Institute
of Materials Research
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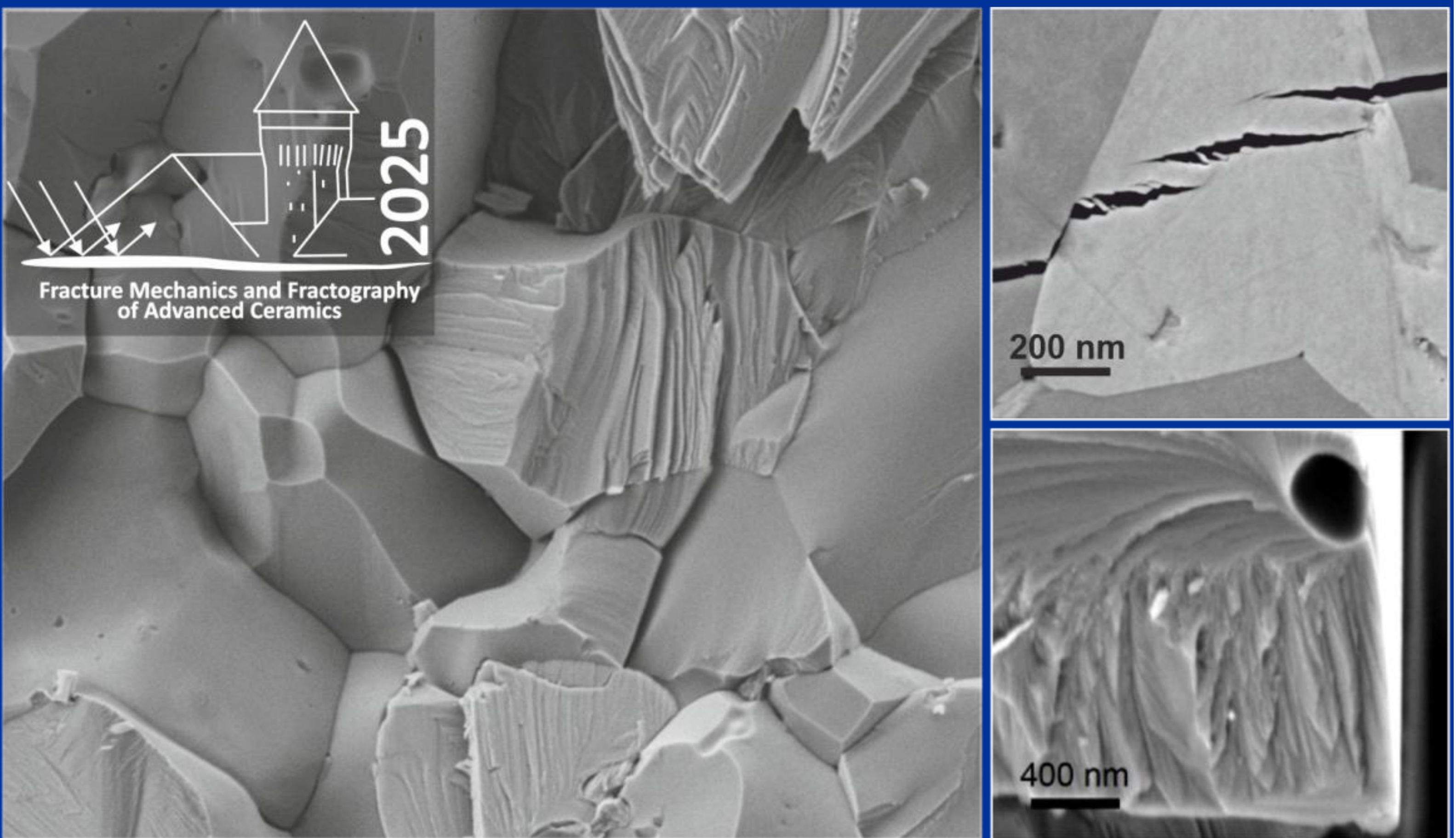
FRACTURE MECHANICS AND FRACTOGRAPHY OF ADVANCED CERAMICS CONFERENCE 2025

THE 8th INTERNATIONAL CONFERENCE

FAC 2025

6-9 October 2025
Smolenice Castle, Slovakia

--- BOOK OF ABSTRACTS ---



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MODELLING BONE REGENERATION BY CERAMIC DESIGN

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ABSTRACT
Several types of biomaterials have been used for total hip replacements (THRs) in orthopaedic surgery since the 20th century. Metallic materials, such as stainless steel, cobalt-chromium, titanium and titanium alloys have been widely used due to their excellent mechanical strength. Stainless steel was replaced soon by the reason of poor biocompatibility and corrosion in the body. Nowadays, Si_3N_4 is a new bioceramic for total hip replacements with extremely good mechanical properties. Hydroxyapatite (HA) is a widely used bioceramic in implantology considering its high bioactivity. A biodegradable and bioactive coating (e. g. HA) on the bioinert ceramic implant's surface (e. g. Si_3N_4) could induce tissue reactions and help avoid the rejection from the body in the critical early few days after the operation.In this presentation, different preparation of ceramic biomaterials will be showed from traditional technologies to novel applications. The main trends of the modern biomaterial science and technology, as well as the fundamental scientific problems will be discussed.

Keywords: bioceramics, implants, nano-hydroxyapatite

EFFECT OF 3D PRINTING DIRECTION ON FRACTURE BEHAVIOUR OF CERAMICS

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ABSTRACT

Additive manufacturing is a technology with growing industrial use, not only of polymers and metals but also for ceramic materials. The advantage is the possibility of manufacturing complex near-net shapes without the necessity of further machining. The drawback of this technology is the directionality of properties caused by layer-by-layer building the shape of the final product, and as a sintered surface. The 3D printing using the Digital Light Processing (DLP) technique is the most widespread technology for manufacturing components from ceramic materials. The knowledge about material behaviour with respect to the printing orientation under loading is essential for design and life lifetime of the component. The directionality of properties is one of the aspects; another one relates to the defects and/or features present at the surface or in the body linked to the processing parameters. Typical wrinkled surface perpendicular to the printing direction, interlayer cracks or trapped air bubbles in the plane of print. The other defects can be created during the debinding process, especially when thick walls or large closed cavities are present can cause crack formation due to overpressure during evaporation of polymeric binder. The fractography analysis was conducted on the fracture surfaces of the specimen for strength and fracture toughness measurements. It can reveal critical defects and demonstrate the effect of orientation on resulting properties in the view of fracture origin, crack initiation and propagation.

