °C. Cubic zirconia ceramics are phase-stable across relevant temperature ranges and possess lower thermal conductivity than tetragonal YSZ. However, cubic YSZ coatings exhibit shorter thermal cyclic lives than tetragonal YSZ coatings, mainly due to the relatively low fracture toughness. In the present work, we have designed and synthesized new cubic zirconia compositions stabilized by 5 to 10 rare earth oxides. Powders were synthesized by the solution combustion synthesis method. X-ray diffraction (XRD) and Raman spectroscopy were used for structural analysis to confirm the cubic phase formation. Relevant property measurements of these newly developed materials require fully dense forms. Sintering studies were carried out from 1200 °C to 1700 °C, for up to 2h. Microstructural and compositional investigations were carried out by scanning and transmission electron microscopy. Thermal conductivity values ( $\kappa \sim 0.7$  W/m.K at 1000 °C) lower than conventional tetragonal YSZ were obtained. Coefficients of thermal expansion were measured and evaluated for their suitability for ultrahigh temperature applications.

#### 4:40 PM

#### (ICC-SYM1-039-2024) Protecting Carbon/Carbon Composites with an Ablation-Resistant and High-Emittance Coating System During Hypersonic Flight

R. Trice\*1; C. Martinez1; A. A. Saad1

1. Purdue University, Department of Materials Engineering, USA

Leading edges during high speed flight can experience high thermal loads, with surface temperatures greater than 1600C expected. Carbon/carbon (C/C) has been used successfully in past leading edge structures, but it is prone to oxidation and ablation damage above 50C. In this study, a novel multilayer coating system is developed to protect the C/C from the air while also maximizing radiative cooling. Individual sublayers consist of SiC,  $ZrB_2$ -SiC,  $ZrC-ZrO_2$ , and  $Sm_2O_3$ - $ZrO_2$  as a topcoat.  $Sm_2O_3$  is a rare-earth oxide and is doped in such a molar concentration that it acts as both an emissivity modifier

and a stabilizing agent for t'- ZrO<sub>2</sub>. The multilayer coating is applied to a C/C surface via pack cementation and plasma spray with variable sublayer thickness. The multilayer coating demonstrated excellent ablation and mass erosion resistance as it reduced the mass ablation rate of C/C by more than 90% while being subjected to an aggressive oxyacetylene torch heating (heat flux of approximately 390 W/cm<sup>2</sup>). Surface temperatures of 2100C were reached after 60s with no apparent oxidation of the C/C. During testing, the Sm<sub>2</sub>O<sub>3</sub> modified ZrO<sub>2</sub> topcoat acted as an insulative layer and demonstrated high spectral emittance (e.g. 0.99 at a12 micron wavelength), maximizing radiative cooling.

#### Symposium 5: Global Ceramics and Glass Industry Trends

### Symposium 5: Global Ceramics and Glass Industry Trends

Room: Montreal 7 Session Chair: Don Lipkin, Texas A&M University

#### 1:30 PM

(ICC-SYM5-007-2024) Advanced Ceramics and Coatings for Mining and Mineral Processing, Oil & Gas Production and Power Generation (Invited)

E. Medvedovski\*1

1. Consultant, Canada

The use of advanced ceramics, composites and coatings in mining and mineral processing, oil & gas production, refinery and power generation constantly grows owing to the development of advanced materials with improved properties and their technologies according to specific application requirements. Particular demands for advanced materials with high reliability or for coatings on complex shape steel components and long tubing with inner surface protection for a variety of applications require novel or optimized processing. The materials include oxide and non-oxide ceramics, ceramic-ceramic and ceramic-metal composites, coatings on metallic components where functional application properties can be achieved. Some principles of selection and manufacturing of these materials, specifically for wear-corrosion environments, are considered. The examples of successful development and processing of wear, corrosion and thermal shock resistant ceramics, composites (e.g., fine grained materials or materials with specially designed grain sizes and phase distribution) and coatings are discussed. The results of wear and corrosion resistance evaluation of monolithic ceramics and thermal diffusion-based boride and aluminide coatings on steels, depending on their structures, are presented. The technology importance on industrial components manufacturing is outlined.

#### 2:00 PM

### (ICC-SYM5-008-2024) Development of High-performance, TiC/Ti(C,N)-based Cermets and Coatings (Invited)

K. P. Plucknett<sup>\*1</sup>

1. Dalhousie University, Mechanical Engineering, Canada

Titanium carbide (TiC) and titanium carbonitride Ti(C,N) based ceramic-metal composites, or cermets, typically exhibit an excellent combination of high strength, hardness, and wear response. Through appropriate choice of metal binder, they also show good aqueous corrosion resistance. Recent work on TiC and Ti(C,N) cermets will be reviewed in the present talk, with a focus on ductile nickel aluminide (Ni<sub>3</sub>Al) and ferrous-based binders. It will be shown that significant opportunities for property modification and improvement exist in these cermet systems. Approaches include incorporation of graphene nanoplatelets or the application of post-sinter heat treatments, which can be applied to Ni<sub>3</sub>Al and select ferrous alloys. Emphasis will be placed on the wear and corrosion responses of these materials, where their properties are invariably shown to improve on current tungsten carbide 'hardmetals'. Extension of these studies to TiC-based coating systems will also be discussed, including the contrast between high velocity oxyfuel and laser directed energy deposition processes, as well as the application of surface diffusion transformation methods (i.e., boriding).

#### 2:30 PM

#### (ICC-SYM5-009-2024) Technical Focus and Accelerating Success Under New Management at TA&T (Invited)

T. Shoulders<sup>\*1</sup>

1. Technology Assessment and Transfer, USA

Technology Assessment and Transfer, Inc. (TA&T) has an over 40-year history as a performer on government funded research programs with a focus on ceramics. After a recent acquisition by SINTX Technologies, Inc., TA&T is strategically focusing R&D efforts as well as small-scale commercial work in 3 key technology areas, namely ceramic matrix composites, 3D printing of technical ceramics, and transparent ceramics. These technology areas present a unique opportunity to serve growing customer needs across the US DoD, DoE, and commercial sectors while maintaining a level of core competency in critical ceramic manufacturing techniques that are becoming rarer and rarer among industry peers. An example of active development from each of the three technology areas will be presented. These include manufacturing of ultra-high temperature CMCs with a hybrid PIP/CVI approach; development of a particulate loaded resin system for DLP 3D printing of silicon nitride; and manufacture of transparent ceramic scintillators for high-energy x-ray CT. In addition to brief technical overviews of each topic, each example will be tied to the company's strategic direction and will show how leveraging capabilities from the parent company with existing technologies is accelerating success for both entities.

#### 3:30 PM

# (ICC-SYM5-010-2024) Chemical tempering with $Ag^{\scriptscriptstyle +}$ and $Cu^{\scriptscriptstyle +}$ : for enhanced mechanical strenghtening and virucidal functionalization of glass surfaces

D. Kaur\*1; V. M. Sglavo2

- 1. University of Trento, Industrial Engineering, Italy
- 2. University of Trento, Italy

The development of antiviral surfaces remains an intriguing area within materials science, particularly in the realm of glass materials. Glasses find diverse applications ranging from windows and building facades to mobile touch screens, food ordering interfaces, laptop and TV screens. Since December 2020, amidst the spread of the coronavirus, safeguarding touch screen surfaces has become a significant focus of research. In this study, commercially available float glass, soda magnesia, and borosilicate glasses underwent chemical tempering with potassium nitrate at varying time intervals and temperatures. Additionally, a second ion exchange step with silver (Ag) and copper (Cu) ions was employed to augment their antiviral properties. The release of silver and copper, crucial for antibacterial and antiviral efficacy, was quantified via Inductive Couple Plasma Mass Spectroscopy on distilled water solutions. Thorough discussions are provided for the detailed methodology, as well as for the structural and morphological characterizations of the annealed copper and silver ion-exchanged samples. Various analytical techniques, including optical microscopy, scanning electron microscopy, and Raman spectroscopy, are utilized. SEM-EDs analyses confirmed the incorporation of potassium, silver, and copper into the glass surface.

### Abstracts

#### 3:50 PM

#### (ICC-SYM5-012-2024) Evaluation of Thermal Diffusion Coatings in 500-°C Molten Salt and Sodium for Nuclear Reactor Applications

Y. Xie\*1; E. Medvedovski2; L. Joyce1; D. Simonton1; E. Frishholz1

- 1. Purdue University, USA
- 2. Consultant, Canada

In nuclear power generation, structural materials, such as steels and alloys, face high-temperature corrosion in molten salt reactor and sodium-cooled fast reactor applications. This corrosion leads to degradation and structural failures of metallic components. To enhance their service life, protective coatings on steels are crucial. This study introduces thermal diffusion protective coatings, based on borides and aluminides. These coatings were tested and evaluated, for the first time, in molten Na-K-Mg-chlorides and liquid Na at 500 °C, simulating a reactor temperature. Structural and compositional analyses showed minimal changes and deterioration in these conditions. After a 30-day test, no significant corrosive scale formation, structural coating transformation, or protective layer reduction was observed. The coatings effectively prevented penetration of corrosive media to steel substrates. The encouraging results suggest the potential for further long-term and higher temperature testing, coupled with irradiation and forced hydraulic flow typical under pyrotopic reactor conditions.

#### 4:10 PM

# (ICC-SYM5-013-2024) From the 1st order phase transition in cristobalite to a compact high-temperature stable chemically stabilized ceramic

L. J. Ortmann $^{*1}$ 

1. QSIL GmbH, Research and Development, Germany

The use of fused silica T > 1300 °C is limited by the deformation and crystallization of the material. Now, the idea arose to develop a technology for the production of a material that combines the very good thermal shock resistance of fused silica with the good deformation resistance at high temperatures of cristobalite by making use of the mechanism of chemical stabilization of the high-temperature phase of cristobalite ( $\beta$ -cristobalite) down to room temperature. This work describes the production of a chemically stabilized ceramic (CSC) via the glass-ceramic route based on surface and bulk crystallization of glasses with high SiO<sub>2</sub> concentrations. Furthermore, the mechanisms of stabilization depending on the crystallization conditions are presented and the resulting effects on the thermal and mechanical properties of the generated material are discussed. The ternary system SiO<sub>2</sub> - Al<sub>2</sub>O<sub>3</sub> - CaO was selected for screening experiments, where the effect of the dopant ratios, the raw material preparation and the aging temperature were investigated with respect to the crystallization behavior as well as the stabilization mechanism of the  $\beta$ -cristobalite phase. Thermal and mechanical properties were discussed based on dilatometry and X-ray diffraction. Furthermore, a technological route to produce materials with a strongly suppressed phase transition is presented.

