

October 29-31, 2025 Mojmírovce, Slovak Republic

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Institute of Inorganic Chemistry, Slovak Academy of Sciences



Slovak Silicate Scientific-Technological Society



Slovak Academy of Sciences

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The  $15^{th}$  workshop on "Processing and Properties of Advanced Ceramics and Glasses" was held in the marvellous baroque-classical mansion Mojmírovce October 29-31, 2025. Due to the consistently high quality of the presentations the workshop was a great success.

The workshops included PhD students, post-doctorands and senior scientists and focused on the presentation and discussion of the newest and advanced trends in the development, processing, characterization and testing of new types of ceramics and glasses.

The topics focused on advanced ceramics and glasses for bio-medical, functional and structural applications including bioactive glasses, silicon carbide ceramics, ultra-high-temperature ceramic matrix composites, materials prepared by additive manufacturing, high entropy/compositionally complex bulk ceramics and coatings for functional and structural application.

The participants of many different nationalities, came from research centres in Slovakia and the Czech Republic.



This year the workshop was dedicated to Professor Pavol Šajgalík, one of the founders of the series of workshops on "Processing and Properties of Advanced Ceramics and Glasses". Prof. P. Šajgalík, with his organizational and scientific expertise, has greatly contributed to the increased level of reached scientific results in Slovakia today.

Dear Palo, on behalf of all your colleagues from the ceramics and glass community, we wish you all the very best for your 70th birthday.

prof. RNDr. Ján Dusza, DrSc.

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# ENHANCING THERMOELECTRIC PERFORMANCE OF STABILIZED HIGH-ENTROPY PEROVSKITE CERAMICS THROUGH Nb<sup>5+</sup> SUBSTITUTION

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### **ABSTRACT**

Novel stabilized high-entropy perovskite oxides, Sr<sub>0.25</sub>Ba<sub>0.25</sub>Ca<sub>0.25</sub>La<sub>0.25</sub>Ti<sub>1-x</sub>Nb<sub>x</sub>O<sub>3</sub> with varying Nb<sup>5+</sup> concentrations, were synthesised using the conventional solid-state route and sintered using field-assisted sintering technology at a temperature range from 1200 - 1350 °C. Novel compositions with a high configuration entropy were selected based on the theoretical predictions, considering tolerance factor, polarizability, electronegativity, configuration entropy, and global instability index GII -value (using "Structure prediction diagnostic software" SPUDS,). The XRD analysis confirmed the formation of a single-phase simple cubic perovskite structure, having a space group of Pm-3m. The SEM and EDX analysis confirmed the formation of dense microstructures with a relative density higher than 95 % and phase purity of the sintered samples. The FTIR analysis confirmed the formation of a single phase and metal oxides bonding in the structures. The UV-visible spectra measured in the diffuse reflectance mode indicated a gradual increase in the optical band gap value of the samples with increasing Nb<sup>5+</sup> concentration. The increase in the configuration entropy of the samples had a significant impact on the thermoelectric performance of the samples. The samples possessed a high negative Seebeck coefficient (S), confirming their n-type semiconducting behaviour. In addition, the samples exhibited a low thermal diffusivity < 1 mm<sup>2</sup>/s, a low thermal conductivity K < 3.8 Wm<sup>-1</sup>K<sup>-1</sup> and a high electrical conductivity of  $\sigma = 1.26$  S/cm for x = 0.05. These parameter favours thermoelectric performance, leading to an increased ZT value. The present work offers a compositional design approach for perovskite oxides to reduce their intrinsic thermal conductivity and enhance their thermoelectric performance.

### **ACKNOWLEDGEMENT**

This work was funded by the EU NextGenerationEU through the Recovery and Resilience Plan for Slovakia under the project No. 09I03-03-V03-00094/2024/VA.

## INSIGHTS INTO ELECTRONIC STRUCTURE AND THERMOELECTRIC BEHAVIOR OF HIGH-ENTROPY PEROVSKITES

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### **ABSTRACT**

The rising global energy demand calls for efficient and eco-friendly technologies to recover waste heat. Thermoelectric (TE) devices offer a promising solution, and high-entropy perovskites (HEPs) have recently attracted attention due to their structural stability, tunable electronic properties, and potential for high-temperature applications. In this work, we investigated the electronic structure and TE properties of HEP oxides derived from SrTiO<sub>3</sub>, doped with multiple A-site (Ca, Ba, La, Gd, Li, etc.) and B-site (Zr, Nb, Al, Hf, etc.) elements. First-principles density functional theory (DFT) calculations, combined with Boltzmann transport theory under the constant relaxation time approximation, were employed to explore the effect of multi-element doping. Complementary experiments were carried out on pristine and doped HEP oxides synthesized via mechanical alloying, solid-state reaction, and spark plasma sintering. Theoretical predictions were systematically compared with experimental transport measurements, providing insights into the structure–property relationships governing the TE performance of HEPs.

#### **ACKNOWLEDGMENT**

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### WASTE GLASS UPCYCLING IN FILAMENT PRODUCTION FOR ADDITIVE MANUFACTURING

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### **ABSTRACT**

Traditional glass forming relay on processing of homogenized glass melt within controlled viscosity working window, while the glass is still viscoelastic having viscosity around 10<sup>2</sup> - 10<sup>4</sup> Pa·s. The process is based on keeping temperature uniform, so the amount of glass is deformed predictably into desired shape without devitrification. For any recycling of glass, the glass scraps need to be sorted and remelted so that traditional shaping could be executed. Nevertheless, thanks to the glass ability to change its viscosity above glass transition temperature, the glass scraps can be joined also above this temperature under viscous flow conditions. This behavior is used as the strategy in shaping recycled glass using additive manufacturing technologies<sup>1,2</sup>. We have verified the use of Fused Filament Fabrication technology for this process using a filament developed at the Department of Inorganic Materials, with composition based on thermoplastic polymer binder and recycled glass powder with desired particle size distribution. Slicing and printing parameters adjustments were done using Repetier Host free-ware software and a commercial printer equipped with a 0.40 and/or 0.60 mm nozzle. After successful debinding and viscous flow sintering the specimens were investigated using Optical microscopy to achieve statistical evaluation of material road withs, connected to the reproducibility of material extrusion using 3D printing and future mechanical properties of the printouts.

### **ACKNOWLEDGMENT**

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## GLASS STRUCTURE FABRICATION BY FUSED DEPOSITION 3D PRINTING

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### **ABSTRACT**

In recent years it has been seen rapid growth in the area development of advanced materials shaping, particularly within the field of Additive Manufacturing (AM). Evident progress reported was in areas such as material design, optimization of printing accuracy, refinement of post-processing, exploration of hybrid manufacturing routes, and industrial applications studies. Nevertheless, the availability of new materials that have not yet been optimized for all AM technologies such as extrusion technologies remain a key challenge.

This study focuses on the development of composite materials for fused filament fabrication of glass (FFFG) and glass-based structures, with an emphasis on evaluating printability and identifying application-related limitations. Two types of recycled glass powders were investigated: PharmaGlass, derived from pharmaceutical vials, and Refaglass, a foamed glass thermal insulation material made entirely from recycled glass and used in various industrial applications. The experimental procedure was conducted in three phases. In the first phase, novel fiber compositions were prepared by incorporating inorganic particles into a two-component thermoplastic binder. The second phase involved optimizing printing parameters using standard FFF software and a conventional printer equipped with a 400  $\mu$ m nozzle. In the third phase, binder removal and sintering processes were carried out. The samples were debinded at a temperature of 600 °C and sintered at a temperature of 750°C in an elevator oven.