Keywords: Additive manufacturing, Fractography, Fracture toughness, Flexural Strength, Ceramics

ACKNOWLEDGMENT

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FABRICATION AND OXIDATION BEHAVIOUR OF Ti–Al–B_4C-BASED UHTC COMPOSITES

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ABSTRACT

The study investigates the formation mechanisms, phase evolution, and high-temperature oxidation resistance of ultra-high-temperature ceramic (UHTC) composites in the Ti–Al–B_4C system. Composites were synthesised using nanopowders of boron carbide and Ti–Al intermetallics via Spark Plasma Sintering (SPS) and Self-propagating High-temperature Synthesis (SHS). Samples were subjected to thermal ablation between 1800 and 2000 °C to simulate extreme operating conditions.X-ray Absorption Spectroscopy (XAS) at the PIRX beamline revealed the formation of key phases such as TiB_2 and protective Al_2O_3. These contributed to the outstanding oxidation resistance and structural stability of the composites. The Al_2O_3 layer, formed during ablation, acted as a dense, adherent barrier that minimized oxygen diffusion and preserved surface integrity.

Elemental mapping confirmed limited elemental migration and validated the effectiveness of the protective oxide layer. Despite the severe thermal environment, the Ti–Al–B_4C composites maintained their microstructural cohesion and phase composition, making them promising candidates for high-performance applications in aerospace and extreme thermal environments.These findings demonstrate the potential of Ti–Al–B_4C composites as next-generation UHTCs and lay the foundation for further optimisation through advanced synthesis and in-situ characterisation techniques.

Keywords: UHTCs, boron carbide, hardness.

ACKNOWLEDGMENT

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STRENGTH TESTS FOR CERAMIC ROLLERS

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ABSTRACT

Cylindrical components made from ceramic materials are frequently subjected to surface stresses during service. During the manufacturing process, strength-limiting surface flaws are introduced in various orientations relative to the roller axis. To accurately characterize the surface strength, testing strategies were developed based on the concept of the standardized notched ball test.

In the Notched Roller Test (NRT) a slender notch is introduced into the roller aligned with the roller axis. When the roller is compressed in radial direction primarily tangential surface stresses are introduced. In the Cross-Notched Roller Test (X-NRT), a test specimen is prepared by introducing a narrow circumferential notch along the circular cross-section of a roller, parallel to its end faces. When compressive forces are applied perpendicular to the notch, axial tensile stresses are generated on the outer surface region opposite the notch. This configuration makes the test particularly sensitive to flaws oriented perpendicular to the roller axis, such as circumferential grinding scratches. The method serves as a complementary approach to the established Notched Roller Test.

The stress distribution in such specimens was investigated using Finite Element Analysis (FEA) across a range of geometrical configurations. The stress amplitude was found to be proportional to the applied load and dependent on both specimen geometry and Poisson's ratio. Strength measurements using the X-NRT and NRT methods were conducted on commercial silicon nitride rollers with varying notch geometries. The results were compared to biaxial strength tests performed on specimens manufactured from the same roller material and failure analysis using fractorgraphy was conducted to interpret the obtained strength results.

THERMAL SHOCK RESISTANCE, WEAR BEHAVIOR AND OXIDATION RESISTANCE OF SILICON NITRIDE BASED NANO-COMPOSITES

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ABSTRACT

Six different sintering aids (Lu_2O_3, Yb_2O_3, Y_2O_3, Sm_2O_3, Nd_2O_3 and La_2O_3) were used for the processing of dense Si_3N_4/SiC micro/nano composites. Thermal shock resistance: A critical temperature difference increased with an increasing ionic radius of RE^{3+} for both the composites and the monoliths. Wear behavior: The friction coefficient as well as the specific wear rate decreased with a decreasing ionic radius of rare-earth elements in both the monoliths and the composites. High bonding strength and the high fracture toughness are the reasons why the ceramics doped by Lu exhibited the best wear resistance. Oxidation resistance: Composites exhibited predominately parabolic oxidation behaviour indicated diffusion as the rate limiting mechanism. Exception was only the Si_3N_4-SiC composite sintered with Lu_2O_3. In this case diffusion of cation has been strongly suppressed because of the beneficial effect of stable grain boundary phase and the presence of the SiC particles predominately located at the grain boundaries of Si_3N_4.

MITIGATING CATASTROPHIC FAILURE: GLASSES WITH ADAPTIVE STRUCTURES AND LAMINAR CERAMICS WITH ENGINEERED STRESS PROFILES

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ABSTRACT

Despite the continuous progress in improving the strength of ceramics and glassy materials, their use remains constrained due to their propensity to catastrophic failure. Here, we present examples of methods that reduce the risk of fracture without warning in brittle materials: (i) developing damage-tolerant glass structures, and (ii) introducing engineered stress profiles (ESP). An ultra-crack-resistant glass was obtained by utilizing a self-adaptive structure in alkali-aluminoborate glasses. The aluminoborate glasses were synthesized using the melt-quench technique and subjected to Li^+/Na^+ or Li^+/K^+ ion exchange. The ion-exchanged glass exhibited remarkable damage resistance, exceeding 100 N, which is significantly higher than the typical oxide glass, often found to be below 30 N. This significant damage tolerance is attributed to a metastable glass structure, enabling adaptive deformation. As an alternative strategy for ceramics, engineered stress profiles have been proposed to prevent spontaneous crack propagation and improve damage tolerance. Laminar ceramics were fabricated by thermo-compressing layers produced by tape casting of ZrO_2, Al_2O_3, and $MgAl_2O_4$. Evaluating the Hertzian contact response and crack formation in the prepared laminar ceramics revealed that the production of residual compressive stress on the surface, due to the different physical properties of different layers, not only enhances strength but also improves the reliability of the ceramics. Critical parameters, key considerations, and new possibilities in the development of damage-tolerant ceramics are discussed.

Keywords: Catastrophic Failure, Glass, Ceramics, Engineered Stress Profile, Adaptive network

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CREEP PROPERTIES OF (Ti-Zr-Nb-Ta-Cr)C HIGH ENTROPY CERAMICS WITH DIFFERENT CR CONTENTS

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ABSTRACT

The compressive creep properties of (Ti-Zr-Nb-Ta-Cr)C high entropy carbides (HECs) with different Cr contents, prepared by hot-pressing of the carbothermal reduction reaction powders, are investigated at 1400~1600 ºC with stresses of 150~250 MPa. The phase composition, microstructure, and dislocation structure of the HECs were investigated by X-ray diffractometry, scan electron microscopy, electron back scatter diffraction, transmission electron microscopy (TEM) and principal component analysis (PCA), respectively. The introduction of Cr reduces the grain size, and an appropriate amount of Cr can enhance the grain boundary strength, thereby improve creep resistance and lower the steady-state creep rate. However, excessive Cr can lead to the formation of brittle phases, which decreases the creep resistance. The results indicate that the steady-state creep rates range from 4.14×10^{-9}/s to 6.22×10^{-7}/s. Under the same creep conditions, the steady-state creep rate of HEC-Cr3 is the lowest. Creep damage includes grain growth, pore formation, and grain boundary cracking. The creep mechanisms involve atomic diffusion, grain boundary sliding, and dislocation slip. At 1500 ℃, burgers vector of dislocation is a/2<011>, and the primary slip system is a/2<011>{111}.