#### 4:30 PM

#### (ICC-SYM5-014-2024) Materials for Extreme Environments: Paving the Way for Lunar Exploration (Invited)

V. L. Wiesner\*1; G. King1; B. Widener2; C. Domack2; K. Gordon1; C. Wohl1

- 1. NASA Langley Research Center, Advanced Materials and Processing Branch, USA
- 2. Analytical Mechanics Associates, USA

As humanity expands its ventures into lunar exploration, the demand intensifies for materials capable of enduring extreme environmental and service conditions. The Moon's surface presents numerous challenges, including ultra-high vacuum exposure, temperature extremes and intense radiation. Additionally, lunar regolith threatens component durability and reusability due to its abrasive nature. Lunar dust particles scoring, adhering or embedding into surfaces and within device-confined geometries, such as bearings, drive mechanisms and connectors, can cause premature component wear or failure. Furthermore, the interaction between the rocket plume and surface during vehicle landings and ascents creates severe erosive conditions near critical vehicle components and adjacent infrastructure. A key barrier hindering materials discovery for lunar dust tolerant applications is a lack of standard laboratory methods to evaluate material properties and performance under representative operating conditions. A case will be presented for research activities supporting identification, processing and characterization of candidate materials in conjunction with innovations in standardized testing and qualification. Results will highlight current and emerging test methods for simulating lunar dust exposure and component degradation, paving the way for a new generation of robust materials and coatings for space exploration.

# Symposium 6: Informatics and data analytics in ceramics and glass

### Symposium 6: Informatics and data analytics in ceramics and glass

Room: Montreal 8

Session Chair: Stefano Curtarolo, Duke University

#### 1:30 PM

(ICC-SYM6-007-2024) Zentropy theory for accurate prediction of free energy without fitting parameters (Invited)

Z. Liu\*1

1. Pennsylvania State University, USA

The density function theory (DFT) is the de facto approach for predicting electronic structures and energetics of ground-state configurations, providing data for training of atomic force fields and machine learning models. However, predictions from DFT calculations and atomic simulations are generally not in quantitative agreement with experimental observations, particularly emergent behaviors as the system "embraces every conceivable combination of configuration and velocities" at finite temperature as stipulated by statistical mechanics. By expanding DFT to symmetry-broken non-ground-state configurations and replacing their total energies in statistical mechanics by their free energies, it is shown that the multiscale entropy approach, i.e., zentropy theory, can accurately predict free energy of a system and its emergent behaviors, including the singularity at critical point and negative thermal expansion free of models and model parameters. Furthermore, zentropy theory can predict entropy and free energy of non-equilibrium states of a system, including unstable states, which enabled us to revise the Onsager flux equations and develop a new theory of cross phenomena capable of predicting the coefficients of internal processes through derivatives of free energy. Their applications to magnetic, ferroelectric, and superconducting materials will be discussed in the presentation.

#### 2:00 PM

### (ICC-SYM6-008-2024) The coordination corrected enthalpies method for the thermodynamics of ionic materials (Invited)

#### R. Friedrich\*1

1. TU Dresden, Theoretical Chemistry, Germany

The computational design of ionic materials such as ceramics relies on accurate enthalpies. While standard electronic structure approaches based on density functional theory can provide quantitatively accurate results for intermetallic compounds, they fail to yield a proper description of the thermodynamics of ionic materials such as oxides with mean absolute errors for formation enthalpies on the order of several hundred meV/atom. This hinders the materials design of for instance high-entropy ceramics or lower dimensional systems such as 2D oxides. To address this pressing issue, we have recently developed the coordination corrected enthalpies (CCE) method based on the number of cation-anion bonds and the cation oxidation states. This correction scheme founded on the bonding topology decreases the prediction errors by almost an order of magnitude down to the room temperature thermal energy scale of ~25 meV/atom for oxides, halides, and nitrides. It is also capable of correcting the relative stability of crystal polymorphs. The efficient implementation of this scheme into the AFLOW framework for materials design in the form of the AFLOW-CCE module enables now the correction of enthalpies in large materials databases as well as for the construction of convex hull phase diagrams. These computational advances are thus an important enabler for the design of novel high-entropy ceramics.

#### 2:30 PM

#### (ICC-SYM6-009-2024) Thermodynamic Analysis of Boride and Carbide Ceramics: What We Know and Data Needs (Invited)

W. Fahrenholtz\*1; S. M. Smith1; G. Hilmas1; S. Curtarolo2

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

Macroscopic thermodynamic analysis is a powerful tool for understanding phase stability, reaction mechanisms, and oxidation of boride and carbide ceramics. This presentation will review two case studies of thermodynamic analysis of ultra-high temperature ceramics. The first is synthesis of zirconium carbide. This analysis revealed that the typical synthesis route of carbothermal reduction is constrained to producing zirconium oxycarbide. The second case study will focus on compositionally complex dual-phase ceramics consisting of a combination of boride and carbide phases with different compositions. The second analysis was able to predict metal segregation in dual phase ceramics that was consistent with experimental reports. The presentation will conclude by identifying gaps in available data that limit the range of analyses that can be completed.

#### 3:30 PM

### (ICC-SYM6-010-2024) Insights into mixing UHTC ceramics from DFT and CALPHAD

X. R. Tang<sup>1</sup>; G. B. Thompson<sup>2</sup>; C. R. Weinberger<sup>\*1</sup>

1. Colorado State University, Department of Mechanical Engineering, USA

2. University of Alabama, USA

There is significant interest in the development of new ceramic materials by mixing a large number of principle elements modeled after the development of high entropy alloys. However, there is a substantial gap in our understanding of what controls mixing from a thermodynamic perspective. In this talk, we use DFT and CALPHAD to examine what controls the mixing of the metal species in multiple principle component element UHTCs including transition metal carbides and diborides. We examine the role enthalpy, mixing entropy, and vibrational entropy play in controlling phase stability in these materials. We further explore how this relates to lattice constant differences, which are usually taken as a surrogate for mixing enthalpies in predicting the mixability of a solution. We further examine phase diagrams of predicted binary solutions and the temperatures required to mix complex solutions of these materials.

#### 3:50 PM

### (ICC-SYM6-011-2024) Understanding Interfacial Defects in High Entropy Ceramics (Invited)

D. W. Brenner\*1; S. Daigle1; M. Mou1; T. Haque1

1. North Carolina State University, USA

The multiple components present in high entropy ceramics provide both opportunities and challenges in understanding how effects such as configurational entropy, multiple atomic radii and different bonding preferences work together to determine defect energies and stresses. Using Density Functional Theory, we have been modeling grain boundaries together with stacking faults, twins and the gamma surfaces that connect them in transition metal high entropy carbides. We show that stable twin and stacking fault energies can be predicted from the respective binaries, while unstable stacking fault and twin energies can be predicted from these via an Evans-Polanyi-Semenov relation. This leads to analytic expressions for twinnability and dislocation dynamics. Grain boundary structures are more complicated, with energies depending on a convolution of different atom properties and grain boundary structures.

#### 4:20 PM

### (ICC-SYM6-012-2024) Quantifying Abnormal Grain Growth with Correlation Analyses and Information Theory (Invited)

J. Rickman<sup>\*1</sup>

1. Lehigh University, Department of Materials Science & Engineering, USA

In this talk I consider the ubiquitous phenomenon of abnormal grain growth (AGG), which involves the unusually rapid growth of a minority of constituent grains and often has a deleterious impact on the thermo-mechanical properties of a system. I will first describe the use of machine learning in the form of a canonical-correlation analysis (CCA) to connect AGG in ceramics with powder processing and, more specifically, the chemistry of specialty ceramics. I next examine the kinetics of evolving synthetic and experimental microstructures, as quantified by their embodied information, to identify temporal signatures of the onset of AGG. This is accomplished by tracking the information content of coarsening microstructures via selected metrics and measures of shared information and interaction strength. Finally, as the information content of a system is a proxy for the entropy, a thermodynamic description of grain growth is also outlined.

#### 4:50 PM

### (ICC-SYM6-013-2024) Qualifying Machine Learning Models for use in Data-Driven Materials Discovery and Manufacturing (Invited)

A. Stebner<sup>\*1</sup>

1. Georgia Institute of Technology, USA

Machine learning is transforming societies and is beginning to see wide adoption in many manufacturing sectors. This seminar will introduce successes and open challenges in application of machine learning to innovate materials and manufacturing technologies using case studies from my research career, as well as serving as the Deputy Editor for the journal Additive Manufacturing. Success will be demonstrated through recent results using machine learning together with pre-existing data to statistically inform the onboarding of a new laser powder bed fusion machine with a more powerful laser than the previous state of the art. Case studies will motivate a rubric for deciding when, how, and why to use machine learning in materials discovery and manufacturing, or more broadly any materials or manufacturing problem, including how to verify that the answer is reliable. This rubric also provides a basis for the "Data Foundations in Machine Learning for Engineers" course at Georgia Tech. Through this presentation, I will propose that it could be the basis for a new standard for documenting the use of machine learning models in data-driven qualifications of materials and manufacturing processes.

#### Symposium 9: Next Generation Bioceramics and Bioglasses

### Symposium 9: Next Generation Bioceramics and Bioglasses

Room: St. Laurent 3 Session Chair: Kalpana Katti, North Dakota State University

#### 1:30 PM

### (ICC-SYM9-001-2024) An industrial example: powder to 3D print production (Invited)

E. M. Valliant\*1

1. Himed, Research and Development, USA

There is no denying excitement for 3D printing of ceramics, including calcium phosphate bioceramics, especially for prototyping new designs and patient-specific medical devices. As this technology is still in the early stages, there are limited examples of the successful adoption of 3D printed calcium phosphate medical devices in industry. We present an example from a manufacturer's perspective embracing this technology covering the full manufacturing chain: from the production of the ceramic powder, to custom 3D printing ink development, to prototype production of hydroxyapatite (HA) medical devices for the customer. We worked with a leader in stereolithography 3D ceramics printing, Lithoz, to develop a custom 3D printing custom slurry with HA powder manufactured at Himed. Himed currently manufactures HA by 7 different methods, and a range of sizes. Key factors in the selection of the best powder will be discussed, including particle size, the distribution, surface area and density. Through iterations, both the powder and the slurry formulation were modified to create the optimum slurry. Slow and careful thermal treatment was required. Challenges faced when printing new prototype medical devices with our customers will be shared. We hope this case study will highlight some of the challenges that are faced when adopting this early-stage technology, from raw material to customer.