The results achieved, confirmed the potential use of recycled glass powders as feedstock materials for fused filament fabrication technology, requiring further research onto sustainable manufacturing of glass-based components. Further results show that three-dimensional glass structures were successfully prepared at temperatures 750 °C. Refaglass and Pharmaglass samples after sintering under different conditions weren't similar. There were differences in colour and homogeneity of the samples, even though they were sintered at the same temperature.

### ACKNOWLEDGMENT

The financial support of Slovak Grant Agency VEGA (grant No. VEGA 1/0070/22) and of the Slovak Research and Development Agency under the Contracts no. APVV-23-0352 and the support by the project Advancing University Capacity and Competence in Research, Development and Innovation ("ACCORD") ITMS2014+: 313021X329, co-financed by resources of European Regional Development Fund are greatly acknowledged.

# DLP-BASED FABRICATION OF 1393B3 BORATE BIOACTIVE GLASS SCAFFOLDS WITH COMPLEX GEOMETRIES AND EMBEDDED NANOPARTICLES

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### **ABSTRACT**

Research on additive manufacturing of bone regeneration scaffolds containing bioactive glasses (BGs) has mainly relied on extrusion-based methods to produce polymer/bioactive glass composites. These approaches avoid the high-temperature densification required for pure BG scaffolds, which typically leads to crystallization and alters bioactivity and ion-release behavior. However, extrusion-based composites possess limited structural resolution and reduced mechanical properties compared to pure BG or glass-ceramic scaffolds. Alternative studies using silicate BGs such as 45S5 and S53P4 have demonstrated glass-ceramic scaffolds with improved strength via Digital Light Processing (DLP) and stereolithography (SLA), but the associated sintering steps promote crystallisation that diminishes bioactivity.

In our previous work, we showed that 1393B3 borate BG can be densified at ~ 600 °C while remaining amorphous. This characteristic enables the additive manufacturing of pure BG scaffolds by vat photopolymerization techniques. Using a DLP 3D printer (Lithoz GmbH), we fabricated 1393B3 BG structures with diverse geometries, including gyroid, cancellous bone-mimicking, cortical bone-mimicking, dense cylinder, and plate designs, across clinically relevant size ranges. In addition, silicate-based therapeutic ion-doped and mesoporous BG nanoparticles were incorporated during the printing process to obtain scaffolds with embedded BG nanoparticles. Scanning electron microscope and X-ray diffraction analyses confirmed full densification while maintaining the amorphous nature. The scaffolds were subsequently characterized and showed innovative textural properties, improved mechanical performance and positive cytocompatibility compared to our previous scaffolds. In the future, in vivo compatibility tests are planned.

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### SLIP SYSTEMS IN ULTRA-HIGH TEMPERATURE CARBIDE GRAINS

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### **ABSTRACT**

The knowledge of active slip systems is essential in terms of the strength and plasticity of ultrahigh temperature carbide grains, which has been investigated in grains of (Hf-Ta-Zr-Nb)C highentropy carbide and monocarbides of TaC and HfC, using various experimental and computational methods. Nanoindentation and micropillar compression were performed to determine strength/hardness and slip activation in grains of different orientations. Critical resolved shear stress (crss) values corresponding to the {110}<1-10> and {111}<1-10> slip systems were calculated by first-principles density functional theory. Micropillar compression, combined with other techniques (SEM, EBSD, TEM), revealed that the crss is the lowest on the {110} glide plane with significantly enhanced value (~2.8 GPa) for (Hf-Ta-Zr-Nb)C compared to the component carbides (1.4-1.8 GPa). Still, there is a possibility to activate the {111} in specific cases. Simulated crss values correlate with experiments and make it possible to describe the experimental orientation-dependence hardness and slip activation using finite element simulations. Thus, nanoindentation is suggested to be a quick and efficient tool to assess the grain strength and to search for the activation of {111}<1-10> systems which endow rock salt carbides with improved plasticity.

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### LUMINESCENCE RESPONSE OF DEFECT FLUORITE PHASES IN MULTICOMPONENT RE<sub>2</sub>Zr<sub>2</sub>O<sub>7</sub> CERAMICS

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### **ABSTRACT**

Rare-earth zirconates (RE<sub>2</sub>Zr<sub>2</sub>O<sub>7</sub>) attract considerable interest for luminescent technologies owing to their high thermal stability, radiation resistance, and versatility in accommodating RE<sup>3+</sup> activator ions. Depending on the composition, these ceramics crystallise either in a pyrochlore or a defect fluorite lattice, with the latter characterised by significant intrinsic structural disorder.

In the present work, RE<sub>2</sub>Zr<sub>2</sub>O<sub>7</sub> ceramics were prepared via a combustion synthesis route, focusing on multicomponent compositions where several rare-earth elements occupy the same crystallographic site. This high-entropy strategy was employed to investigate how increasing configurational complexity influences the photoluminescence behaviour of the defect fluorite phase.

The results reveal that, contrary to expectations for a disordered matrix, the incorporation of multiple rare-earth cations enhances luminescence intensity. With increasing entropy, the disordered fluorite lattice locally stabilises more favourable environments for activator ions, which mitigates the detrimental effects of non-radiative relaxation. Consequently, multicomponent defect fluorites exhibit improved emission performance, highlighting the potential of entropy engineering to optimise optical properties in complex oxide ceramics.

### **ACKNOWLEDGMENT**

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### ADDITIVE MANUFACTURING OF POROUS PHOTOCATALYTIC GLASS-CERAMIC MEMBRANES FOR WASTEWATER TREATMENT

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### **ABSTRACT**

The amount of pharmaceutical glass waste generated during COVID-19 raises concerns about the environmental impact and exposes the shortcomings of current hospital waste management practices. Due to high energy and time requirements, metallic contamination and compliance with safety and hazardous waste protocols, the non-recyclable glass waste ends up in landfills and leads to ecological stress [1, 2]. This research repurposes borosilicate waste (Grade 1, hydrolytic class) into porous photocatalytic glass ceramic membranes for water treatment, using additive manufacturing. Photocatalytic components (TiO<sub>2</sub>, Fe<sub>2</sub>O<sub>3</sub>) are embedded into the glass matrix via low molarity (3 M) alkali activation, followed by spray drying of the suspension and flame synthesis to optimise the precursor porous glass microspheres (~35 µm) used in 3D printing to improve flowability and mechanical strength. A slurry for digital light processing (DLP) was prepared by mixing glass microspheres (< 35 µm) and resin in a 60:40 weight ratio, which was then used to print gyroid structures. For direct ink writing (DIW), paste inks were prepared by mixing glass microspheres, waste glass powder (< 30 µm), polyethylene glycol (PEG), and water. Various 3D-printed structures were fabricated and subjected to thermal analyses (TG-DTA and high-temperature observatory furnace) to study their sintering behavior using a three-stage heat treatment process. The sintered samples were then analyzed using XRD and SEM-EDS to investigate their amorphous and crystalline phases. In further steps, these designs will be used to study the degradation of dye or pollutant by photocatalytic activity under UV-Visible light. Thus, through this multidisciplinary approach, we aim to promote sustainable waste management practices and develop advanced water purification process.