Keywords: (Ti-Zr-Nb-Ta-Cr)C, High-entropy ceramics (HECs); Creep properties; Grain boundary strengthening; Dislocation motion

FRICTION AND WEAR BEHAVIOR OF ALUMINA-BORON CRABIDE COMPOSITES WITH SILVER AND GRAPHENE NANOPLATELET SOLID-LUBRICANT ADDITIVES

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ABSTRACT

This study evaluates the friction and wear of Al_2O_3–B_4C composites modified with Ag and graphene nanoplatelets (GNPs). Powders were homogenized and sintered by the SPS method (1700 °C/50 MPa, Ar); microstructure and phases were analyzed using SEM-EDS and XRD. Mechanical properties were determined by HV10 measurement, fracture toughness KIC, and nanoindentation (hardness H, modulus E). Reciprocating sliding (ball-on-flat) tribological tests were performed with steel and SiC tribological partners (balls) of 6 mm diameter at 3 N load and a total sliding distance of 300 m. The friction curves show average steady-state COF values: against SiC 0.53 (Al_2O_3–B_4C), 0.50 (Al_2O_2–B_4C–5Ag–1GNPs), and 0.45 (Al_2O_3–B_4C–5Ag–3GNPs); against steel 0.78 (Al_2O_3–B_4C) and 0.63 for both modified composites (5Ag–1GNPs and 5Ag–3GNPs). The specific wear rate Ws [mm3/(N·m)] for (steel/SiC) was: $7.60\times10^{-7}/3.58\times10^{-6}$ for Al_2O_3–B_4C, $1.66\times10^{-7}/2.62\times10^{-6}$ for Al_2O_3–B_4C–5Ag–1GNPs, and 6.83×10−8/2.47×10−6 for Al_2O_3–B_4C–5Ag–3GNPs. Compared to the base material, Ws with the steel tribopartner decreased from 7.60×10^{-7} to 1.66×10−7 (Al_2O_3–B_4C–5Ag–1GNPs) and 6.83×10^{-8} (Al_2O_3–B_4C–5Ag–3GNPs) $mm^3/(N\cdot m)$; with SiC, it decreased from 3.58×10^{-6} to 2.62×10^{-6} and 2.47×10^{-6} $mm^3/(N\cdot m)$. For HV10, the base Al_2O_3–B_4C composite exhibited the highest hardness (18.50 ± 0.87 GPa); after lubricant addition, it decreased to 17.94 ± 1.99 GPa for 5Ag–1GNPs and 16.46 ± 1.53 GPa for 5Ag–3GNPs. Conversely, fracture toughness KIC improved after modifications: from 3.12 ± 0.29 $MPa\cdot m^{1/2}$ (Al_2O_3–B_4C) it increased to 3.44 ± 0,37 $MPa\cdot m^{1/2}$ (5Ag–1GNPs) and 3.62 ± 0.42 $MPa\cdot m^{1/2}$ (5Ag–3GNPs), with the 5Ag–3GNPs variant showing the highest KIC. Nanoindentation revealed that the base Al_2O_3–B_4C composite had the highest hardness H and modulus E (H = 37.42 ± 6.87 GPa; E = 464.4 ± 29.7 GPa). After lubricant addition, these values decreased: for 5Ag–1GNPs to H = 31.63 ± 6.12 GPa and E = 407.3 ± 34.5 GPa. The 5Ag–3GNPs variant showed slightly higher hardness than 5Ag–1GNPs (H = 32.87 ± 6.18 GPa) and a similar modulus (E = 410.1 ± 38.8 GPa). From a tribological perspective, the Ag/GNPs additives form an effective tribofilm: COF decreases especially against SiC (min. 0.45 for 5Ag–3GNPs), and the wear rate is most significantly reduced in contact with steel. This represents the very first systematic tribological evaluation of Al_2O_3–B_4C composites with 5 wt.% Ag and 1–3 wt.% GNPs in reciprocating sliding, additionally with a parallel comparison of two tribological partners (SiC vs. steel) under identical conditions, providing a complete set of quantitative metrics (COF,

Ws, HV10, KIC, H, E) on the same samples, thereby demonstrating the link between mechanical properties and tribological behavior.

Keywords: Al_2O_3–B_4C composites, Graphene nanoplatelets, Coeficient of friction, Wear rate.

ACKNOWLEDGMENT
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DEVELOPMENT OF ULTRA-HARD UHTCS FROM MAX PHASES AND BORON CARBIDE

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ABSTRACT
Composites belonging to the ultra-high-temperature ceramics (UHTCs) group are characterised by high melting points, high hardness, good mechanical properties and the ability to operate at high temperatures. These materials include metal borides and carbides of transition metals and their production often involves the application of high temperatures or sintering aids, e.g. high pressure or electric current. A reactive sintering approach can significantly mitigate the sintering conditions and produce composites with a dense and fine-grained microstructure. The use of boron carbide and MAX phases as precursors has never been explored in the literature, but it shows considerable potential in terms of process and resulting properties.
Here, UHTCs were obtained from B_4C and Nb_2AlC upon reactive spark plasma sintering (RSPS) in the 1800-2000°C temperature range, depending on the MAX volume fraction. X-ray diffraction and SEM-EDS analyses confirmed complete conversion of the MAX phase into ultra-fine NbB_2 grains and Al_4C_3, reaching microhardness about 30-32 GPa. The microstructure evolution process, preliminary mechanical properties and the overall energy balance are discussed.

Key words: UHTCs, MAX phase, boron carbide, hardness.

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ULTRA-FAST SINTERING OF BIXBYITE-STRUCTURED HIGH-ENTROPY OXIDES FOR SOLID OXIDE FUEL CELL APPLICATIONS

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ABSTRACT
Among the recent advancements in ceramic processing, ultra-fast high-temperature sintering (UHS), introduced in 2020, has transformed sintering by reducing processing time from hours to mere seconds [1]. In this study, single-phase bixbyite-structured high-entropy $CeO_{2-\delta}\cdot(RE,La,Sm,Y)_2O_3$ [RE = Gd, Nd] powders were synthesized by combustion synthesis and consolidated using UHS. Chemical homogeneity and phase purity of the sintered samples were confirmed by X-ray diffraction (XRD) and energy-dispersive X-ray spectroscopy (EDX). By optimizing UHS parameters such as applied current and dwell time, dense ceramics (relative density > 95%) with nanometric grains and minimal porosity were successfully obtained. The rapid heating rates inherent to UHS effectively suppressed grain growth while maintaining phase stability. Sintering under an argon atmosphere induced the reduction of Ce^{4+} to Ce^{3+}, as confirmed by X-ray photoelectron spectroscopy (XPS), resulting in the formation of oxygen vacancies within the lattice. These structural features contribute to enhanced ionic conductivity, making the material a promising candidate for solid oxide fuel cells (SOFCs) and oxygen sensors [2]. This work underscores UHS as a fast, energy-efficient route for fabricating high-entropy oxide ceramics for next-generation energy applications [3].