#### 2:00 PM

# (ICC-SYM9-002-2024) Study on mechanical and biological properties of 3D printed Fe<sub>3</sub>C nanoparticles blended bioactive glass scaffolds with different pore architectures

A. Vishwakarma<sup>1</sup>; A. Raichur<sup>1</sup>; N. Sinha<sup>\*1</sup>

1. Indian Institute of Technology Kanpur, Mechanical Ingineering, India

Tissue engineering for bone is always inpurtant when dealing with bone illnesses like osteosarcoma It domands the simultaneous destruction of malignant cells and the generation of bone for which magnetic field assisted by a remembra has received attention. In this investigation, we have prepared magnetic bioactive glass (MBG) by incorporating 044 i.km) arbide nanoparticles in the bioactive glass (45SiO<sub>2</sub>-20NAO-33CaO-6P<sub>2</sub>O<sub>5</sub>-2.5B<sub>2</sub>O<sub>3</sub>-1ZnO-2MgO-0.5CaF<sub>2</sub>) synthesized by col-gel method. Extrusion-based 3D printing was used to drease the scaffolds with grid, gyroid, and schwarz P architecture. The phases and functional groups present in MBG were identified using X-ray diffraction analysis (XRD) and Fourier transmission infrared spectroscopy (FTIR). Field scanning electron microscope was used to analyze the morphology of scaffolds. We have conducted mechanical, in vitro bioactivity, anti-bacterial and biofilm formation tests on these MBG scaffolds. The results indicated the influence of scaffold architecture on these properties. These studies help in determination of optimum pore architecture for bone tissue engineering.

#### 2:20 PM

#### (ICC-SYM9-004-2024) Pore Size Graded Bioceramic Scaffolds by 3D Printing Promote Angiogenesis and Osteogenesis

- Y. Wang<sup>1</sup>; J. Bai<sup>2</sup>; M. Wang<sup>\*1</sup>
- 1. The University of Hong Kong, Pokfulam Road, Hong Kong, Department of Mechanical Engineering, Hong Kong
- 2. Southern University of Science and Technology, China, Department of Mechanical and Energy Engineering, China

In human long bones, there is a pore size gradient with small pores in the exterior cortical bone and large pores in the interior cancellous bone. But most current bone tissue engineering (BTE) scaffolds only have homogeneous porous structures. Pore size graded (PSG) scaffolds are attractive for BTE since they can provide biomimicking structures that may lead to enhanced bone regeneration. In the current study, PSG scaffolds of biphasic calcium phosphate were designed and then fabricated via digital light processing 3D printing. The scaffolds were designed using gyroid of triply periodic minimal surface (TPMS), with small pores (400  $\mu$ m) in periphery and large pores (400, 600, 800 or 1000 µm) in the center of scaffolds (designated as 400-400, 400-600, 400-800 and 400-1000 scaffold, respectively). The porosity was the same for all scaffolds. Mechanical and fluidic properties of scaffolds were assessed. In vitro investigations showed that PSG scaffolds resulted in better osteogenic differentiation than scaffolds of uniform pores, with 400-800 scaffolds providing the best performance. In vivo investigations revealed that 400-800 and 400-1000 scaffolds could significantly improve neovascularization and new bone formation. This study highlights the importance of structural design and optimization of BTE scaffolds for achieving balanced mechanical, fluidic/transport and biological performance.

#### 2:40 PM

### (ICC-SYM9-005-2024) The impact of albumin on bioactive glass reactions in a dynamic in vitro environment

M. Siekkinen\*1; A. Stiller1; X. Wang1; L. Hupa1

Bioactive glass dissolves and develops a calcium phosphate layer in physiological-like solutions. The reactions in vitro are commonly studied in static protein-free solutions, e.g., simulated body fluid (SBF). Adding proteins to the solution increases the knowledge of the initial reactions. In this study, the bioactive glass particles (S53P4) were reacted for 3 days with flowing SBF, then the solution was changed to BSBF (SBF with bovine serum albumin, 1 mg/ml) and the tests continued for 3 days. Secondly, BSBF was used for 6 days. Lastly, reference samples were reacted for 6 days with SBF. Parallel static experiments were also conducted. Compared to the gradual pH increase of the static solutions, the pH increased rapidly in all solution outflows. At 24 h, the pH had stabilised above the reference pH. The phosphorus concentration in the outflows suggested delayed precipitation on the glass particles in contact with BSBF compared to SBF, which was also observed as less developed layers on the cross-sections at 72 h. The protein concentration in the flowing solution stayed initially close to the reference solution, indicating that the flow-through setup can be used with proteins without adsorption in the thin tubes. However, protein concentrations closer to the real in vivo values are needed in the future.

<sup>1.</sup> Åbo Akademi University, Finland

#### 3:30 PM

### (ICC-SYM9-006-2024) Structure analysis of CaO- $P_2O_5\text{-}TiO_2$ glasses synthesized with liquid phase method

S. Lee<sup>\*1</sup>; A. Obata<sup>2</sup>

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

Phosphate glasses with invert composition, which are mainly composed of ortho- and pyro-phosphate groups, can stimulate cellular functions by released inorganic ions. Our group has succeeded the synthesis of bioactive phosphate glasses with the liquid phase method at room temperature. The method can synthesis at room temperature and pressure. In this work, CaO-P<sub>2</sub>O<sub>5</sub>-TiO<sub>2</sub> glasses were prepared by liquid phase method, and their structures were analyzed. Raman spectra of the glasses showed the peaks corresponding to ortho- and pyro-phosphate, and titanium-containing compositions were showed additional peak of P-O-Ti bond. <sup>31</sup>P MAS-NMR spectra of the glasses showed peaks in the region of ortho- and pyro-phosphate, and the peaks with higher titaniumcontaining compositions were widen to metaphosphate region. However, the peaks corresponding to metaphosphate were not observed in the Raman spectra. Thus, the glasses contain chain-like structure (-O-P-O-P-O-Ti-O-)<sub>n</sub>, which was formed by pyrophosphate units bridged with  $TiO_{y}$  (denoted by  $Q_{p}^{-1}$ -Ti). The  $Q_{p}^{-1}$ -Ti chain structure is novel in phosphate glasses, and has not been reported in conventional melt-quench-derived glasses. In the case of low titanium-containing compositions, their structure was similar to that of phosphate invert glasses prepared by melt-quenching method.

#### 3:50 PM

# (ICC-SYM9-007-2024) Borophosphate glasses for soft-tissue regeneration: correlation between structure and dissolution properties

M. Abbasi\*1; A. Krishnamurthy2; S. Kroeker1

- 1. University of Manitoba, Department of Chemistry, Canada
- 2. Purity-IQ, Canada

Bioactive glasses are a class of biomaterials with diverse medical applications including soft- and hard-tissue regeneration. While silicate-based glasses have been commercialized for bone- and dental-related trauma, borate and borophosphate glasses are frontrunners for soft-tissue wound healing, as they dissolve on a timescale appropriate for delivering therapeutic ions to the injury site. Establishing a relationship between the borophosphate glass structure and dissolution behaviour is essential to enable researchers and engineers to design glasses with controllable degradation rates for various applications. We have characterized a series of sodium borophosphate glasses before and after standardized dissolution tests using <sup>31</sup>P and <sup>11</sup>B magic-angle spinning nuclear magnetic resonance (NMR) spectroscopy. The connectivity between boron and phosphorus cations was probed using rotational echo double resonance (REDOR) NMR spectroscopy. The NMR data are interpreted using Pauling bond strengths and charge-compensation arguments to conclude that and dominate in glasses rich in phosphorus, while boron-rich glasses comprise mainly and species. Solution <sup>31</sup>P NMR provides real-time information about the phosphate species released into solution, revealing that the balance of B - O - B and P - O - B linkages is a determining factor in the degradation rate of borophosphate glasses.

#### 4:10 PM

### (ICC-SYM9-009-2024) Do carbonate minerals accelerate wound healing? (Invited)

J. Barralet\*1

1. McGill University, Canada

Many inorganic compounds have been used to repair bone, and in some circumstances have been found to be osteoconductive and even osteoinductive. Usually, ions are introduced to wounds as ointments or creams in an attempt to reduce infection, for example zinc, silver, iodide and peroxide. Given their biological importance, carbon dioxide and its aqueous ions have been studied and is reported to increase fibroblast proliferation, and collagen synthesis. However, when delivered as a salt, such as calcium carbonate, it is very challenging to attribute an effect to solely the cation or anion, or a combination of both, or an indirect effect e.g. through modulation of inflammation, or interaction with a bioactive compound. Many prior preclinical studies on wound healing augmented with biomaterials has been performed in full thickness wounds in otherwise healthy animals, yet the clinical need for acceleration would seem to be in compromised tissue wounds. Here, we examined whether a commercial medical grade calcium carbonate was effective in accelerating healing in ischemic full thickness wounds in a preclinical model. We then explored whether healing was similar with other calcium carbonates, and then expanded the study to consider carbonates of different metals. Wound healing parameters were measured from histological sections and photographs and included scar tissue area, blood vessel density, hair follicle number and wound area.