### **ACKNOWLEDGMENT**

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### DEFORMATION AND FRACTURE OF DUAL – PHASE HIGH ENTROPY CERAMICS AT NANO/MICRO SCALE

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### **ABSTRACT**

The deformation and fracture characteristics of recently developed dual phase high entropy boride/carbide ceramics were investigated applying nano-indentation and micro-cantilever bending tests. Advanced processing routes have been applied for the preparation of the investigated systems, as boro/carbothermal reduction and reactive sintering.

The microstructure and fracture characteristics were investigated using X-ray diffraction (XRD), scanning electron microscopy (SEM) in combination with electron back scattered diffraction (EBSD) and transmission electron microscopy (TEM). Atomic structure and local chemical disorder were determined by means of scanning transmission electron microscopy (STEM) in conjunction with energy dispersive X-ray spectroscopy (EDS). Depth-sensing nanoindentation of individual grains and grain boundaries has been applied to study the nanohardness and deformation characteristics on the non-deformed and worn surfaces of the composites prepared by different processing routes.

During micro-cantilever tests in bending deformation and fracture characteristics of individual grains and grain boundaries have been investigated. The bending strength of micro-cantilevers was strongly dependent on the character/size of the fracture origins mainly in the form of grain boundaries.

#### **ACKNOWLEDGMENT**

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### LASER ABLATION ICP-MS AS AN ANALYTICAL TOOL FOR GLASS TECHNOLOGY

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### **ABSTRACT**

Laser Ablation Inductively Coupled Plasma Mass Spectrometry (LA-ICP-MS) has emerged as a powerful analytical technique in material science, offering high spatial resolution and trace element sensitivity. This method enables direct solid sampling, minimizing contamination and sample preparation time, while providing quantitative and qualitative insights into elemental distributions. We explore the application of LA-ICP-MS in characterizing advanced glass materials of various compositions. Case studies highlight its role in quality control, inhomogeneity identification, the trace element determination. The integration of LA-ICP-MS into material science supports our understanding of material quality and contributes to the understanding, how the chemical composition influences the glass properties with tailored functionalities.

### **ACKNOWLEDGMENT**

This work was created in the frame of the project APVV 22-0062.

### OXYACETYLENE TORCH RESISTANCE AND THERMAL CONDUCTIVITY OF ADDITIVE-FREE SILICON CARBIDE CERAMICS

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### **ABSTRACT**

Fully dense (t. d. > 99 %) silicon carbide ceramics without any sintering additives were successfully prepared by combination of freeze granulation of silicon carbide powder, annealing of granulated powder and subsequent rapid hot pressing at 1900°C with dwell time from 5 to 80 minutes under a uniaxial pressure of 70 MPa in vacuum. Different ratio of alpha and beta silicon carbide has been achieved by adjusting dwell time during sintering. Thermal diffusivity, specific heat capacity, thermal conductivity, density and microstructure of additivefree SiC with various  $\alpha/\beta$  content has been investigated. The thermal diffusivity of additive-free SiC ceramics increased from 47.3 to 68.4 mm<sup>2</sup>/s with increasing holding time at sintering temperature and therefore increasing α-SiC content from 63 to 94 % as well as increasing grain size. The highest thermal conductivity ( $\lambda$ =165 W/m.K) among the SiC ceramics sintered at temperatures ≤ 1900°C was achieved for additive-free SiC sintered at 1900°C under vacuum for 80 minutes. To the best of our knowledge, this is the highest value of thermal conductivity for silicon carbide ceramics sintered at T ≤ 1900°C up to now. In addition, such prepared materials were tested in oxy-acetylene torch at 1700°C, 1800°C and 1900°C for 5 minutes and even at higher temperatures for shorter time. In the dynamic regime the additive-free SiC ceramics survived 1900 °C for 5 minutes. It seems that this material could be suitable for ultrahigh-temperature applications.

### **ACKNOWLEDGMENT**

Financial support for this work through the grant APVV-24-0442 is gratefully acknowledged.

## PROCESSING STRATEGIES FOR DENSE HIGH-ENTROPY A<sub>3</sub>B<sub>2</sub>C<sub>3</sub>O<sub>12</sub> GARNET CERAMICS

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### **ABSTRACT**

A high-entropy structure can enhance photoluminescent properties of ceramics when an appropriate combination of elements is employed, justifying the development of such materials for advanced optical applications. However, for their further use, it is essential to study and describe their sintering behaviour to ensure high density and to eliminate the occurrence of any secondary phases, which can be a non-trivial task in such complex systems.

The present study focuses on optimising the processing strategy for  $RE_3Al_3Sc_1Ga_1O_{12}$ : $Er^{3+}$  garnet compound (RE = Lu, Yb, Y, Gd, La). In the initial phase, conventional reactive sintering from a mixture of oxides with the addition of common sintering aids for garnets (MgO and/or  $SiO_2$ ) was performed. However, the reaction between the starting oxides and the sintering aids resulted in forming a stable secondary oxide phase, which hindered the formation of a single-phase garnet composition, as confirmed by SEM-EDX analysis.

To overcome this limitation, an alternative route based on sintering pre-synthesised single-phase garnet powder was proposed. The high-entropy composition was obtained via combustion, followed by calcination at 1000–1600 °C, dispersion in suspension with selected sintering aids and homogenisation on rollers. After sintering, samples were characterised by XRD and SEM-EDX. The influence of sintering aids on the density and microstructure was evaluated.

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### DYNAMIC OXIDATION OF SELECTED NON-OXIDE CERAMICS

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#### **ABSTRACT**

Dense  $Si_3N_4$  substrates (98.2 % TD, with the addition of  $Y_2O_3$ ) and fully dense SiC without the addition of any sintering oxide particulates were prepared by FAST. Yttria as a sintering additive was chosen because of the good oxidation resistance of the composite at higher temperatures. SiC ceramics were prepared without any sintering additives in order to slower oxygen diffusion on grain boundaries. The surface of the ceramic substrate was oxidized by oxy-acetylene torch by means of the formation of a protective surface layer. The surface morphology and the character of the damaged area was studied in dependence from the reached temperature on the surface of the substrate, the dwell time on the annealing temperature and gas flow rate. Finally, the influence of the mentioned parameters on the degree of oxidation of the surface layer, its porosity, thickness and phase composition will be evaluated.