Keywords: High-entropy oxides, Ultra-fast high-temperature sintering, Microstructure, Ionic conductivity

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THE EFFECT OF MULTI-METAL ENVIRONMENT ON THE FRACTURE TOUGHNESS OF (Hf-Ta-Zr-Nb)C HIGH-ENTROPY CARBIDE GRAINS

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ABSTRACT

Ultra-high temperature high-entropy carbides are promising new materials for extreme environment applications. The room-temperature deformability of their grains can be tuned by varying the effective valence electron concentration of incorporated metals. This was studied in terms of fracture toughness in (Hf-Ta-Zr-Nb)C grains and was compared with HfC and TaC, known to be the most brittle and the least brittle rock salt carbides, respectively. The fracture toughness was determined for the {001} planes, as the lowest-energy cleavage planes, using microcantilever bending of notched bars and by first-principles density functional theory (DFT) calculations. The samples were sintered by spark plasma sintering, and the crystallographic orientations of grains were determined by electron back-scatter diffraction. The notched bars were focused ion milled from grains of {001} and {101} facets with beam directions perpendicular to the {001} cleavage planes and were subjected to bending experiments. Using DFT simulations, both the elastic constants of the relaxed crystals and the surface energies resulting from the separation of large supercells were determined, and these values were used for the calculation of fracture toughness. Experiments revealed that the lowest fracture toughness corresponds to HfC (K_{IC}=3.0 $MPam^{0.5}$) and (Hf-Ta-Zr-Nb)C (K_{IC}=3.1 $MPam^{0.5}$) high-entropy carbide, while TaC exhibits a higher value of ~10% (K_{IC}=3.3 $MPam^{0.5}$). Simulations yielded comparable but lower values than experiments, with the same trend, resulting from their different electronic structures.

Keywords: High-entropy carbide grains, fracture toughness, surface energy, microcantilever bending, DFT simulation.

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FRACTOGRAPHIC CHARACTERIZATION OF DLP-PRINTED MULTI-POROUS HYDROXYAPATITE SCAFFOLDS WITH GYROID ARCHITECTURE

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ABSTRACT
Triply periodic minimal surface (TPMS) structures, such as gyroids, are increasingly explored in the field of additive manufacturing due to their unique combination of high surface area, tunable porosity, and mechanical robustness [1]. Among these, multi-porous gyroid structures—those integrating porosity at both the macro- and micro-scale—offer intriguing possibilities not only for biomedical applications [2]. Recent advancements in 3D printing technologies, particularly in vat photopolymerization, have enabled the fabrication of highly complex gyroid geometries with controlled porosity. However, the mechanical performance and failure mechanisms of these architected materials remain subjects of ongoing investigation. In particular, fractographic analysis—examining the morphology of fracture surfaces—can provide essential insights into the interplay between hierarchical porosity and crack propagation. This study presents a comprehensive analysis of multi-porous gyroid structures fabricated via digital light processing (DLP) from hydroxyapatite suspensions. The multi-porous character of the microstructure was achieved using proper design of gyroid structure, by adding a combustible organic phase (cellulose and starch fibres/particles) and nanoporosity between grains. We investigate the mechanical response of the samples under compressive loading and characterize the fracture behavior using detailed fractography. By correlating structural design parameters with observed fracture modes, this work aims to deepen the understanding of how multi-level porosity influences failure behavior in architected lattices.

Keywords: additive manufacturing; digital light processing; hydroxyapatite, mechanical properties, fractography

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INFLUENCE OF ENTROPY ON THE PIEZOELECTRIC AND MECHANICAL PERFORMANCE OF BCZT CERAMICS

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ABSTRACT

Present study aims to investigate the effect of entropy on the piezoelectric behaviour of barium calcium zirconate titanate (BCZT) ceramics. Specifically, it compares the conventional composition $(Ba_{0.85}Ca_{0.15})(Zr_{0.15}Ti_{0.85})O_3$ with a high-entropy counterpart, $(Ba_{0.5}Ca_{0.15}Sr_{0.15}La_{0.15}K_{0.15})(Zr_{0.15}Ti_{0.90})O_3$. The ceramic powders were synthesised via a self-combustion method, followed by deagglomeration in a stabilised suspension using milling balls on rollers. The powders were then processed through spray drying and subsequently sintered using Spark Plasma Sintering (SPS).

To confirm the formation of a single-phase structure and assess the microstructure, X-ray diffraction (XRD) and scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy (SEM-EDS) analyses were performed. The electrical properties of the ceramics were comprehensively evaluated, including measurements of dielectric constant, dielectric loss, breakdown voltage, piezoelectric coefficient (d_{33}), electromechanical coupling factor, and ferroelectric characteristics such as polarisation–electric field (P–E) hysteresis loops, remanent polarisation, and coercive field. Additionally, the mechanical performance of the samples was assessed through detailed analysis of their elastic properties, hardness, and fracture toughness, with particular attention given to fractographic evaluation.

Keywords: High Entropy, BCZT, SPS.

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SMALL SCALE DEFORMATION AND FRACTURE OF BORIDE/CARBIDE HIGH ENTROPY CERAMICS

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ABSTRACT
The fracture characteristics of dual – phase high entropy ceramics were investigated using micro mechanical testing and detailed microstructure and fractography.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 was determined by means of scanning transmission electron microscopy (STEM) in conjunction with energy dispersive X-ray spectroscopy (EDS).. Micro - cantilevers prepared by focused ion beam from oriented facets of grains were studied. 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 present fracture origins (nano – pores or grain boundaries) which were in all cases in nano-metric range.

Keywords: High entropy ceramics, microcantilever, fractography.

ACKNOWLEDGEMENTS
This research work has been supported by the EU Next Generation EU through the Recovery and Resilience Plan for Slovakia under the project No. 09101-03-V05-00009.