#### Symposium 10: Optical and Magnetic Ceramics Materials and Devices

#### Symposium 10: Optical and Magnetic Ceramics Materials and Devices

Room: St. Laurent 4

Session Chairs: Robert Pullar, Ca' Foscari University of Venice; Victoria Blair, US Army Research Laboratory

#### 1:30 PM

#### (ICC-SYM10-007-2024) Tailoring Magnetic Properties in Superexchange-Coupled Nanocomposites: Insights from Monte Carlo Simulations and Spark Plasma Sintering (Invited)

P. Maltoni<sup>41</sup>; G. Barucca<sup>2</sup>; B. Rutkowski<sup>3</sup>; N. Yaacoub<sup>4</sup>; G. Varvaro<sup>5</sup>; J. A. De Toro<sup>6</sup>; M. Vasilakaki<sup>7</sup>; K. trohidou<sup>7</sup>; R. Mathieu<sup>8</sup>; D. Peddis<sup>1</sup>

- 1. University of Genoa, Chemistry and Industrial Chemistry, Italy
- 2. Universita Politecnica delle Marche, Italy
- 3. AGH University of Krakow, Poland
- 4. Le Mans University, France
- 5. CNR, Italy
- 6. IRICA, Spain
- 7. Demokritos, Greece
- 8. Uppsala University, Sweden

Permanent magnets (PM) are crucial in energy applications. Focus on sustainable materials has turned to ferrites, especially  $SrFe_{12}O_{19}$ . This study investigates strategies like cation substitution and superexchange-coupling to enhance ferrite magnetic properties. Through a scalable sol-gel self-combustion process, superexchange-coupled nanocomposites (SrFe<sub>12-x</sub>Al<sub>x</sub>O<sub>19</sub>/CoFe<sub>2-y</sub>Al<sub>y</sub>O<sub>4</sub> 90/10 wt%) were designed. Structural analysis via synchrotron X-ray powder diffraction and electron microscopy showcased controlled size, morphology, and composition of the composites; Monte Carlo simulations and experimental magnetic results aligned, revealing the possibility to tune the magnetic response of the systems. Spark plasma sintering (SPS) was used to fabricate dense bulk magnets (SPS\_NC and SPS\_NC\_SM), preserving the structural properties and enhancing alignment of particles along the easy-axes, to maximise the magnetic performance. Hysteresis loops, studied for randomly and parallel-oriented anisotropy axes, unravelled the intricate interplay of factors influencing magnetic properties. Importantly, the interface differences in NC and NC\_SM nanocomposites were explored, shedding light on their unique characteristics. This study provides insights into designing advanced permanent magnets for sustainable energy applications.

### Abstracts

#### 2:00 PM

### (ICC-SYM10-008-2024) Integration of NiCuZn ferrites into LTCC multilayer modules

- J. Topfer<sup>\*1</sup>; C. Priese<sup>1</sup>; A. Bochmann<sup>1</sup>; B. Capraro<sup>4</sup>; B. Mieller<sup>2</sup>; C. Fresemann<sup>3</sup>
- 1. Ernst-Abbe-Hochschule Jena, Germany
- 2. BAM Federal Institute for Materials Research and Testing, Germany
- 3. Technical University Berlin, Germany
- 4. Fraunhofer IKTS, Germany

Soft ferrites with adequate permeability profiles are required for the fabrication of multilayer ferrite inductors (MLFI) either as monolithic passive components or integrated inductors in complex LTCC (Low Temperature Ceramic Co-firing) modules. Ni-Cu-Zn ferrites are standard soft ferrites used for multilayer inductors at intermediate operating frequencies. We report on the effects of stoichiometry, temperature and oxygen partial pressure on the sintering behavior, microstructure and permeability of bulk and multilayer ferrites. Integration of ferrite layers into LTCC laminates requires matching of sintering and shrinkage behaviors and minimization of thermal expansion mismatch. We investigated cofiring of Ni-Cu-Zn ferrite tapes with low-K dielectric CT708 LTCC tapes. To reduce silver migration, cofiring was performed at reduced oxygen partial pressure. Cofiring of ferrite- and LTCC tapes was studied using ferrite powders with different particle size, and its effect on cofiring and inductor performance was examined. A data base representing the complete fabrication process of integrated multilayer inductors was established, including ferrite powder morphology, tape casting formulations, lamination and cofiring conditions, and inductor performance. The dataset is transferred into an ontology and complete digital description of the multilayer fabrication process is achieved.

#### 2:20 PM

### (ICC-SYM10-009-2024) Towards nano-based ceramic permanent magnets (Invited)

C. de Julian Fernandez<sup>\*1</sup>

1. Institute of Materials for Electronics and Magnetism - CNR, Italy

Hard ferrite-based magnets are the most produced type of permanent magnets (PMs) in the world and, after rare - earth PMs, the second market in terms of sales. The recent crisis of the rare earths, that gives rise to the criticality in the production of rare-earth PMs, and the strong request of PM for Green Transition technologies like wind generators and electric mobility have gave rise to an increasing interest in the improvement of the magnetic performances of the ceramic PMs. The main aim is to increase their energy product (>30 kJm-3), the remanence induction (>0,4 T) but also their greener production. In this presentation, I will illustrate different research lines covering these challenges considering the use of novel nanomaterials. I will show that nanoparticles exhibit better magnetic properties (mainly high coercive field) that conventional micrometric powders, reaching the theoretic limit of the performances. I will discuss also on alternative materials like doped M-type ferrites and hybrid ferrite-metal nanomaterials. Finally, I will illustrate different specific methods to produce ceramic PMs using nanomaterials. This research has been supported by the EIT-RM in the project INSPIRES (prop. 20090) and by the European Union - NextGenerationEU (National Sustainable Mobility Center CN00000023, Italian Ministry of University and Research Decree n. 1033 - 17/06/2022, Spoke 11 -Innovative Materials & Lightweighting).

#### 3:20 PM

#### (ICC-SYM10-010-2024) The HyPerMag Project: Novel Rare-Earth Element (REE) Free Permanent Magnets Based On Hexaferrite Nanocomposites (Invited)

R. C. Pullar\*1

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

At the moment there is a genuine need for high performance hard magnetic materials with large magnetisation, remanence and coercivity values which are not based on Rare-Earth Elements (REEs), especially with the ever-increasing use of electric motors and magnetic components in transport and sustainable energy generation (windmills, automotive, etc.). The HyPerMag project aims at realising a novel class of REE-free / critical element free permanent magnets based on hybrid nanocomposites, by coupling a soft and a hard phase at the nanoscale. This will utilise techniques such as exchange or dipolar coupling to achieve this ambitious result. This presentation will look at our initial work investigating the preparation of hard and soft nanometric hexaferrite phases with metal ion substitution for use in exchange coupled nanocomposites and core@ shell hard-soft hybrid nanostructures. Such hybrid powders will eventually be used to fabricate both bonded and dense permanent magnets, with improved energy product with respect to commercial products based on Alnico or ferrites.

#### 3:50 PM

# (ICC-SYM10-011-2024) Investing structural, magnetic and dielectric properties of ferrites using green synthesis approach (Invited)

R. Jotania\*1

1. Gujarat University, Department of Physics, Electronics and Space Science, India

Ferrites are ferrimagnetic ceramicsand exhibit good magnetic and electrical properties. Magnetic parameters like saturation magnetisation, remanent magnetisation and coercivity, as well as the dielectric permittivity, of ferrites can be tuned by divalent or trivalent metal ions substitution for various applications. Sustainable extracts of plants, roots, flowers, fruits and seeds containing different natural antioxidants that can work as both capping and reducing agents are being used for the green synthesis of ferrites. I would like to coverthestructural, magnetic and dielectric properties of M and X-type hexaferrites, spinel and hexaferrite/spinel composites  $(MgFe_2O_4, CaFe_{12}O_{19}, X-type Ba_2Zn_2Fe_{28}O_{46}, SrFe_{12}O_{19}/NiFe_2O_4)$ prepared using a green synthesis approach. The benefits of a green synthesis route regarding the structural, magnetic and dielectric properties of these materials will be discussed. The effects of various parameters such asheating temperature, weight ratios of extracts, and the types of leaves, roots and fruits used on various properties of theferrites will be also discussed.

#### 4:20 PM

#### (ICC-SYM10-012-2024) Green synthesis of Nd substituted M-type hexaferrites using moringa oleifera leaves for multifaceted applications

- A. A. Gor\*1; N. M. Devashrayee2; C. C. Chauhan2; R. Jotania3
- 1. Pandit Deendayal Energy University, Department of Physics, India
- 2. Nirma University, Institute of Technology, India
- 3. Gujarat University, Department of Physics, Electronics and Space Science, India

In this study we explore the structural, thermal, magnetic, surface morphology, Raman spectroscopic, and dielectric characteristics of Nd-substituted M-type  $Ba_{0.75}Sr_{0.25}Fe_{12-x}Nd_xO_{19}$  hexaferrites, aiming to elucidate their potential applications. Employing a green synthesis approach utilising Moringa Oleifera Leaves (MOL) extract, we synthesized a range of M-type hexaferrites with varying levels of Nd substitution ( $0.0 \le x \le 0.5$ ). The heat treatment method was

utilised for the green synthesis of the samples, with calcination done at 1100°C. Thorough characterisation encompassing structural, thermal(TGA), magnetic, Raman spectroscopic, and low-frequency dielectric analyses was carried out. X-ray diffraction (XRD) findings confirmed the formation of the M-phase, while EDAX and Raman spectroscopic analysis indicated the successful incorporation of Nd in lieu of Fe ions, with the substituted samples retaining their hexagonal shape, as evidenced by field emission scanning electron microscopy (FESEM) images. All samples exhibited a hard magnetic nature with coercivity doubling from 0.15 T (x=0.0) to 0.30 T (x=0.2). lowfrequency dielectric data provides insights into the influence of grain and grain boundaries. These results unveil promising prospects for the utilization of these hexaferrites across diverse applications.

#### 4:40 PM

### (ICC-SYM10-013-2024) Nonlinear optical properties of Barium Hexaferrite-PMMA polycrystalline thin film

A. A. Gor<sup>\*1</sup>; P. Baruah<sup>1</sup>; Y. Bulsara<sup>1</sup>; A. Lokhandwala<sup>3</sup>; N. M. Devashrayee<sup>2</sup>; C. C. Chauhan<sup>2</sup>

- 1. Pandit Deendayal Energy University, Department of Physics, India
- 2. Nirma University, Institute of Technology, India

3. Imperial College London, Department of Physics, United Kingdom

In this study, we explore the use of Barium hexaferrite(BaM) film fabricated on indium tin oxide (ITO) coated glass substrate (IG), with an additional buffer layer of poly(methyl methacrylate) (PMMA) for non-linear optical application. Barium hexaferrite film is sputtered on ITO coated glass substrate using RF Sputtering while the PMMA is spin coated on top. The study covers the characterisation of IG//BaM//PMMA, IG//BaM, IG/PMMA and IG respectively. XRD confirms the M type phase formation of BaM. FTIR confirms the presence of Fe-O and Metal - oxide bond vibration modes in BaM films. SEM images show the morphology of the films. The UV VIS spectra are used to find the optical band gap of the films. The non-linear optical characterisation is done using Z-Scan with a 100mW 532 nm DPSS laser. Our investigation aims to enhance optical nonlinearity that has been observed for potential applications in optical limiting devices and optoelectronic applications. The detailed results will be presented in the conference.