#### **ACKNOWLEDGMENT**

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### ISOVALENT DOPING IN NaSICON STRUCTURE TO UNDERSTAND THE IONIC CONDUCTIVITY

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### **ABSTRACT**

The isovalent substitution at the octahedral site of the NaSICON structure is an appropriate way to study its ionic conductivity, which is dictated by the size of the "bottleneck," according to Hong's theory [1]. This approach was effectively adopted in a recent study [2] to investigate

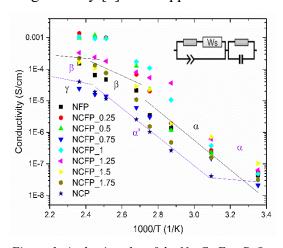


Figure 1: Arrhenius plot of the  $Na_3Cr_xFe_{2-x}P_3O_{12}$  composition

the effect of Al-doping in the Na<sub>3</sub>Fe<sub>2</sub>P<sub>3</sub>O<sub>12</sub> composition, avoiding the side effect of changes in cationic density due to charge compensation. This allowed the authors to isolate and highlight the well-known beneficial impact of Al-doping in solid electrolytes. Here, the same approach is applied to examine the effect of Cr-doping on the ionic conductivity of the NaSICON composition Na<sub>3</sub>Cr<sub>x</sub>Fe<sub>2-x</sub>P<sub>3</sub>O<sub>12</sub>. The Arrhenius plot reported in Figure 1 clearly shows a definite shift of the trend on the logarithmic demonstrating the change in ionic conductivity across the full range of compositions (x = 0, ..., 2). The conductivity initially increases at x = 0.25, then decreases at x = 0.75, before recovering at

x=1. At x=1.5, the structure undergoes a crystalline transition from the  $\alpha$  phase to the  $\alpha'$  phase, characteristic of the Na<sub>3</sub>Cr<sub>2</sub>P<sub>3</sub>O<sub>12</sub> composition (x=2). This behavior is similar to that observed for Al-doping of the same structure, with the difference that beyond x=1.25, the Aldoped structure transforms into a cubic phase typical of Na<sub>3</sub>Al<sub>2</sub>P<sub>3</sub>O<sub>12</sub>, leading to a pronounced decline in ionic conductivity. Density Functional Theory (DFT) calculations for the Na<sub>3</sub>Cr<sub>x</sub>Fe<sub>2-x</sub>P<sub>3</sub>O<sub>12</sub> compositions confirm both the increase in bottleneck size for the compositions showing the highest ionic conductivity, and the phase transition at x=1.5. Thus, this study demonstrates that isovalent substitution of iron with either Al or Cr is beneficial for the NaSICON structure.

### **ACKNOWLEDGMENT**

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# LASER ABLATION IN CHEMICAL CHARACTERIZATION OF GLASS: PRINCIPLES, STANDARDS AND APPLICATIONS

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### **ABSTRACT**

Laser ablation is a method for preparing samples for chemical analysis of glass and mapping of its composition. Using short, high-energy laser pulses, the material is vaporized and transferred for analysis — typically by mass spectroscopy using plasma as ionization source (ICP-MS) or related methods — without the need for complex sample preparation, which minimizes contamination.

Reference materials and standardized calibration procedures are essential to ensure quantitative accuracy, reproducibility, and comparability of results. This combination of principles and standards makes laser ablation a versatile tool for research, quality control, and industrial or archaeological applications.

In this contribution, we will present an example of experimental work focused on the spatial mapping of glass composition as well as the preparation of the internal calibration standards, currently in a development process. A series of model glasses with a silicate matrix SiO<sub>2</sub>–Na<sub>2</sub>O–CaO–Al<sub>2</sub>O<sub>3</sub>–K<sub>2</sub>O–Li<sub>2</sub>O were prepared. The main composition was based on the NIST standard 61X series (NIST SRM – National Institute of Standards and Technology Standard Reference Materials). The Li<sub>2</sub>O content was incrementally increased up to 11 wt.%. To extend the applicability of the standard to the analysis of impurities, the glasses may additionally contain 0.05 wt.% or 0.1 wt.% of selected oxides.

#### ACKNOWLEDGMENT

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# CONTROL OF STOICHIOMETRY AND MECHANICAL PROPETIES OF DC MAGNETRON CO-SPUTTERED HfZrTiTanb-C<sub>x</sub> COATINGS

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### **ABSTRACT**

High-entropy ceramics (HEC), as a part of compositionally complex ceramics (CCC), combine high hardness, thermal stability, and chemical resistance [1, 2]. The greatest interest is in multicomponent transition metal carbides and borides, which unite these properties and offer broad application potential - primarily as protective coatings for extreme environments [3, 4]. This work focuses on the effect of the carbon fraction in carbide coatings based on five transition metals (Hf, Zr, Ti, Ta, Nb) on their structural-phase composition and mechanical properties (hardness and elastic modulus). The coatings were deposited by DC magnetron cosputtering: both carbon and the transition metals (TM) were simultaneously sputtered from multiple independent targets. This approach enabled precise control of the chemical composition and phase formation.

The stoichiometry in the system between carbon and 5TM was controlled by varying the power on the carbon target from 200 to 600 W (in 100 W increments) with a constant power on the HfZrTiTaNb composite target. A comprehensive structural-phase analysis of the obtained coatings was carried out using electron microscopy (SEM, TEM), X-ray diffraction (XRD), and spectroscopic methods (EDS, Raman). Their mechanical properties and tribological characteristics were also investigated. In addition, nanoindentation was applied to determine the nanohardness and elastic modulus. Increasing the power on the carbon target from 200 to 600 W led to an increase in the hardness (H) from 19,5 to 26,9 GPa and contributed to the formation of a more stable carbide phase, which was reflected in an increase in both hardness and elastic modulus.

### **ACKNOWLEDGMENT**

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### DEVELOPMENT OF ULTRA-HIGH TEMPERATURE MULTICOMPONENT CERAMICS: FROM MONOCARBIDES TO TERNARY CARBIDES

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### **ABSTRACT**

Ultra-high temperature ceramics are a limited and select set of carbides, nitrides and borides of the group IV and V transition metals, which are typically defined as having melting temperatures over 3300 K, high hardness, elastic modulus and resistance to thermal shock and chemical attack. For technologies such as high temperature nuclear reactors, jet engines and hypersonic vehicles they therefore represent the only suitable class of materials available to make or protect components that are placed under the most extreme of operating environments [1]. This work is focused on development of multicomponent carbides, namely 15 binary carbides, 2 ternary carbides (ZrHfTa)C, (HfNbTa)C, and high entropy carbide (TiZrHfNbTa)C. However 6 monocarbides were also sintered to compare the microstructure evolution and basic mechanical properties. In summary, we successfully fabricated dense single phase multicomponent ceramics with density above 95 % TD by two step spark plasma sintering. More metal elements are beneficial for higher hardness, elastic modulus, and strength. All multicomponent carbides show high nanoindentation hardness of around 38-40 GPa, higher than any of their constituents.

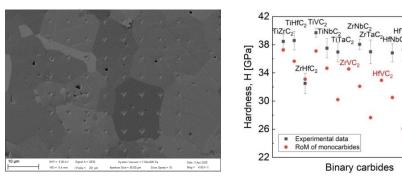


Figure 1: SEM image of typical indents performed in binary carbides; measured nanohardness and calculated hardness (by role of mixture of monocarbides) values of binary carbides

#### **ACKNOWLEDGMENT**

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### A NOVEL APPROACH TO UTILIZING NON-RECYCLED GLASS WASTE FOR A SUSTAINABLE SOCIETY

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### **ABSTRACT**

Glass, with its high optical transparency, mechanical strength, and resistance to chemical and thermal degradation, is widely used in everyday products as well as in advanced optical, biological, photonic, and optoelectronic applications [1, 2]. Despite its versatility, the glass industry faces major challenges in reducing environmental impact and managing non-recyclable glass waste. Closed-loop recycling is often impossible for high-quality applications (e.g., lenses, pharmaceutical containers) or fine glass dust generated during soda-lime glass recycling, which is typically landfilled.