MICROCANTILEVER BENDING STRENGTH OF REACTIVE SINTERED NOT-EQUIMOLAR DUAL-PHASE HIGH-ENTROPY CERAMICS

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ABSTRACT

Microcantilever bending tests, based on linear beam theory and supported by micro/nanofractographic analysis, were used to investigate the bending strength of non-equimolar dual-phase high-entropy ceramics: $(Ti_{0.49}Zr_{0.12}Nb_{0.13}Hf_{0.11}Ta_{0.15})C$ – $(Ti_{0.82}Zr_{0.04}Nb_{0.08}Hf_{0.03}Ta_{0.03})B_2$. The ceramics were synthesized via reactive spark plasma sintering and exhibited average grain sizes of 2.2 µm for the carbide phase and 3.4 µm for the boride phase. High-resolution analysis revealed that most grain/phase boundaries exhibited a continuous and sharp segregation layer (~1.5 nm wide) enriched with Fe, Co, and Ni impurities. Both carbide and boride grains demonstrated excellent nanohardness, with average values of 38.3 GPa and 41.3 GPa, respectively. Microcantilevers were fabricated using focused ion beam (FIB) milling from carefully polished surfaces at random locations, ensuring that both carbide and boride grains—as well as grain/phase boundaries—were present within the cantilever volume. All tested microcantilevers failed at grain/phase boundaries, indicating these as the primary fracture origins. The measured bending strength ranged from 3.8 to 7.3 GPa, depending on the boundary's location, size, orientation, and character. Fractographic analysis showed that most fractures initiated at carbide/boride interfaces, with an average strength of 4.7 GPa. However, fractures originating at boride/boride and carbide/carbide boundaries were also observed, exhibiting slightly higher strength values.

Keywords: high-entropy ceramics, boride, carbide, strength, grain boundaries

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THE INFLUENCE OF THE LOADING TYPE ON THE APPLICATION POTENTIAL OF INDUSTRIAL MONO- AND MULTILAYER TRIBOLOGICAL COATINGS

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ABSTRACT

Four types of industrially applied monolayer PACVD coatings (TiBN, TiAlN, CrN, CrAlN; v = 3-6 µm) and their DLC top-layered variants (TiBN+DLC, TiAlN+DLC, CrN+DLC and CrAlN+DLC; v = 2.2-3.4 µm), applied on a precipitation hardened X153CrMoV12 tool steel substrate was subjected to scratch tests with increasing loading force (F = 2-150 N) to characterize their tribological performance. From the friction coefficient and normal load vs. scratch length diagrams, the critical loading force causing the complete detachment of the coatings was determined, and the subcritical and critical damage mechanisms were identified using 3D OM analysis. The critical loading forces for monolayer coatings were 100-150 N, whereas for DLC top-layered coatings, they were 40-80 N, indicating that DLC multi-layered coatings are much more sensitive to scratch-type loading. Ball-on-disc wear tests were performed with SiC ball counterpart (F = 10 N, v = 0.1 m/s, L = 1000 m) on the same mono- and multilayer coating systems demonstrating that the monolayer coatings can reduce the specific wear rate (k) of the tool steel by 74-88%, while the DLC top-layered versions of the same coatings by 96-98% (Fig. 1) compared to the uncoated base material. The observed difference is explained by the change in wear mechanism, which was abrasive for the monolayers and tribochemical for the DLC top-coated multilayers. For the same reason, the friction coefficient of the tested monolayer coatings was closely identical to that of the uncoated reference material; however, it was reduced by a minimum of 80% when a DLC top layer was applied to the coatings. It is concluded that for the tested precipitation-hardened cold-forming tool steel used in tribological applications, the selection of the optimal coating material is significantly influenced by the type of loading. Monolayer coatings offer higher scratch resistance, whereas DLC multilayer systems more effectively enhance wear resistance.

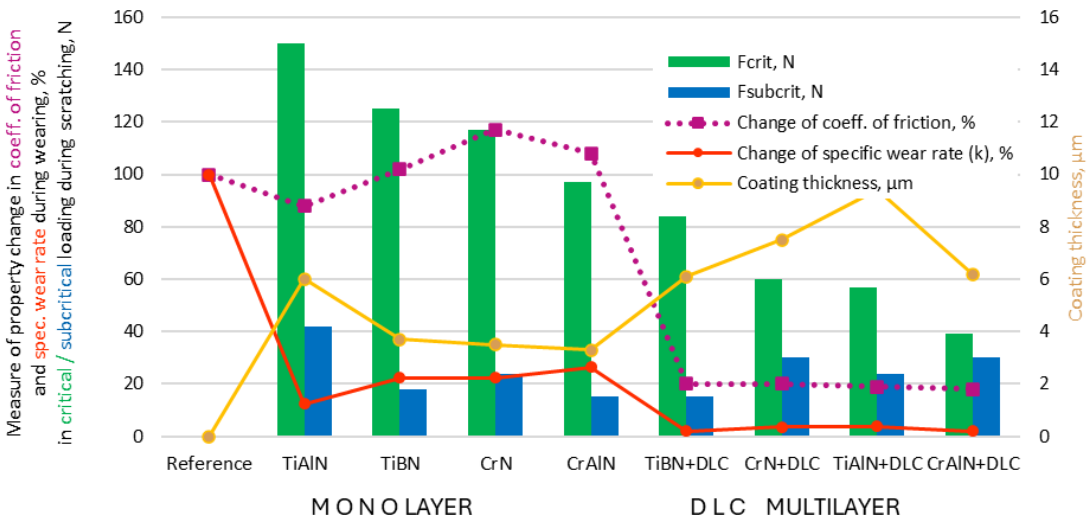

Fig. 1. Complex evaluation of the tribological performance of the tested coating systems

Keywords: ceramic coating, DLC multilayered coating, scratch test, ball-on-disc test

ACHIEVING BROADBAND EMISSION FROM K_2ZNGEO_4 POLYCRYSTALS

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ABSTRACT

In recent years, self-activated phosphors (SAP) have attracted significant attention for several applications, including lighting devices, displays, scintillators, sensors, bioimaging, and luminescent security ink. However, their recent progress in white-light illumination has been hindered by significant challenges in achieving broad emission. In this study, we report a new white colour emitting SAP based on the K_2ZnGeO_4 germanate. The polycrystalline powder was synthesized using the solid-state reaction method. X-ray diffraction and scanning electron microscopy (SEM) analyses confirmed the single-phase crystal structure and revealed irregularly shaped particles. The luminescence properties were investigated in detail. Remarkably, under excitation by ultraviolet (UV) and X-ray sources, this material exhibited broadband white light emission spanning from 400 to 700 nm. The CIE 1931 coordinates (x, y) = (0.388, 0.441) were calculated from the PL spectra recorded at 366 nm excitation. The significant role of structural defects on the luminescence of this SAP was also investigated. The combined results indicate that this SAP is a promising candidate for white-light illumination and possibly other luminescent devices.