#### Symposium 12: Transportation and Infrastructure

#### Symposium 12: Transportation and Infrastructure

Room: Montreal 6 Session Chairs: John Provis, Paul Scherrer Institut PSI; Jeffrey Bullard, Texas A&M University

#### 1:30 PM

### (ICC-SYM12-001-2024) In Situ Resource Utilization Approach to the Deployment of Lunar Infrastructure (Invited)

- L. Valentini<sup>\*1</sup>; A. Driouich<sup>2</sup>; M. Favero<sup>1</sup>; M. Dalconi<sup>1</sup>
- 1. University of Padova, Geosciences, Italy
- 2. University of Padova, CISAS Centre for Space Research, Italy

With a revived interest in space exploration, space agencies have initiated programs to develop human settlements on the Moon. This step will represent a milestone towards a human mission to Mars. To achieve the goal of building lunar infrastructure and habitats, it is essential to meet the specific requirements for lunar construction, and to prioritize environmental and economic sustainability. Sustainable construction can be achieved by maximizing the amount of raw materials sourced on the Moon, according to the ISRU (In Situ Resource Utilization) approach. The aim of this research is to develop building components by alkali activation of lunar regolith. Optimized formulations of alkali-activated regolith were developed using a Design of Experiments (DoE) approach. This involved studying the effects of key parameters on the rheological and mechanical properties of the system. These included the type and amount of alkaline activator used, the addition of reactive powders, and the curing temperature. In order to enable the construction of lunar infrastructure and habitats, alkali-activated regolith must be designed to withstand the extreme conditions present on the Moon. One further critical aspect is related to the workability and the kinetics of setting and hardening, which must be compatible with the efficient extrusion of the material in the fresh state, through a 3D printing process.

#### 1:50 PM

### (ICC-SYM12-002-2024) In situ Quantification of $C_3A, C_2S,$ and $C_3S$ Dissolution Kinetics at the Nanoscale (Invited)

A. S. Brand\*1

1. Virginia Polytechnic Institute and State University, USA

Quantifying the dissolution kinetics of cementitious minerals has been identified as a significant research need, as these data have been demonstrated to be critical in understanding hydration mechanisms and developing cement hydration models. Using two quantitative phase microscopy techniques - digital holographic microscopy (DHM) and spectral modulation interferometry (SMI) - the dissolution kinetics of surfaces can be quantified in real time, in solution, and at the nanoscale. Both DHM and SMI capture surface topography data with submicron lateral resolution and nanoscale vertical precision, allowing for the calculation of dissolution flux by tracking surface topography changes. Data have been collected for tricalcium aluminate (C<sub>3</sub>A) and dicalcium silicate (C<sub>2</sub>S), and, more recently, tricalcium silicate (C<sub>3</sub>S) as well. These data indicate that the dissolution of cement minerals is spatially and temporally heterogeneous and that the dissolution flux is better represented by an extreme value distribution. Integrating complementary data from electron microscopy and X-ray photoelectron spectroscopy, the mechanism of dissolution is posited to occur through coupled interfacial dissolution-reprecipitation. While further study is required, these data are critical for understanding the mechanisms involved during hydration, particularly during Stage I within the first few minutes of reaction.

#### 2:10 PM

### (ICC-SYM12-003-2024) Electrochemical production of carbon-negative cement (Invited)

J. Li\*1

1. Lawrence Livermore National Laboratory/University of Michigan, USA

Cement production accounts for 8% of global CO<sub>2</sub> emissions. Emissions from manufacturing Portland cement are intractable as 60% of CO<sub>2</sub> emissions are attributed to the decomposition of limestone, the primary feedstock of cement manufacturing. We design a scalable decarbonization approach to eliminate the use of limestone in cement production by using carbon-free calcium silicate rocks or wastes. We couple water electrolysis to produce H+ and OH- that accelerate Ca leaching from silicates and captures air  $CO_2$  to form  $CaCO_3$ . When powered by renewable electricity, the electrochemically produced CaCO<sub>3</sub> can be fed to existing cement kilns to decarbonize by 60% in business-as-usual cement manufacturing without increasing cement production costs or capital investment. The electrolyzer generates value-added amorphous SiO<sub>2</sub> and green H<sub>2</sub>. This scalable air CO<sub>2</sub> capture, storage, and utilization technology can be used synergistically with other mature and emerging cement decarbonization strategies. Blending cement with our air-CO<sub>2</sub>-sequestrated CaCO<sub>3</sub> and electrochemically produced amorphous SiO<sub>2</sub> at 35% enhances the strength of cement while abating 75% of  $CO_2$  emissions. Using the produced green  $H_2$  to fuel cement kilns, the cement produced in this approach has potential to be carbon-negative with enhanced performance. This electrochemistry-based cement decarbonization approach using abundant ubiquitous silicate feedstock enables renewable energy and green H<sub>2</sub> at the gigaton scale.

### Abstracts

#### 2:30 PM

#### (ICC-SYM12-004-2024) Rheology and Microstructure Development of Hydrating Cement Systems for Additive Manufacturing (Invited)

S. Jones\*1; A. J. Allen2; J. Hipp3

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

Simultaneous rheology and small angle neutron scattering (SANS) measurements are made on hydrating cementitious systems to study the relationship between microstructure development and flow. The setting time of synthetic triclinic and monoclinic tricalcium silicate is shown to be related to the average total surface area of nano-scale calcium silicate hydrate, independent of polymorph or set retarders. Extending this approach, SANS measurements are conducted on Portland limestone (PLC) used in an additive manufacturing process. The flow properties of the PLC paste are characterized by yield stress, plastic viscosity, and a measure of yield stress recovery after shear stress removal. The Weissenberg and Deborah Numbers characterize the additive manufacturing process and correlate with a print quality measurement. SANS measurements show that high print qualities are related to the total surface area of nano-scale calcium silicate hydration, which produces Weissenberg numbers between two and five and Deborah numbers between 0.025 and 0.038.

#### 3:20 PM

# (ICC-SYM12-005-2024) Advantages of durable and reliable borosilicate glass for the transportation sector of today and tomorrow

J. Brandt-Slowik\*1

1. SCHOTT TGS, Germany

Windshield replacements are costly, especially as increasing levels of technology are integrated around the glass. Tough borosilicate glass as the outer glass pane especially helps commercial vehicles to run effectively and efficiently. Less downtime is beneficial for the logistic companies and reduces the negative impact on the environment, as during the lifetime less windshields need to be replaced. Borosilicate glass has a superior stone impact resistance in comparison to conventionally used soda lime glass, because in this special glass the load you need to apply to initiate a crack is much higher. In comparison to the standard windshield solution, the integration of borosilicate glass as the outer glass pane leads to a prolonged lifetime of the glazing. Scratch and sand abrasion resistance are much higher, which is especially beneficial for safety-relevant cameras and sensors behind the glazing. Furthermore, the driver benefits from less irritating light reflections as less defects cause less haze, which offers less distraction and therefore a better driving experience, especially when driving towards sunset or at night. The density of borosilicate glass is lower than one of soda lime glass, which enables low weight windshield solutions and helps to save money and to reduce the CO<sub>2</sub> footprint.

#### 3:40 PM

### (ICC-SYM12-006-2024) Ceramic construction and demolition waste as a supplementary cementitious material

T. Kronberg\*1; J. Eriksson1; T. Männistö1; L. Hupa1

1. Åbo Akademi University, Finland

Recycling ceramic construction and demolition waste (CCDW) is a way forward to a circular economy. Today, the utilization of CCDW is low, mainly due to the lack of systematic collection systems. Usually, much of typical whiteware ceramic waste is mixed with other demolition waste, e.g., concrete and bricks. The ceramic waste was collected, milled, and sieved to fractions less than 1 mm, 100  $\mu$ m, and 50  $\mu$ m. Mixtures of cement, sand, ceramic waste, and water were prepared. Cement was partially substituted with 5%, 10%, 15% and 20% of ceramic waste. The strength development

was determined after 7, 28, and 91 days. To verify the results and to exclude the impact of particle size distribution, inert quartz sand in similar fractions and amounts was also substituted for cement. The microstructure of the samples was examined by SEM/EDS. The reactivity of the ceramic waste depended on their particle size distribution and phase composition. The strength development in the samples with ceramic waste compared to cement was slower after 7 days but increased after that. The ceramic waste with a higher amorphous content, i.e., denser sintered products, showed a higher pozzolanic effect. Ceramic tile samples milled with the mortar remaining showed a slightly higher compression strength than the corresponding clean tile. The results showed that 15 - 25% of cement could be substituted by ceramic waste.

#### 4:00 PM

### (ICC-SYM12-007-2024) Investigation of joining techniques for advanced ceramics

J. Alexander\*1; J. Binner2

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

Fabrication of large / complex ceramic parts is challenging. The ability to easily bond multiple smaller parts could enable a cost-effective way to create these. As well as this, being able to join dissimilar materials enables the production of components with tailorable properties across them. Joining of ceramics is also a stepping stone in the repair process. This is of high interest since the fabrication of advanced ceramic materials is costly both financially and energetically, and therefore would enable the repair of monolithic and composite parts that fall out of spec. during manufacture, and the extension of service life of damaged components. However, challenges exist when trying to produce joints which yield mechanical properties sufficient for use in challenging environments. The current work explores some joining methods and assesses their physical and mechanical properties.