The Glass4Ever project aims to transform unrecycled waste glass into spherical glass particles via flame synthesis, offering a sustainable precursor for additive manufacturing (AM). These particles will be applied in AM using SLA, DIW, and Binder-jet techniques to fabricate 3D structures for two main areas: (i) optoelectronics, as matrices for phosphor-in-glass (PiG), and (ii) construction, in lightweight facade panels with high reflectivity and insulation performance.

The project focuses on the synthesis, processing, and characterization of solid and hollow glass spheres and optimization of post-processing steps (drying, debinding, sintering). By upcycling landfilled glass waste into high-value AM feedstock, Glass4Ever contributes to sustainable material development and complements the activities of the EverGLASS project.

### **ACKNOWLEDGMENT**

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# IMPROVEMENT OF SILICON/GRAPHITE ANODE PERFORMANCE BY THE COMBINATION OF ATOMIC LAYER DEPOSITED ZnO COATING WITH FLUOROETHYLENE CARBONATE ADDITIVE

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### **ABSTRACT**

The properties of silicon/graphite anodes have been investigated with the aim to improve their electrochemical performance and use them in Li-ion batteries (LIBs). The work was focused on the impact of ZnO coatings and the addition of fluoroethylene carbonate (FEC) to the electrolyte on the properties of LIBs. We systematically compared the effects of the thickness of ultrathin ZnO coatings on the silicon/graphite anode, prepared by using atomic layer deposition (ALD), with and without the FEC additive in the electrolyte.

Both the ZnO coatings and the FEC additive significantly influenced the rate capability and long-term cycling stability of the anodes. The combination of ALD deposited ZnO coatings with the FEC additive in the electrolyte exhibited the best performance, enhancing both rate capability and capacity retention over extended cycling. These findings were further corroborated by electrochemical impedance spectroscopy (EIS), which highlighted the improvements in anode performance. Additionally, post-mortem analysis using X-ray photoelectron spectroscopy indicated an increased amount of LiF in the solid electrolyte interphase layer. This increase in LiF content could contribute to the enhanced stability and performance observed in ZnO coated silicon/graphite anodes when combined with the FEC additive in the electrolyte.

#### **ACKNOWLEDGMENT**

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### STOICHIOMETRY AND TRANSITION METAL TYPE EFFECTS IN THE COMPOSITIONALLY COMPLEX (TiZrHf-V/Nb/Ta)<sub>100-z</sub>-N<sub>z</sub> COATINGS: A REVIEW

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### **ABSTRACT**

High entropy nitrides, by definition, should correspond to homogeneous solid solutions of five or more metals with equimolar composition stabilized by configurational entropy. However, the compositions of the real multi-metal nitride coatings usually deviate from equimolar composition of metals and their nitrogen concentrations may cover wide range of stoichiometry. Therefore, the terms "complex concentrated" or "compositionally complex" nitride (CCN) coatings were used for this family of materials. Among CCN, the systems based on transition metals from the 4<sup>th</sup> and 5<sup>th</sup> group of elements being strong nitride formers were extensively investigated due to their attractive mechanical properties. The current work aims to review the influence of the addition of metals from the 5<sup>th</sup> group (Ta, Nb and V) into reactive sputtered (TiHfZr)-N coatings on their structure and mechanical properties as a function of nitride stoichiometry in comparison with (TiHfZr)-N and (TiHfZrVNbTa)-N coatings. Thus, not only the effects of individual TMs and nitrogen stoichiometry, but also the transition from the medium- to high-entropy stabilized CC-nitrides can be elucidated. The reviewed coatings were deposited using reactive DC magnetron sputtering (rDCMS) and reactive High Target Utilization Sputtering (rHiTUS) in hysteresis-free regime. The phase composition evolved, and the corresponding mechanical properties exhibited systematic changes depending on the type of TM added to the initial TiZrHf-matrix and nitrogen concentrations. The highest hardness and indentation moduli were observed in TiZrHf-N coatings with near-stoichiometric compositions. The additions of V, Nb and/or Ta slightly reduced the corresponding values. The differences between the individual metals were within the scatter of measurement, but the additional DFT suggested that Nb should be the most favorable from the viewpoint of the energy of formation.

### ACKNOWLEDGMENT

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### DESIGN, SYNTHESIS AND CHARACTERISATION OF HIGH-ENTROPY PEROVSKITE OXIDES FOR APPLICATIONS IN THERMOELECTRICS

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#### **ABSTRACT**

The effect of generating an electric voltage from heat energy, due to the difference in temperature between two dissimilar conductors, is the principle behind thermoelectricity. Even with the increasing energy demand in the world, only 34% of the energy is being efficiently utilised and the rest is wasted as released heat. Thermoelectrics can be the next step forward in the field of energy, since they can utilise the waste heat and convert it into electrical energy. The field of high entropy oxides has been fast gaining pace with applications ranging from ionic conduction, energy storage, energy conversion, etc. In high entropy perovskite oxides (ABO<sub>3</sub>), the improved functional properties arise from the combination of different elements on the A site, B site, or both, and the associated crystal structure distortions. Thus, the aim of the present work was to create a completely new thermoelectric material by entropy engineering.

High entropy perovskite oxides with 4 elements on the A-site (Ba, Ca, La and Sr), and 3 transition metal elements on the B-site (Ti, Zr and Nb) were synthesized using the solid-state method and spark plasma sintering. Thorough investigation of the synthesis, phase formation, microstructure evolution for these new materials was carried out. Further, electronic and thermal property characterisations for applications in thermoelectrics were conducted. The thermal conductivity, Seebeck coefficient, and electronic conductivity were measured, as these are the most important parameters for obtaining a good thermoelectric material. A very low thermal conductivity, ranging from 1.4 to 1.6 W/mK, has been achieved for these materials at 1273 K.

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# THE INFLUENCE OF FLAME-SPHEROIDIZED BIOACTIVE GLASS MICROSPHERES ON PLA COMPOSITE SCAFFOLD FUNCTIONALITY FOR BONE REPAIR

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### **ABSTRACT**

This study investigates how the incorporation of flame-spheroidized bioactive glass microspheres (BGMs) into poly(lactic acid) (PLA) matrices affects both mechanical properties and cell response of three-dimensional printed bone scaffolds. PLA-based filaments were extruded with either flame-created BGMs or irregular-shaped bioactive glass particles, and scaffolds were fabricated via fused deposition modeling. SEM analysis revealed that the BGMs exhibited superior dispersion within the matrix and contributed to improved filament tensile strength in comparison to irregular particles. Cube-designs PLA/BGM scaffolds displayed enhanced compressive strength and stable deformation behavior under loading, attributable to efficient stress propagation and reduced porosity. Biological assessments using MG-63 osteoblast-like cells indicated higher viability on PLA/BGM scaffolds after seven days, affirming the cytocompatibility benefits of spheroidized fillers. In vitro bioactivity tests confirmed robust calcium phosphate formation regardless of particle shape, but flamespheroidized microspheres yielded notable improvements in overall scaffold performance. These results establish BGMs as promising filler candidates to optimize mechanical integrity and biological outcomes in PLA-based 3D-printed scaffolds for bone regeneration, offering guidance for advanced composite design in tissue engineering applications.