Keywords: Germanate phosphor, Broadband emission, Defects, Luminescence, wLED

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DISLOCATION STRUCTURES AND SLIP SYSTEMS IN SUBSTOICHIOMETRTIC TACX CERAMICS

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ABSTRACT

Tantalum carbide (TaC), as one of the least brittle ultra-high temperature ceramics, is a promising material for extreme environment applications, such as leading edges of spacecrafts. The deformability and fracture behaviour of TaC grains are strongly related to their dislocation structure and active slip systems through to control of the metal-to-carbon ratio. In the present study, this was investigated in substoichiometric TaC ceramics using indentation and transmission electron microscopy (TEM). Three TaC_x samples with stoichiometry in the range of x=0.8-1 were prepared from precursor carbide and metal powders using spark plasma sintering. Dislocations were introduced into the crystal structure through instrumented indentation. Based on recently published computational predictions, it was anticipated that the depletion of carbon sublattice induces a change in glide plane of dislocations from the 'ductile' {111} to the 'brittle' {110} type around x=0.9. This hypothesis was experimentally verified by means of image analysis and electron diffraction in a TEM. Furthermore, the presence of stacking faults within the atomic structure was examined, since such defects can facilitate the formation of partial dislocations and thereby influence the selection of the operative slip system. Through this analysis, new insights were obtained regarding the relation between local stoichiometry, defect configurations, and the mechanisms of plastic deformation and fracture in TaC.

Keywords: Tantalum carbide, stoichiometry, slip systems, TEM, indentation.

ACKNOWLEDGMENT

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UNDERSTANDING BRITTLE FRACTURE BEHAVIOUR OF SINGLE CRYSTALLINE MATERIALS

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ABSTRACT

Brittle single crystals play a vital role in microelectronic components, where mechanical reliability under thermo-mechanical loading is critical during both manufacturing and operation. These materials are usually chosen for their functional properties, but often their mechanical performance—particularly strength and fracture behaviour—remains insufficiently understood. Anisotropy, surface condition, and crystal orientation all contribute to significant variability in failure responses.

This study investigates the mechanical performance of single crystals through fracture experiments conducted across multiple length scales and under varying loading conditions. Results show a pronounced dependence of strength on crystallographic orientation and surface preparation, with grinding and polishing direction—especially relative to cleavage planes—emerging as a key factor in determining mechanical integrity. Fractographic analysis reveals the origins of surface- and sub-surface damage, offering insight into the mechanisms that govern fracture initiation and propagation.

These findings contribute to a deeper understanding of the mechanical reliability of brittle single crystals and support the development of processing strategies tailored to optimize both structural robustness and functional integration in microelectronic systems.

Keywords: Brittle Single Crystals, Anisotropy, Crystal Orientation, Biaxial Strength, Surface Preparation

DAMAGE TOLERANCE AND STRENGTHENING EFFICIENCY IN ION-EXCHANGED GLASS-CERAMICS

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ABSTRACT

Glass-ceramics are now emerging as a promising alternative to chemically strengthened glasses in wearable electronics, particularly since they are expected to be insensitive to damage occurring during processing, resulting in lower production costs and complexity. However, there is limited information available regarding the damage tolerance of glass ceramics and its impact on the efficiency of the strengthening process in glass-ceramics. In the present work, we investigated the impact of prior surface defects on the efficiency of chemical strengthening in glass-ceramics. Ion-exchangeable aluminosilicate glasses containing various amounts of a nucleating agent were prepared using the melt-quench technique. Afterward, these glasses underwent a ceramization heat treatment to produce glass-ceramics. Rectangular samples were cut from the pristine glass and polished to create defect-free surfaces; then, controlled surface defects were introduced using instrumented indentation with a Vickers tip. The indented samples were subjected to chemical strengthening by immersion in a molten KNO_3 salt bath at 450°C for 4 to 8 hours. The impact of ion exchange and its efficiency were evaluated by measuring the bending strength of the indented samples. Additionally, the damage resistance of the chemically strengthened glass-ceramics was assessed using indentation techniques. The results indicated that glass-ceramics are generally less sensitive to surface damage and demonstrate a higher efficiency in ion exchange. However, extensive crystallization decreased the damage tolerance of the glass-ceramics. The effects of glass crystallization on ion-exchange efficiency and the damage tolerance of glass-ceramics are critically discussed.

Keywords: Glass-ceramic, Aluminosilicate, Chemical Strengthening

ACKNOWLEDGMENT

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EFFECT OF IN SITU SiC FORMATION ROUTE ON THE MICROSTRUCTURE AND MECHANICAL PROPERTIES OF (TiZrHfNbTa)C-SiC COMPOSITIES

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ABSTRACT

High-entropy ceramics (HECs) are a promising class of materials known for their excellent thermal and mechanical stability. However, their inherent brittleness and poor sinterability pose significant challenges for practical applications. A viable approach to overcome these issues is the incorporation of a secondary phase, such as silicon carbide (SiC), which improves fracture toughness and oxidation resistance. In particular, in situ formation of SiC during sintering enhances interfacial bonding and promotes uniform dispersion. In this study, high-entropy (TiZrHfNbTa)C ceramics with in situ formed SiC (5 wt.%) were synthesized via three distinct routes: (1) carbothermal reduction of SiO_2, (2) reaction of elemental Si with carbon, and (3) reaction of elemental silicon with residual carbon in the initial powders. All samples were consolidated by Spark Plasma Sintering (SPS), which allows rapid densification while minimizing grain growth. Phase composition was characterized by X-ray diffraction (XRD), microstructure by scanning electron microscopy (SEM), and mechanical properties by Vickers hardness and fracture toughness testing. The results showed that the route of SiC formation significantly affected the microstructure and properties manufactured samples. The most effective route—elemental Si with carbon—produced composites with the best combination of hardness, toughness, and microstructural uniformity.

Keywords: High entropy carbides; Silicon carbide; Spark Plasma Sintering; Microstructure; Mechanical properties

ACKNOWLEDGMENT

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WEAR CHARACTERISTICS OF REACTIVE SINTERED HIGH ENTROPY (TI-ZR-NB-HF-TA)B_2 + (TI-ZR-NB-HF-TA)C COMPOSITE

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ABSTRACT

The present study is aimed to investigate the tribological characteristics of reactive sintered ($Ti_{0.82}$-$Zr_{0.04}$-$Nb_{0.08}$-$Hf_{0.03}$-$Ta_{0.03}$)B_2 + ($Ti_{0.49}$-$Zr_{0.12}$-$Nb_{0.13}$-$Hf_{0.11}$-$Ta_{0.15}$)C dual-phase high entropy ceramics and to describe characteristic wear mechanisms using linear reciprocating tribological tests and detailed fractography. The investigated composite is a high-density material with grain sizes of the carbide and boride phases of 2.2 µm and 3.4 µm, respectively. The majority of grain and phase boundaries are decorated by a continuous sharp ~ 1.5 nm wide segregation of Fe, Co and Ni impurities. Thanks to the chemical compositions, the boride and carbide grains have excellent nanohardness with average values of 41.3 GPa and 38.3, respectively. The average friction coefficient values during the test with 5 and 25 N were relatively stable, with values of 0.65 and 0.59 after a 5000 sec testing procedure. During the testing with 50 N, the average friction coefficient value slightly decreased to 0.48. The specific wear rate under the load of 5 N is very low, with a value of 1.87×10^{-7} mm^3/Nm. The specific wear rate is increasing to a value of 1.64×10^{-6} mm^3/Nm for 25 N and 2.64 × 10-6 mm^3/Nm for 50 N load, respectively. Detailed fractography revealed different wear mechanisms depending on the applied testing load.