#### 4:20 PM

#### (ICC-SYM12-008-2024) The Effect of Acid Type on Phlogopite Dissolution in The Presence of Ethylenediamine

M. Akbarzadeh Khoei\*1; M. Alzeer1; J. Yliniemi1

1. University of Oulu, Fiber and particle Engineering, Finland

Phyllosilicate-type mineral wastes could contribute to the alkaline activation process by participating in the hydration and hardening reactions. These minerals contain relevant elements for cementitious reactions and carbon sequestration. In the broader context of sustainable construction, the use of phyllosilicates aligns with the industry's pursuit of eco-friendly alternatives to traditional Portland cement and aggregates. Investigating the dissolution of these minerals and trying to increase their reactivity is important research subject in material science. Phlogopite, a crystalline phyllosilicate, is explored to improve its dissolution, enabling its utilization as an alternative raw material for cementitious substances and carbonation. In this work, the effect of ethylenediamine on dissolution of phlogopite under acidic condition is studied. The effect of anionic part of the acid (HNO3 and HCl) on the extent of dissolution is compared. The composition of the solution after dissolution experiments was determined with ICP-MS, and remaining solid part was analysed by means of XPS, FTIR, and zeta potential. According to the results, proton-metal exchange reactions govern the overall dissolution, while the anionic part of the acid (Cl- and NO3-) also contributes to the dissolution. In addition, the presence of the ligand contributes to the efficiency of the HNO3 and increases the dissolution.

#### 4:40 PM (ICC-SYM12-009-2024) Toward fundamental characterization of pozzolanic reaction kinetics

J. W. Bullard<sup>\*1</sup>; Y. Han<sup>1</sup>; N. Van Dam<sup>1</sup>

1. Texas A&M University, USA

The rates and mechanisms of pozzolanic reactions in cement-based materials are critical for determining strength development, transport properties, and microstructure evolution when large volume fractions of supplementary cementitious materials are mixed with portland cement. The general course and effect of pozzolanic reactions is understood in broad terms. However, the phenomenon is quite complex, comprising multiple subreactions such as dissociation of silicates from the pozzolan, dissolution of portlandite, and the production or conversion of C-S-H with low Ca/Si ratios. The rate equation and mechanism of each of these processes have not been systematically investigated but are important for prediction and control of cement-based material properties. Here we take a step toward the goal of better understanding pozzolanic reactions by focusing on silica fume-a simple model material to represent a pozzolan-and portlandite to experimentally determine surface-normalized rate equations for their dissociation as a function of temperature and solution composition. These rate equations will be shown to provide evidence of likely rate-controlling steps in pozzolanic reactions. The application of these data to leaching and alkali-silica reactions will also be discussed.

#### 5:00 PM

### (ICC-SYM12-010-2024) Can we focus cements research to benefit both the environment and human development?

J. Provis<sup>\*1</sup>

1. Paul Scherrer Institut PSI, Switzerland

The very high environmental footprint of global cement production is well known and widely discussed. However, the availability of inexpensive, reliable and durable cement-based construction materials has underpinned the enormous advances in global development and human wellbeing that have been made during the past decades. So, we need to focus on improving how we produce and use cements and concretes, to provide the infrastructure needed for high quality and healthy life while also protecting the environment and ecosystems. Central to this discussion is the role of cements in generating wastes, but also in consuming wastes, and in safely treating wastes to remove potential contaminants from the biosphere. This presentation will touch on different aspects of cement materials science and engineering, spanning from fundamental materials characterisation to discussion of durability testing and efficient materials design, aiming to highlight the contributions (ongoing and potential) that cement science can bring to the global transition to sustainable development and environmental protection.

#### Thursday, July 18, 2024

#### **Plenary Session**

#### Plenary Session: Hala Zreiqat

Room: Montreal 1-3

8:30 AM

### (ICC-PLE-005-2024) Innovations in Nanostructured 3D-Printed Bioceramics for Personalized Bone Healing

H. Zreiqat\*1

1. University of Sydney, Biomedical Engineering, Australia

The growing clinical need for synthetics that specifically enhance the repair of critical large bone defects and aged bone matched by the escalating demand for grafts, is driven largely by an ageing population whose natural regenerative responses are impaired. This presentation will describe the following: 1) our strategies in developing a platform of patented engineered nanostructured, 3D-printed biomaterials for cell-free personalised treatment to promoting bone healing in load bearing challenging situations. 2) our unique fabrication strategies that will enable customisation of the implant's shape, size, structure and architecture to meet patient-specific requirements (Fig 1), 3) Identification of the composition of bioceramics that achieves antibacterial effects. Our technologies open avenues for skeletal and soft tissue regeneration in various clinical applications.

# Symposium 1: Aerospace Ceramics and Composites

#### Symposium 1: Aerospace Ceramics and Composites Room: Montreal 1-3

Session Chair: Rebekah Webster, NASA Glenn Research Center

#### 9:50 AM

(ICC-SYM1-040-2024) Materials development for thermal barrier coatings in advanced gas turbine

1. Tsinghua University, China

Materials with low thermal conductivity and higher mechanical toughness are crucial for the performance and durability of thermal barrier coatings in advanced gas turbine. We discover the thermal conductivity of rare earth niobates in fully dense ceramic could reach 1.2 W/mK at high temperature and the large chemical inhomogeneity lead to the high scattering rate of vibration states and diffusive thermal conductivity in them. We disperse platinum nanoparticles into a TBC material and form a metamaterial, in which the tunable surface plasmons can effectively shield NIR thermal radiation at high temperatures. In order to improve the mechanical properties, we develop high entropy ceramics, in which the ultra-high density dislocations lead to enhanced fracture toughness and a high thermal cycling lifetime.

#### 10:10 AM

#### (ICC-SYM1-041-2024) Investigating Performance of Novel High-Entropy Zirconate Thermal Barrier Coatings

H. Vakilifard\*<sup>1</sup>; R. Lima<sup>3</sup>; M. Pugh<sup>1</sup>; C. Moreau<sup>2</sup>

- 1. Concordia University, Mechanical, Industrial and Aerospace Engineering, Canada
- 2. Concordia University, Mechanical and Industrial Engineering, Canada
- 3. National Research Council of Canada, Canada

High-entropy ceramics represent an innovative approach to materials science, distinguished by novel compositions and unique properties. The novelty of high entropy ceramics lies in their

C. Wan\*1

variance from conventional materials, providing a platform for exploring new horizons in terms of materials design and engineering. High configurational entropy offers superior thermal stability, as well as mechanical and physical properties, making them promising candidates for applications in extreme environments such as high-temperature coatings, aerospace components, and power generation. In this research, the novel field of high-entropy ceramics is explored. By integrating insights from high-entropy ceramics, the authors aimed to improve the durability and performance of thermal barrier coatings (TBC) to meet the requirements of harsh operating conditions. TBCs play a critical role in extending the life and efficiency of components exposed to high temperatures in gas turbines. In this research, novel high-entropy zirconate (HEZ) ceramic with a chemical composition of  $(5RE_{1/5})_2Zr_2O_7$  was synthesized using the solid-state sintering method. Furthermore, the HEZ ceramic was deposited on superalloy substrates by suspension plasma spray (SPS) method, using the Axial III Mettech torch. Finally, TBC system performance and durability are investigated at high temperatures, which is critical to long-term performance.

#### Symposium 5: Global Ceramics and Glass Industry Trends

### Symposium 5: Global Ceramics and Glass Industry Trends

Room: Montreal 7 Session Chair: Kristin Breder, Saint Gobain Crystals

#### 9:50 AM

### (ICC-SYM5-015-2024) One view on the future of advanced glass, glass-ceramics and ceramic materials (Invited)

W. Cutler<sup>\*1</sup>

1. Corning Incorporated, USA

Glass, glass-ceramics, and ceramic materials have often held a position of the "materials of last resort." As the world changes, there are more applications where these materials have moved up the pecking order and are now the most desirable materials for the application. The author will provide examples of future growth areas for glass, glass-ceramics, and ceramic-based products across a variety of applications from displays and communication to clean energy and the environment.

#### 10:20 AM

### (ICC-SYM5-016-2024) Foundation of a start-up in the field of technical ceramics (Invited)

M. M. von Witzleben\*1

1. INMATEC Technology GmbH, MD, Germany

New ideas, innovative technologies often prevail just through start-ups. The example of INMATEC Technologies GmbH in Germany shows the experiences that have been made with the foundation and development of the company. The aim of the lecture is to let the audience participate in these experiences and to motivate ceramic researchers to do the same. With the support of young, brave and technology inspirated scientist, new and possibly disruptive ceramic technologies have a greater chance in a competitive world and can speed up the general technological development. First, the technological idea that forms the basis of the company to this day is presented, and then the findings from the founding of the company to the growing up of the company are reported.

#### 10:50 AM

### (ICC-SYM5-017-2024) Additive manufacturing of ceramic materials by direct selective laser sintering (Invited)

B. Cui\*<sup>1</sup>; X. Zhang<sup>1</sup>; L. Wadle<sup>1</sup>; F. Wang<sup>1</sup>; H. Dong<sup>1</sup>; Z. Wu<sup>1</sup>; Y. Lu<sup>1</sup>

1. University of Nebraska-Lincoln, USA

Direct selective laser sintering (SLS) process is a novel and promising approach for additive manufacturing of various ceramic materials such as BaTiO<sub>3</sub> and ZrC. Without polymer binders or resins, a dense ceramic layer could be formed with a thickness of several hundred micrometers in which pores or microcracks are absent. The SLS process may induce ultrafast densification, metastable phase transformation, and extraordinary chemistry at grain boundaries. For example, a Ti-rich secondary phase was distributed at the grain boundaries of the SLSed hexagonal BaTiO<sub>3</sub>. This talk will discuss the new phenomenon and fundamental questions related to lasermaterial interactions during SLS of advanced ceramics.

#### 11:20 AM

#### (ICC-SYM5-018-2024) Recent Advances in Electrolytic Plasma Technology for Ceramic Coatings on Light Alloys (Invited)

A. Yerokhin<sup>\*1</sup>; A. Rogov<sup>1</sup>; A. Matthews<sup>1</sup>

1. University of Manchester, United Kingdom

Current trends towards sustainable, resource- and energy efficient manufacturing bring ceramic coatings on lightweight substrates at the forefront of research interest. Plasma Electrolytic Oxidation (PEO) attracts significant attention as an advanced technology platform for development of high-performance protective and functional coatings on Al, Mg and Ti alloys. PEO affords uniform treatments of irregular surfaces that are inaccessible by conventional line-ofsight coating technologies thus offering a greater design freedom for lightweight structural components, functional devices and consumer products. A large number of variables that influence coating characteristics provide significant challenges for PEO process development and optimisation. To overcome these challenges we propose intelligent approaches that rely upon embedded quasi-autonomous digital control loops featuring appropriate diagnostic tools and digital twins. Recent progress in the development and application of experimental methods for active diagnostics of PEO processes reveals important insights into fundamental mechanisms underlying electrochemical behaviour of materials under transient conditions of high voltage pulse and pulse-reverse polarisation. Practical significance and implications of these findings for process energy efficiency and real-time control over coating characteristics and properties are discussed.