#### ACKNOWLEDGMENT

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### ENHANCED HARDNESS AND FRACTURE TOUGHNESS IN DIBORIDE MULTILAYERED FILMS

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### **ABSTRACT**

The concurrent improvement of hardness (H) and fracture toughness ( $K_{IC}$ ) via multilayer structuring challenges the traditional assumption that these properties are inherently contradictory. Here, such a strategy is applied to transition metal diborides, a class of materials whose intrinsic brittleness has long limited their practical use. The mechanical behavior of  $TiB_2/TaB_2$  multilayers is explored as a function of the bilayer period ( $\Lambda$ ), using a combination of theoretical modelling and experimental validation. Density Functional Theory (DFT) is employed to examine the structural stability and mechanical performance of stoichiometric, hexagonal  $TiB_2/TaB_2$  superlattices over a  $\Lambda$  range of 3.9–11.9 nm. The simulations indicate that the optimal mechanical performance — with a hardness of 38 GPa and a fracture toughness  $K_{IC}(100)$  of 3.3 MPa.m<sup>1/2</sup> — is achieved at  $\Lambda$  = 5.2 nm.

Inspired by these predictions, multilayer coatings with  $\Lambda$  ranging from 4 to 40 nm were fabricated using direct current magnetron sputtering. However, due to sputtering-induced effects, the resulting films deviate from perfect stoichiometry and idealized structure. Structural characterization reveals that at  $\Lambda=4$  nm, the TiB<sub>2</sub>/TaB<sub>2</sub> layers form coherent superlattice interfaces. For larger  $\Lambda$  values, parts of the TaB<sub>2</sub> layers undergo a transition from crystalline to a disordered phase. These structural transformations are reflected in the mechanical response, assessed through nanoindentation and micromechanical bending tests. Hardness follows a Hall-Petch-like trend, peaking at 42 GPa for  $\Lambda=6$  nm. Fracture toughness enhancement, however, results from more intricate mechanisms, yielding two distinct maxima: 3.8 MPa.m<sup>1/2</sup> at  $\Lambda=6$  nm and 3.7 MPa.m<sup>1/2</sup> at  $\Lambda=40$  nm.

### **ACKNOWLEDGMENT**

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## HIGH-TEMPERATURE ARC AND MICROWAVE ASSISTED PYROLYSIS FOR DURABLE SILICA BASED COATINGS ON WOOD

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### **ABSTRACT**

The pyrolytic formation of a protective carbonized layer on wood surfaces—commonly referred to as charring—offers an environmentally friendly approach to extending the service life of wooden structural elements exposed to outdoor weathering. This study introduces novel wood surface pyrolysis methods based on: (a) a non-thermal kHz arc discharge operating in ambient air, and (b) rapid 2.45 GHz microwave surface heating, leading to either (i) microwave discharge breakdown or (ii) selective surface heating.

Spectroscopic measurements, interpreted using a grey-body radiation model, determined the arc discharge plasma temperature to be  $1,900\pm50\,^{\circ}\text{C}$ , approximately  $500\,^{\circ}\text{C}$  higher than that achieved by conventional naked-flame charring. Using a series of silica-based precursor formulations, the arc discharge plasma was evaluated for its ability to produce protective and/or microwave-absorbing coatings on wood surfaces. A formulation containing polyvinyl alcohol (PVA) and sodium silicate (Na<sub>2</sub>O<sub>3</sub>Si) increased the Brinell hardness of beech wood surfaces by nearly 50%. To develop high-quality microwave susceptors for wood heating, experiments were conducted in an oxygen-free environment to prevent ignition from combustible gases. Arc-pyrolyzed layers incorporating 5 wt.% tungsten carbide nanoparticles (55 nm) exhibited exceptional microwave absorption efficiency but also induced thermal runaway and undesirable microwave discharge breakdown. Ongoing work focuses on moderating microwave heating to achieve uniformly charred wood surfaces without localized overheating.

### **ACKNOWLEDGMENT**

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## NANOSCALE PROTECTIVE COATINGS ENHANCE Li-ION BATTERY PERFORMANCE

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### **ABSTRACT**

Although Li-ion batteries have become standard part of modern devices in electronics, mobility and energy storage, a quest for an increase in their energetic capacity and performance is still ongoing. Nanoscale protective coatings prepared by atomic layer deposition (ALD) has emerged as a powerful method to protect the electrodes and enhance the battery performance.

We have protected silicon/graphite (20 wt% Si, 80 wt% graphite) by ultrathin ZnO film grown by ALD. The ZnO-coated anode showed improved electrochemical performance at higher charging/discharging rates compared to the uncoated pristine electrode [1]. A combination of the ALD ZnO coating with fluoroethylene carbonate additive in the electrolyte showed an improvement of cyclability, resulting in a capacity retention of 60% after 500 cycles using the charging/discharging rate of 1 hour. Ultrathin Al<sub>2</sub>O<sub>3</sub> films were prepared on LiFePO<sub>4</sub> cathode using ALD technique. Similarly, as for the ZnO coated anode, the Al<sub>2</sub>O<sub>3</sub> covered cathode exhibit improved performance at higher charging/discharging rates and excellent cycle life. The protection of the Li-ion battery electrodes by ultrathin ALD coatings were shown to be an effective way of stabilizing the electrode/electrolyte interface. Nanoscale protective ALD coatings both on anodes and cathodes enhance the Li-ion battery performance in particular at higher charging/discharging rates.

### **ACKNOWLEDGMENT**

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### SPARK PLASMA SINTERING OF THE SAME HIGH ENTROPY CARBIDE BY VARIOUS MACHINES

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### **ABSTRACT**

Spark plasma sintering (SPS) is an established method for sintering various ceramic materials. However, manufacturers are using various power sources and experimental settings. Recently, High Entropy Ceramics (HECer) have been often prepared by SPS due to the necessity to prepare multi-component compositions, containing at least five different elements in equimolar or near-equimolar ratios, at high temperatures and pressures. The high mixing entropy and unique combination of various chemical bonds lead to a unique combination of properties exceeding the mixture rule. The formation of high entropy carbides is especially challenging due to the high temperatures needed for densification and electrical conductivity of the starting powders. The internal temperature in a reaction system depends on various parameters, and transferring these parameters between different SPS devices is difficult. This work used the model reaction between VNbMoTaW high entropy alloy and carbon to study the differences among various SPS devices. The formation of HECer and secondary phases indicates reactivity in various SPS devices when the same processing parameters were set up. The impact of the temperature control and electrical current on reactive sintering is investigated and discussed.

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### Ti-DRIVEN ORDER-DISORDER TUNING IN MULTICATIONIC A<sub>2</sub>B<sub>2</sub>O<sub>7</sub> ZIRCONATES

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#### **ABSTRACT**

The role of B-site titanium in controlling the order–disorder balance between pyrochlore (Fd-3m) and defect fluorite (Fm-3m) in multicationic A<sub>2</sub>B<sub>2</sub>O<sub>7</sub> high-entropy zirconates was investigated. Powders were obtained by solution-combustion synthesis and subsequently heat-treated to develop long-range order. Phase identification relied on X-ray diffraction with Rietveld refinement supported by validated CIF models; pyrochlore was discriminated via superstructure reflections - (111), (311), (331), (511/333) - that are absent in defect fluorite, whereas fundamental fcc lines overlap in both structure types. The A-site, composed of an equimolar rare-earth mixture, satisfies conventional high-entropy criteria (multi-principal cations with limited size mismatch). Increasing Ti decreases the average B-site radius and raises rA/rB. This shift correlates with a consistent evolution toward ordered pyrochlore, while Zrrich compositions remain partially disordered under identical processing. These results establish composition (Ti content, rA/rB) as a practical handle to steer order–disorder in A<sub>2</sub>B<sub>2</sub>O<sub>7</sub> zirconates and inform processing routes aimed at single-phase pyrochlore.