Keywords: high-entropy ceramics, composites, reactive SPS, mechanical properties, wear.

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TEXTURE ENGINEERING OF PIEZOELECTRIC CERAMICS

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ABSTRACT
Texture engineering has attracted significant attention as a promising process for developing advanced lead-based and lead-free piezoceramics due to their outstanding piezoelectric properties that are comparable to those of piezoelectric single crystals. The fabrication of textured piezoceramics involves synthesis of plate-like crystals to be used as templates, alignment of these templates along a preferred crystallographic direction within a randomly distributed matrix powder, and their growth by re-precipitation of the dissolved matrix onto the surface of the templates during sintering. The growth behavior of the template is strongly dependent on its stability during calcination and sintering, which ultimately determines piezoelectric properties of the textured piezoceramics by influencing their chemical composition and microstructure. In this presentation, the stability of template during texturing of lead-based and lead-free piezoceramics is reviewed in terms of its effects on the chemical composition, microstructure, and piezoelectric properties of textured piezoceramics.

Keywords: piezoelectric ceramics, texture engineering, template, stability

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MECHANICAL PROPERTIES AND FRACTURE CHARACTERISTICS OF ULTRA-HIGH TEMPERATURE BINARY CARBIDES

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ABSTRACT

Transition metal elements of group IV and V of PTP, namely Ti, Zr, Hf, V, Nb, and Ta were selected to prepare all possible combination of binary carbides. The rationale to select these chemical compositions is due to higher thermal stability of their mono carbides, oxides or borides, known as UHTC ceramics. This study investigates the mechanical properties, microstructure and fracture characteristics of high-temperature binary carbides. Binary carbides were prepared using ball milling for 4 hours of starting powder of monocarbides. The samples were spark plasma sintered at 2100°C for 10 minutes at 70MPa in argon atmosphere, respectively. Almost fully dense single phase carbides were prepared. However, the XRD confirmed low amount of secondary phases, mainly oxides. Hardness, elastic modulus, fracture toughness, and mechanisms of toughening were estimated and the influence of chemical composition of carbides on mechanical properties was analysed. It has been observed that chemical composition significantly influenced grain size of carbides. Nanohardness for binaries shows an enhancement compared to the rule of mixtures values.

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INDENTATION HARDNESS AND A NOVEL APPROACH DETERMINING FRACTURE TOUGHNESS OF HARD COATINGS

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ABSTRACT

The fracture toughness of hard ceramic coatings, particularly nitride and boride-based, is a critical property that significantly affects their durability in various applications [1][2]. Measuring this property is challenging due to the thin-film nature of the coatings (typically up to 5 micrometers thick) and the influence of the underlying substrate. Key factors influencing fracture toughness include residual stress, material composition, and microstructure [3]. Internal residual stress within the coating can increase its susceptibility to cracking, and controlling this stress is crucial for improving toughness. The type of ceramic material and its composition—including substoichiometric structures and point defects—can enhance fracture toughness, especially in nitride coatings [4]. In addition to scratch testing and nanoindentation methods [5], several other approaches are currently being investigated to evaluate fracture toughness. These include analytical models based on crack spacing—such as the Internal Energy Induced Cracking (IEIC) method [6]—Micro-Cantilever Bending Tests [4], and stress-to-energy conversion techniques. Effective management of internal stresses remains a key strategy for enhancing the fracture resistance of ceramic coatings.

Keywords: fracture toughness, hard coatings

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WEAR DAMAGE IN THE HARD $(NbMoTaW)_{100-z}$-$(CN)_z$ COATINGS DURING DRY FRICTION IN AIR

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ABSTRACT

The influence of nitrogen and carbon stoichiometry, z, in two sets of $NbMoTaW_{100-z}(CN)_z$ coatings deposited using reactive DC magnetron co-sputtering on tribological behavior has been investigated during dry friction in ambient air. Co-sputtering occurred from a composite NbMoTaW target at a constant DC power (300 W) and C was co-sputtered at 300 W and 600 W. Thus, the coating with two levels of carbon of ~18 at% (low carbon concentration series) and ~40 at% (high carbon concentration series) respectively, were achieved. The additions of nitrogen in both series resulted in the stoichiometry ranged from 30 at% to 65 at%. The hardness and indentation moduli of the coatings were controlled by the carbon/nitrogen ratio and the overall stoichiometry. The coatings with high carbon concentrations were >5 GPa harder values; nitrogen influence depended on the phase composition and stoichiometry. The highest hardness values ~ 50 GPa, were achieved in the near-stoichiometric fcc coatings with high carbon content. However, superhardness level was exceeded even in the (near-)stoichiometric coatings with low carbon content. Friction and wear behavior depended on the composition and hardness, respectively. The coefficients of friction (COF) in the hardest (near-)stoichiometric coatings tend to be high with the wear controlled by abrasion from the wear debris pulled out from the wear track. The COF and wear rates were lower in the over-stoichiometric coatings due to the presence of free carbon.

Keywords: compositionally complex carbonitride coatings, reactive DC magnetron co-sputtering, superhardness, wear damage, friction

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A COMBINED SCRATCH BEHAVIOR ANALYSIS OF SUPER-ADHERENT, THICK POLYCRYSTALLINE DIAMOND COATINGS ON STEEL

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ABSTRACT
Polycrystalline diamond (PCD) coatings with a TiBN interlayer were produced by HF-CVD on an X46Cr13 (1.4034) steel substrate, utilizing a unique cooling treatment during the CVD process to reduce residual stresses. The substrate had different surface topographies created by ultrasonic vibration superimposed machining (UVSM) [1], and the coated samples were grouped into two categories: finer (Ra ~ 1 µm) and rougher (Ra ~ 2 µm). The adhesion of the coatings was studied using non-standard scratch tests with a steel-ball stylus. The scratch diagrams and optical microscopy provided insufficient information on the scratch resistance, as the applied maximum normal load (150 N) did not cause delamination of the well-adhered PCD coatings. 2D and 3D profilometry were efficient in obtaining information on the scratching behavior of the finer coatings, as the skewness (Rsk and Ssk) was closely related to scratch resistance. In contrast, no relevant information was obtained for the rougher coatings. The residual stresses in the scratched diamond layers were determined using Micro-Raman mapping, based on the findings of a previous work [2]. The current study showed a clear correlation between the scratch groove profiles and residual stresses. Scratch damage mechanisms were studied by SEM. The tribological behavior of the investigated thick, super-adherent PCD coatings, without coating delamination, can be successfully characterized by combining a non-standard scratch testing procedure with a complex failure analysis that correlates some purposefully selected surface topography features with the residual stress data.