#### Symposium 9: Next Generation Bioceramics and Bioglasses

### Symposium 9: Next Generation Bioceramics and

#### Bioglasses

Room: St. Laurent 3 Session Chairs: Esther Valliant, Himed; Sungho Lee, National Institute of Advanced Industrial Science and Technology (AIST)

#### 9:50 AM

#### (ICC-SYM9-010-2024) 3D/4D Printing of Bioceramics and Biomedical Composites for Bone and Osteochondral Tissue Regeneration (Invited)

M. Wang $*^1$ 

1. The University of Hong Kong, Pokfulam Road, Hong Kong, Department of Mechanical Engineering, Hong Kong

Bioceramics play an important role in modern biomaterials development. They are commonly used for treating human hard tissue defects. On the other hand, bone-mimicking ceramic-polymer composites are developed to repair hard tissues, overcoming the brittleness problem of bioceramics. 3D printing is a powerful manufacturing platform for making porous scaffolds for regenerating different body tissues (J.Lai, M.Wang, Journal of Materials Research, 38 (2023), 4692-4725). Based on our substantial work in developing bioceramics and biomedical composites for bone substitution (M.Wang, Biomaterials, 24 (2003), 2133-2151), we have been investigating since 2003 3D printing (and later, 4D printing) and their applications in tissue engineering. Digital light projection was studied for fabricating BCP scaffolds for bone tissue engineering. Selective laser sintering was assessed for making nanocomposite scaffolds for regenerating bone. Cryogenic printing was developed for producing growth factor-encapsulated composite scaffolds for bone regeneration. Shape-morphing composite scaffolds were 4D printed for regenerating bone at sites with irregular defect shapes. Graded scaffolds were 3D printed for osteochondral tissue engineering. This talk will present some of our 3D/4D printing work in tissue engineering and also discuss relevant issues in materials and printing technology developments.

#### 10:20 AM

#### (ICC-SYM9-011-2024) Nanoclay Based Biomaterials for Development of Testbeds of Cancer Metastasis (Invited)

K. S. Katti<sup>\*1</sup>; D. R. Katti<sup>1</sup>

1. North Dakota State University, Civil Construction and Environmental Engineering, USA

Breast-cancer and Prostate-cancer are among the most prevalent cancers in women and men, respectively. The World-Health-Organization estimates that about a million deaths occur due to breast and prostate-cancer worldwide due to these cancers each year. Although often curable when detected early at the primary site, both of these cancers are incurable when the cancer metastasizes to a distant location in the body, which for these two cancers is eventually bone. There is a scarcity of available human samples and animal models fail due to death preceding bone metastasis; hence a huge unmet need for development of robust in vitro models of bone metastasis. We have developed novel biomaterials made by tuning the mechanical and biological properties of nanoclay-polymer nanocomposites using various amino acids. We have also used BMP modifications and a patented 'lego-block' design with hMSC seeding followed by seeding of prostate and breast-cancer cells from commercial and patient derived sources to generate tumors. The use of appropriate cocultures as well a specially designed horizontal flow bioreactor enables a true model of the metastasis. This testbed is used to develop novel spectroscopic and mechanobiological markers of metastasis. The testbed has a potential for use in screening drugs as well as provide personalized therapies

#### 10:50 AM

#### (ICC-SYM9-012-2024) Laser-Assisted Metal-Free Hermetic Sealing of Ceramic-based Bionic Devices using Glass/Cellulose Paste

S. N. Faisal\*1; S. Parsons1; G. Suaning1

1. The University of Sydney, Biomedical Engineering, Australia

Glass-ceramic bonding using thermal annealing is a conventional technique for sealing ceramic-based devices. However, this technique is not suitable to seal active implantable medical devices (AIMDs) due to the incompatibility of the electronics with the high temperatures. Laser-assisted welding is an alternative sealing technique which applies highly localised energy to form bonding, without impacting upon the electronics. For AIMDs, the sealing needs to be gas tight (hermetic) to ensure that both moisture and fluids are prevented from penetrating into a sealed electronic package. Hermetic sealing of AIMDs significantly influences the reliability and lifetime once implanted. Laser assisted hermetic sealing is often achieved by metal brazing which contributes to artifactual distortions when the implant recipient undertakes medical imaging procedures. It is therefore advantageous to achieve metal-free hermetic sealing. An alternative to metal brazing is the deployment of fritted glass powder. However, due to non-transparent properties of the ceramic, melting of typical fritted glass powders using a laser passed through the ceramic is challenging. Here, we propose a modified process using a fritted glass and cellulose paste as a soldering material which is cast onto the edges of ceramic-to-ceramic interfaces, and hermetically sealed using a short pulsewidth laser.

#### 11:10 AM

#### (ICC-SYM9-013-2024) Diamond Biomaterials for Next-Generation Implantable Devices: A Review of Properties, Applications, and Fabrication Methods

- K. Edalati\*1; S. Prawer1; D. J. Garrett2
- 1. University of Melbourne, Australia
- 2. RMIT University, Australia

Diamond's unique mechanical strength, chemical stability, and biocompatibility render it highly promising for next-generation implantable devices. This review discusses diamond's advantages for enhancing implants' durability, longevity, and performance. Critical dental, orthopedic, neural, and cardiovascular applications are explored. However, realizing diamond-based devices requires surmounting fabrication and integration challenges. Recent progress in utilizing biocompatible gold active braze alloys to join diamond components is highlighted. Reactive ion etching has emerged as an effective technique to eliminate harmful surface contaminants after brazing. Ongoing research aims to fully leverage Diamond's assets to provide safer, smarter, and more effective implants through innovations in materials engineering and bioelectronic technology.

#### \* Denotes Presenter

А
Abbasi, M.*
Abubakar, E
Adediran, A
Agrafiotis, C. C.*
Ahmad, Z.*
Aidhy, D.*
Akbarifakhrabadi, A.*
Akbarzadeh Khoei, M.*
Albonetti, S
Albunio, C.*
Alexander, J.*
Alizadeh, E.*
Alkanj, M. W
Allan, S. M.*
Allen, A. J
Almohammed, B.*
Alzeer, M
Amegadzie, M. Y 19
Anagri, A
Anand, R 15
Anasori, B.*
Antunes Leão, M 43
Apostolov, Z. D
Arugay, I. B
Ashrafi, B 15, 33
Ashutosh Suresh, G
Azami, N

#### B

Backman, D 33
Backman, L
Backman, L.*
Bae, C 28, 33
Bae, C.*
Bae, S
Baek, Y 32
Bai, J
Barba, D
Barralet, J.*
Barsoum, M14, 25, 42
Barsoum, M.*13
Baruah, P 57
Barucca, G 55
Basile, D
Beidaghi, M.*
Ben Miled, M 30
Benavente, R.*
Berger, T
Bernard, S
Bertrand, M 46
Besnard, C
Best, J
Bezek, L
Binner, J
Binner, J.*
Blacksher, C
Blacksher, C. H
Bochmann, A
Boissiere, J. D.*
Bordia, R 36, 48
Borgonia, J. P
Borrell, A
Bowman, R.*
Boyom Tatchemo, F 42
Braem, A
Brand, A. S.*
Brandt-Slowik, J.*
Brandt, O.*
Brenner, D. W.*
Brinkman, K.*

Brodersen, K
Browar, A
Buffy, J
Bulejak, W
Bullard, J. W.* 59
Bulsara, Y 57

#### С

#### D

D'Orazio, G.*
Dabera, D
Dai, J
Daigle, S 53
Dalconi, M
Dalslet, B. T
Dancer, C. E.*
David, A. M.*
de Julian Fernandez, C.*
De La Pierre, S.*
De Maddis, M 10
de Oliveira Lima, C. T
de Souza, F. L.*
De Toro, J. A
Delobel, F.*
deOliveira, L
Devashrayee, N. M 56, 57
Diaw, A.*
Diegeler, A.*
Diwald, O 14, 42
Dokyoung, E.*
Dollé, M
Domack, C 52
dondi, m
Dong, H 60

# Doucet, F. 46 Driouich, A. 57 Dubois, G. 25 Dudukovic, N. 25 Dutta, A.\* 32 Dylla-Spears, R. J.\* 25

#### E

Eckardt, D	
Edalati, K.*	
Edmonds, I. M 15	
El Gabaly Marquez, F 18	
Eltayeb, A	
Eriksson, J 58	
Escobar, G 28	

#### F

Faber, K	
Faber, K.*	
Fadaie, H	
Fahrenholtz, W.*	
Fahsbender, R.*	
Faisal, S. N.*	
Falkowski, P	
Fang, X.*	
Faral, M.*	
Favero, M	
Fears, T	
Feng, T.*	
Ferraris, M	
Firdosy, S	
Flauder, S.*	
Fotiadis, K	
Fradin, M.*	
França, C. A	
Franchin, G	
Fransson, M	
Fresemann, C	
Freudenberg, W.*	
Friedrich, R.*	
Frishholz, E	
Fu, Z	
Fuierer, P. A.*	
Fuji, M	
Fulanovic, L.*	

#### G

3
Gadea, C
Gaignon, R.*
Gao, B.*
Garrett, D. J
Gauspohl, J 20
Gauzere, L
Gelinas, A
Genest, M
Ghuman, K. K 17
Goins, P
Göksel, B.*
Golt, M
Gomwalk, N 45
Gor, A. A.*
Gorbar, M.* 14
Gordon, K
Gorman, B. P.*
Gorzkowski, E 27
Grabowski, G
Grabowy, M
Grasset, F.*
Green, C
Gregorova, E 40
Grimley, C 23

Guarini, G
Gubarevich, A 10
Gubarevich, A.*
Gubernat, A
Gump, C.*
Gusmão Santos, I. B 43
Gusmão Santos, I. B.*
Guziewski, M