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### MECHANICAL PROPERTIES OF SIC/NbC CERAMIC COMPOSITES: COMPARISON OF CONVENTIONAL AND REACTIVE PROCESSING METHODS

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#### **ABSTRACT**

Silicon carbide (SiC) and niobium carbide (NbC) are advanced ceramic materials characterised by outstanding hardness, thermal stability, and chemical resistance, making them suitable for applications in extreme environments. Despite its potential as a ceramic matrix material, the intrinsic brittleness of SiC restricts its broader implementation, highlighting the need for strategies to enhance its fracture toughness. In this study, SiC/NbC composites were fabricated by spark plasma sintering (SPS) using two distinct approaches: conventional powder consolidation and partially reactive processing via in-situ SiC formation. NbC was introduced as a secondary phase in amounts of 5, 10, and 20 wt.% in the form of fine particles. The resulting composites were systematically examined in terms of microstructure, phase composition, and mechanical performance. X-ray diffraction and scanning electron microscopy confirmed a homogeneous distribution of NbC within the SiC matrix, with pronounced variations in interfacial bonding depending on the processing route. Mechanical testing revealed that composites produced by reactive processing exhibited superior hardness and fracture toughness, which are attributed to strong SiC-NbC chemical interactions and refined microstructural features. Particular attention was given to the chemical incorporation of NbC particles into the SiC matrix, a crucial step toward the future reinforcement with NbC fibers. Such reinforcement is anticipated to further improve damage tolerance and fracture resistance. Overall, the findings demonstrate that controlled NbC incorporation combined with optimised processing strategies enables the development of SiC-based composites with enhanced mechanical reliability for extreme environment applications.

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## SCREENING AND TESTING OF COOPER (II) PHOSPHATE AS A POTENTIAL BIOMATERIAL

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### **ABSTRACT**

Copper phosphate is known as a fungicide and pesticide to protect crops from fungal diseases and pests. It can also serve as a fertilizer, supplying essential copper and phosphorus for plant growth. New challenges relate also to the development of Cu<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub>/C composite as a high-capacity cathode material for rechargeable Na-ion batteries. In the field of biomaterials, the copper phosphate exhibits antimicrobial properties, which can be leveraged in medical applications to help prevent the spread of infections.

The objective of this study was to prepare copper phosphate (CuP) bulk ceramic materials with the main aim to investigate its cytotoxic effect. The prepared sintered phosphate materials were analysed by X-ray phase analysis (XRD), heating microscope and scanning electron microscope (SEM). In the second phase of the study, the biological response to the material was detected using in vitro assays, including MTS, LDH, and contact toxicity assay. X-ray phase analysis showed that the CuP sintered sample contained a single phase of Cu<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub>. SEM analysis of the fracture and surface samples revealed a microstructure that was almost dense, characterised by big grains. The ion release of material was performed in two media. The concentration of ions released in distilled water was found to be comparatively lower than that released in DMEM over the same time interval. Two different tests on the cytotoxicity were performed; however, the results of this study show that CuP samples sintered in a single batch exhibited inconsistent results in terms of cytotoxicity. Therefore, it should be noted that these partial results are yet to be verified by further testing.

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### NEXT GENERATION THERMOELECTRICS FOR SUSTAINABLE ENERGY: AN INTRODUCTION TO THE NEXTGENERATIONEU PROJECT

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#### **ABSTRACT**

The presentation will be focused on the introduction of the EU NextGenerationEU project: Next Generation Thermoelectrics for Sustainable Energy, funded through the Recovery and Resilience Plan for Slovakia. This project aims to establish a world-class research team, equipped with cutting-edge infrastructure, dedicated to the design and development of next-generation thermoelectric materials with markedly enhanced energy-conversion efficiency. Central to this endeavour is an innovative strategy to design entropy-stabilised perovskite oxides, thereby generating new and significant insights into the role of multi-principal element doping at both the A- and B-sites of ABO<sub>3</sub> perovskite structures in shaping thermoelectric performance. For the first time, the effects of non-equimolar multi-principal element doping on the thermoelectric properties of perovskite oxides will also be systematically investigated. Furthermore, the project introduces a novel manufacturing strategy, integrating entropy stabilisation with nanostructuring and vacancy engineering, to further optimise thermoelectric performance. Collectively, these approaches will make a substantial contribution to advancing sustainable energy technologies and supporting Europe's commitment to achieving climate neutrality by 2050.

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#### SILICA CROSS-LINKING IN ALKALI-ACTIVATED Si<sub>3</sub>N<sub>4</sub>

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#### **ABSTRACT**

Silicon-based binary hard ceramics (SiBHC), such as Si3N4 and SiC, are widely renowned for their exceptional mechanical properties, especially in term of structural strength and hardness, together with a fairly good thermal and chemical stability. For this reason, these materials have found applications in several fields where mechanical resistance is required. Recently, these materials were explored – albeit with varying degrees of success – for alternative applications such as electronics, in the case of SiC due to its semiconducting properties, and biomaterials for osteo-implants, as in the case of Si3N4 due to its exceptional chemical stability [1].

The densification of SiBHC powders requires, however, high temperatures and long dwelling times in muffle furnaces. Spark plasma sintering (SPS) is a good method to mitigate these extreme sintering conditions, though it comes at the cost of some carbon contaminations due to the use of conductive graphite molds. Alkali-activation [2] is a non-sintering method of densification, in which a secondary phase is produced through chemical attack and crosslinking. Silica, which is readily soluble in caustic soda (NaOH), form a sodium-silicate compound. SiBHC particles usually possess a passivated surface layer of silica which protects them from further air oxidation. While it is possible to produce pure SiBHC powder, the refinement process significantly increases costs. However, this silica layer might be a blessing in disguise, as it can be chemically tailored with relative ease [3]. Therefore, alkali-activation is a suitable technique for chemically tailoring and utilizing the silica layer to form a secondary thin layer between particles, which can function as a crosslinking agent and additive to modify the material properties. In this work, we present our efforts in the densification of SiBHCs through alkali activation and the development thereof.

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# STRATEGIES FOR ORGANIC RESIDUE ELIMINATION IN VAT PHOTOPOLYMERIZATION OF GLASS POWDERS FOR 3D-PRINTED OPTICAL-GRADE COMPONENTS

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#### **ABSTRACT**

The advancement of vat photopolymerization has opened pathways for the fabrication of customizable, high-precision glass structures, yet commercial realization is still hindered by limitations of optical clarity and compositional flexibility. Recent literature, such as studies on chemical structure-property relationships in photocurable monomers [1] and the effects of resin formulation on printed ceramic accuracy [2], emphasizes the critical influence of binder chemistry and resin design on process outcomes.

In this study, barium tableware glass was recycled to develop a particle-based resin system, utilizing a photocurable organic polymer as the binder for Digital Light Processing (DLP) 3D printing. The feedstock was optimized by systematically adjusting particle size (<40 µm and (40-63) μm) and controlling the slurry composition. The final formulation consisted of 57 wt% glass powder, 38 wt% photocurable resin, and 5 wt% polyethylene glycol, which ensured both printability and shape fidelity in the resulting printed objects. Printed gyroid scaffolds underwent post-printing debinding and carefully tuned multi-stage sintering, and the results were analyzed via X-ray diffraction, rheometry, and electron microscopy. The study reveals that the polymer matrix enables room-temperature shaping and supports intricate geometry while presenting challenges such as carbon entrapment from incomplete binder removal. critically affecting transparency and densification. Despite obtaining translucent scaffolds with clear visible light transmission, achieving full transparency remains dependent on further resin optimization, particle size distribution of precursor, complete debinding, and refined thermal treatment. This work provides a practical case study for the structure-property paradigm in resin selection, laying a foundation for future advances in multi-oxide glass 3D printing for nextgeneration optical, sensing, and photonic device applications.

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### ULTRA-HIGH-TEMPERATURE CERAMIC MATRIX COMPOSITE: DESIGN AND ABLATION RESISTANCE

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#### **ABSTRACT**

Among advanced ceramic materials, Ultra-High-Temperature Ceramic Matrix Composites (UHTCMCs) stand out as attractive options for extreme conditions owing to their remarkable thermal stability and strength. In this study, Cf/C and Cf/SiC ceramic matrix composites (CMCs) were developed and subsequently coated with a ZrB<sub>2</sub>-based ultra-high-temperature ceramic (UHTC) layer to enhance their ablation resistance. The coating was applied via a roomtemperature deposition process and cured at 200 °C in multiple layers. The microstructural and phase composition analyses were conducted using scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDS), and X-ray diffraction (XRD), confirming uniform coating distribution and phase formation. The coated samples underwent annealing at 1400 °C under an Ar atmosphere to remove organic compounds and improve thermal stability. Ablation performance was evaluated through an oxyacetylene torch test at surface temperatures of ~2300 °C for Cf/C and ~2050 °C for Cf/SiC, with real-time temperature monitoring via optical pyrometry. The results demonstrated a significant reduction in mass ablation rates, with the coated and annealed samples exhibiting up to 50% improvement in ablation resistance compared to uncoated counterparts. These findings highlight the potential of UHTC coatings for enhancing the performance of CMCs in aerospace and hypersonic applications.

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## AN ALTERNATIVE SYNTHESIS ROUTE FOR MATERIALS WITH HIGH CONFIGURATIONAL ENTROPY

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#### **ABSTRACT**

High entropy ceramic and ceramic systems with increased entropy are a very interesting subsection of materials that has gained popularity since the publication of Rost et al. in 2015. The main benefits are the enhanced structural stability and their enhanced functional properties, compared to a conventional doped system. This work presents a novel approach to produce an equimolar (Ba<sub>0.25</sub>Ca<sub>0.25</sub>Mg<sub>0.25</sub>Sr<sub>0.25</sub>) TiO<sub>3</sub> perovskite system with increased entropy. The common approach for the synthesis of such materials is to use carbonates or oxides of A-site elements (Ba, Ca, Mg, and Sr, in this case) and titanium dioxide as precursors, when using the conventional synthesis route. However, this approach necessitates the use of caustic alkaline earth oxides, including caustic calcium oxide or the use of carbonates of the alkaline earth elements (AEE), both of which do require the handling of soluble barium salts, which increases the risk of safe handling during the synthesis.

The aim of this work is to investigate the viability of an alternative synthesis route – from individual titanates of Ba, Ca, Mg, and Sr – to avoid the use of toxic barium compounds.

A series of samples was prepared by the mechano-synthesis route using equimolar concentrations of each AEEs using a high-energy mill. Samples were produced using various intensity of milling and processing time. These samples were subsequently thermally processed in high-temperature furnace at 1300 °C and 1400 °C. A conventional solid-state sintering sample was also prepared to compare against the samples prepared by this novel approach. The structure of prepared samples was studied by means of XRD, SEM, and EDS.

One of the unexpected results of the experiments is the finding, that the some of the samples sintered at the temperature of 1300 °C show intriguing properties and are, in fact, in some aspects superior to the samples sintered at higher temperature.

The comparative measurements of dielectric properties of samples produced by conventional solid-state sintering and high-energy mechano-synthesis indicate obvious differences between these materials. The prepared materials show promising results for the synthesis of AEE perovskites with high levels of configurational entropy.

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### PROCESS OPTIMIZATION FOR NITRIDATION OF 3D PRINTED MULLITE-BASED CERAMICS

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#### **ABSTRACT**

Mullite-based ceramics are materials with excellent thermal stability, low thermal expansion, and good resistance to thermal shock, making them suitable for various applications. Using additive manufacturing, mullite ceramics can be 3D-printed into complex geometries that are difficult to achieve with traditional ceramic forming techniques, offering greater design flexibility. These 3D-printed ceramics can be further modified through thermal treatments, such as nitridation, to enhance their properties.

Nitridation is a thermal process that incorporates nitrogen into the ceramic lattice, improving key properties such as hardness, wear resistance, thermal stability, and chemical durability. These enhancements make nitridated ceramics highly attractive for demanding engineering applications, including high-temperature structural components, cutting tools, electronics, and biomedical applications. Achieving uniform and controlled nitridation, however, requires careful optimization of process parameters, including temperature, nitrogen flow, and heating rates, as these factors strongly influence reaction kinetics and degree of conversion, phase composition, microstructure, and in final overall material performance.

In this work, nitridation of 3D printed mullite-based ceramics was studied with the aim of optimizing the process to enhance material properties. Cylindrical samples with infill at the level of 30 %, to ensure opened observable internal structures, were fabricated using Fused Filament Fabrication of Ceramics and were exposed to thermal treatments under various conditions. Sintering was carried out in a nitrogen atmosphere and the influence of temperature and nitrogen flow on samples properties was studied. Various techniques such as optical microscopy, scanning electron microscopy (SEM), X-ray diffraction analysis (XRD), and elemental analysis (CHN analysis) were used to evaluate the nitridated samples.

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### ENHANCED HIGH TEMPERATURE OXIDATION RESISTANCE IN HIGH ENTROPY BORIDE SYSTEMS

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#### **ABSTRACT**

Advanced materials are critical for next-generation systems operating under extreme thermal and mechanical loads. This work addresses this challenge by reporting the successful development of novel high-entropy boride composites with significantly improved oxidation performance. High-entropy diboride (Ti-Zr-Hf-Nb-Ta) was prepared via a boro/carbothermal reduction method of oxide precursors. This powder was then combined with various amounts of silicon carbide to create fully dense composites using Spark Plasma Sintering (SPS) at  $1800^{\circ}$ C. X-ray diffraction patterns of the synthesized powders and sintered compacts of HEB showed a single solid solution with hexagonal AlB2 structure without any residual oxide impurities. X-ray energy dispersive Spectroscopy (EDS) mapping results indicated the uniform elemental distribution of transition metals in the HEB phase. The optimized composite not only exhibits a notable increase in hardness, strength, and fracture resistance but also demonstrates superior stability against high-temperature dynamic oxidation. This material can offer a promising new class of high-performance materials for aerospace applications.

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