#### Н

11
Ha, J
Ha, J.*
Habrioux, A
Hagino, Y
Han, Y 59
Hanzel, O 21
Haque, T
Harder, B. J 50
Hassig, M.*
Hata, Y.*
Hay, R.*
Hayden, J 22
He, P.*
Heintz, J.*
Hemmer, E.*
Herlin Boime, N 25
Hernández-García, A 46
Hičák, M
Hilmas, G 53
Нірр, Ј 58
Hirsch, S. G
Hobart, S
Hoesch, K.*
Holzer, L
Homa, J
Homma, G 12
Hu, L.*
Huang, K 30
Huang, Y 28
Hupa, L

#### Ι

Ionescu, E
Ipilakyaa, D. T
Ito, M
Iwazaki, Y.*

#### J

)
Jabile, L
Jabile, L. M
Jakubinek, M 15
Jang, S 33
Jayan, B.* 12
Jayaram, V.*
Jeong, B 41
Ji, W.*
Jonderian, A 17
Jones, G 23
Jones, S.*
Jotania, R
Jotania, R.*
Joyce, L

#### Κ

Kalra, V
Kamibe, T 13
Karagiannakis, G.* 29
Karamzadeh, V 15
Katoch, V.*
Katti, D. R
Katti, K. S.*
Kaufman, J 7
Kaufman, J.*

Kaur, D.*
Kaze, R
Kalley, K
Kellogg, F
Keshmiri, H
Kilo, M
Kim, D
Kim, H
Kim, J
Kim, K
Kim, S
Kim, T.*
King, G
King, J. A.*
Kishimoto, A 16
Klimczyk, P.*
Kluczowska, A
Koch, D
Komuro, H
Kondo, S
Kontic, R
Koo, J
Koos, E
Korycka, K
Kotani, M
Kotrbova, L
Koviciova, E
Koviniuz, 2
Kozien, D.*
Krishnamurthy, A
Krishnan, N.*
Kriven, W. M
Kroeker, S
Kronberg, T.*
Ku, A.*
Ku, N.*
Kulkarni, A.* 15
Kumagai, Y.* 11
Kumar, P
Kumar, R.*
Kunz, S. L.*
Kupferberg, A
Kweon, C
Kwon, O.*
,

#### L

E
Lacelle, T 15
LaGrost, C 25
Lal, D
Laminsi, S
Langhof, N
Large, M
Laskoski, M
Laszkiewicz-Lukasik, J
Latifi, M
Laventure, A
Lee, H
Lee, J
Lee, K
Lee, K.*
Lee, M
Lee, S.*
Lee, T
Lee, Y.*
Li, J
Li, J.*
Li, L
Li, Y
Liang, J
Lima, R
Limbaga, E. A
Lindblad, D
Lindner, F 16

Liu, L
Liu, M
Liu, M.*
Liu, Z.*
Lokhandwala, A
Lonchambon, P
Losego, M. D
Losego, M. D.*
Loughney, P.*
Lu, K.*15, 31
Lu, Y
Luo, J

#### Μ

Ma, H.*
Madrid Madrid, J. C.*
Maltoni, P.* 55
Manlupig, S. E
Manlupig, S. E.*
Männistö, T 58
Mao, X.*
Maria, J
Marmet, P
Marrero, N
Marrero, N.*
Martinez-Rubi, Y 15
Martinez, C 50
Masson, O
Masuda, H
Mathieu, R
Mathur, S 24
Matthews, A
Maxwell, J
Mazaheri, M.*
McCalla, E
Medvedovski, E
Medvedovski, E.*
Mendoza, G
Meunier, C
Michaud Paradis, M.*
Mieller, B
Mills, S. C
Minkiewicz, J
Mitrousis, V
Mock, C
Molina, A. M
Molinari, C
Molinari, C.*
Momot, K
Moreau, C
Moreau, C.*
Morita, K
Mota Garboggini Guerra, M. E
Mota Garboggini Guerra, M. E.*
Mou, M
Mourlas, A 12
Mrázek, J
Mullins, V.*

#### Ν

IN	
Nagano, K	
Nakayama, R	
Nambu, K	
Nauriyal, S.*	
Navrotsky, A.*	
Nečina, V.*	
Neige, E 14	
Nelson, T.*	
Nemani, S	
Neuhauser, K.*	
Ngo'o Ze, A	
-	

Ngono Mbenti Emvana, M 42
Nguyen, D. T
Nieroda, P
Nnakwo, K. C.*
Noel, J
Nohut, S
Nomura, K
Nordlander, J
Ntouala, R.*
Numkiatsakul, P.*
Nwambu, C. N.*

#### 0

Obata, A
Obradovic, N.*
Okajima, Y.*
Olisakwe, H. C.*
Onana, V
Opetubo, O
Ortmann, L. J.*
Özcan, L
Özmen, E
Özmen, E.*

#### Р

D 1 / 147 10	26 40 40
Pabst, W12,	
Pacheco Evangelista, R	
Pagkoura, C	
Pakravan, K	
Panda, D. K.*	
Pandey, O	
Panhard Lathoud, H.*	
Papakollu, K	24
Park, S	28, 33
Park, Y	
Parsons, S	61
Pasiut, K	
Patel, C. K.*	39
Patel, P. B.*	
Patterson, E	
Paulose, M	8
Peddis, D	55
Pedzich, Z	21, 38
Pedzich, Z.*	21, 37
Pein, M	11
Penner, D	14
Petit, C	12
Piechota, A. S.*	
Pirou, S.*	
Pitap Mbowou, M.*	
Plonska, M.*	
Plucknett, K. P.	19
Plucknett, K. P.*	
Podsiadlo, M	
Porcincula, D.	
Prawer, S.	61
Priese, C	56
Provis, J.*	59
Psyllaki, P.*	12
Puchas, G	16
Puchas, G.*	16
Pugh, M	
Puleo, B	
Pullar, R. C.*	

#### R

Raichur, A
Rajan, G.*
Ramachandran, S.* 50
Ramanadham, V 50
Razouq, H
Reddy, M
Resabal, V. T

64

Rickman, J.*53
Rifai, K
Ritucci, I
Rocha Palma, A. J
Rödel, J
Rodriguez, J
Roeb, M
Rogov, A
Root, H
Rosales, J 10
Rosenkranz, J
Rousselot, S
Rueschhoff, L
Rueschhoff, L.*
Ruggles-Wrenn, M.*
Ruiz, S
Rumiantseva, Y
Rutkowski, B
Ryou, H

#### S

8
Saad, A. A 50
Sadiq, Y. O 41
Safizadeh, F
Sakakibara, D.* 10
Salamon, D
Salvador, M
Sampath, S 50
Sanchez, B. H
Sanchez, E
Sant, G.*
Santillan, J.*
Sasan, K
Sato, Y.*
Sattler, C
Schaffer, L
Schafföner, S
Scherer, M
Schwenk, G. R
Schwentenwein, M10
Sciti, D
Sciti, D.*
Scott, K.*
Seager, T
Seredynska, B
Sglavo, V. M
Shahzad, A
Shakibania, S
Sharma, N.*
Shashoua, A
Shen, T
Shi, J
Shin, D
Shin, J
Shoulders, T.*
Shu, L
Siahpour, P
Siekkinen, M.*
Sieron, K
Sima, A
Simonova, P.*
Simonton, D
Singh, S.*
Sinha, N.*
Sitter, J. D.*
Skidmore, C
Smith, S. M
Sobhani, S
Sobhani, S.*
Sohrabi, A.         15           Soldi De Souza, G.         30
Somers, N
Son, H

Song, I
Song, X.*
Sponenberg, N. R 43
Spurling, R.*
Stebner, A.* 53
Stiller, A 54
Stotts, C 10
Suaning, G 61
Sugahara, Y.* 13
Suh, M
Sumi, H 39
Sun, T
Sunahara, I 39
Sundaram, S. K
Surble, S 25

#### Т

Tafu, M
Taiwo, O.*
Takeno, K 50
Takeuchi, T
Tan, X
Tang, X. R 53
Tanska, J
Tatarko, P 21
Taufour, V.*
Taylor, B 10
Taylor, O.*
Teranishi, T.* 16
Tessier, F
Thompson, G. B
Thompson, G. B.* 10, 21
Toher, C.*
Topfer, J
Topfer, J.*
Torigoe, T 50
Toyota, Y16
Treadwell, L
Trice, R.*
trohidou, K
Tseng, T.*

#### U

#### v

Vakilifard, H.*
Valasek, D
Valdez, A
Valdivieso, F
Valentini, L.*
Valenzuela-Heeger, E.*
Valliant, E. M.*
Van Dam, N
Van Meerbeek, B 25
Varathan, E
Vargas-Gonzalez, L
Varghese, O. K.*
Varvaro, G
Vasilakaki, M 55
Vellas, D
Vetrone, F
Vinci, A
Virtudazo, R. V
Virtudazo, R. V.*
Vishwakarma, A 54
Vleugels, J
von Witzleben, M. M.*60
Vuillaume, P

#### $\mathbf{W}$

Wadle, L	 
Wagner, L.*	 16

Waligo, D
Wall, R
Walter, A. D.*
Walton, R
Wan, C.*
Wang, F
Wang, J.*
Wang, M.*
Wang, X
Wang, Y
Wardle, B
Watkins, B. R
Weaver, J.*
Webster, R.*
Weinberger, C. R 10, 21
Weinberger, C. R.*
White, M.*
Wich, F
Widener, B
Wiecinska, P
Wiecinski, P
Wieclaw-Midor, A. M
Wieclaw-Midor, A. M.*
Wiesner, V. L

Wiesner, V. L.*
Wiggins, J
Wohl, C
Wojteczko, A
Wu, Y.*
Wu, Z
Wyatt, B

#### Х

Y
Yaacoub, N 55
Yadav, M.*
Yamaguchi, S.*
Yamaguchi, Y.*
Yamamoto, T
Yamanaka, R 16
Yang, Y 13
Yang, Z 28
Yasui, S.*16

Yavari, R 17
Yazdani Sarvestani, H
Yazdani Sarvestani, H.*
Ye, B 41
Yee, T
Yerokhin, A.*
Yliniemi, J
Yoo, Y
Yoshida, H.*
Yoshida, K 10, 12
Yucel, O.*

#### Ζ