Keywords: PCD coating, scratch test, steel-ball stylus, surface roughness, residual stresses

ACKNOWLEDGMENT
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MECHANICAL AND TRIBOLOGICAL BEHAVIOR OF Al_2O_3–ZrO_2 CERAMIC COMPOSITES REINFORCED WITH CARBIDES UNDER STEEL CONTACT

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ABSTRACT

This study compares an Al_2O_3–ZrO_2 matrix ceramic with two composites formed by adding 42 vol.% of either titanium carbide (TiC) or mixed tungsten–titanium carbide (WTiC), all consolidated under identical spark plasma sintering (SPS) conditions. We assess how the carbide reinforcement chemistry modifies the microstructure, mechanical response (nanoindentation hardness H, Young's modulus E), and tribological behavior (coefficient of friction, μ; specific wear rate, W_s) during dry reciprocating sliding against 100Cr6 steel at normal loads of 10 N and 25 N. Relative to the Al_2O_3–ZrO_2 matrix, both reinforcements modestly increase stiffness and hardness (TiC: E +≈5.2%, H +≈3.2%; WTiC: E +≈9.0%, H +≈4.3%). At 10 N, the steady-state friction of all three materials is statistically similar within the reported scatter. At 25 N, the TiC composite exhibits a lower COF ≈ 0.467 than both the matrix (≈ 0.524) and the WTiC composite (≈ 0.545). Reinforcement markedly improves wear resistance versus the matrix at both loads: at 10 N, W_s decreases by ≈1.9× (TiC) and ≈1.6× (WTiC); at 25 N, the reduction is ≈14× (TiC) and ≈5× (WTiC). Overall, TiC delivers the most favorable tribological response, particularly under the higher load. These results demonstrate that adding a hard carbide phase substantially benefits wear performance relative to the Al_2O_3–ZrO_2 matrix, with TiC outperforming WTiC under the studied conditions. We attribute the difference primarily to reinforcement-dependent tribolayer stability and microchemical effects at the sliding interface.

Keywords: alumina, zirconia, titanium carbide, tungsten–titanium carbide, wear.

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NANOINDENTATION OF HIGH – ENTROPY BORIDE/CARBIDE COMPOSITE REINFORCED BY SiC WHISKERS

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ABSTRACT

The investigated boride/carbide composites reinforced by SiC whiskers (5, 10, 15, and 20vol. % SiCw) were prepared by reactive sintering at 2100°C/ 70 MPa for 10 min. Nanoindentation was done using a diamond Berkovich tip on a KLA (former Agilent) G200 NanoIndenter. In each sample, 64 indents (arranged in arrays of 8x8) were performed using a Continuous Stiffness Measurement (CSM) mode with a maximum displacement of 150 nm a (see below SEM images). Indents were examined by scanning electron microscopy (SEM) using a Zeiss Auriga Compact SEM/FIB microscope. Hardness and Youngs modulus were evaluated as the average of the depth range of 90-110 nm assuming a Poisson ratio of ν=0.2. The nanohardness of the boride grains changing from 45.1 GPa to 46.4 GPa according to the composition of the investigated composites. The nanhardness of the carbide grains is higher, changing from 46.9 GPa to 48.7 GPa.

DEGRADATION MECHANISMS OF THE CATALYST CERAMIC LAYER CONTAINING ELECTROSPUN FIBERS

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ABSTRACT

The Hydrogen Evolution Reaction (HER) is a crucial component of electrochemical water splitting, and carbon-based nanofibers (CNFs) have shown significant catalytic potential. This study details the synthesis and characterization of electrospun carbon-ceramic composite nanofibers and investigates their degradation mechanisms during prolonged HER application in both acidic and alkaline electrolytes. Electrospun fiber non-woven mats were thermally treated under an Ar/H_2 atmosphere and utilized directly as independent electrode materials. The long-term stability was rigorously evaluated by chronoamperometry at 1.6 V for 22 hours in KOH (alkaline) and H_2SO_4 (acidic) media. Comprehensive post-test characterization (XRD, SEM-EDX, ATR-FTIR, and Raman spectroscopy) revealed that the reaction environment dictates the degradation pathway, though it only slightly compromises the overall electrocatalytic performance. The prepared fibers consist of a carbon matrix embedding oxide and phosphide nanoparticles on the surface and within the bulk (displayed in Fig. 1). In the alkaline medium, HER operation led to the etching of the outer carbon layer, exposing the ceramic nanoparticles. Conversely, exposure to the acidic medium primarily resulted in minor morphological and phase composition changes to the active phosphide nanoparticles. The overall results demonstrate that the catalyst maintains relatively stable HER activity under prolonged operation in both media with minimal overall degradation, confirming its robust potential.

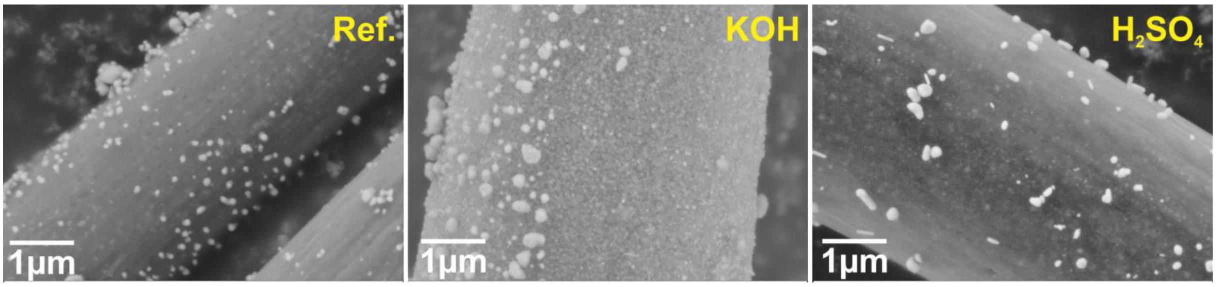

Figure 1: SEM image of the prepared microfibers: pristine electrocatalytic fibers, fibers tested in alkaline and acidic media, respectively.

Keywords: fibers, electrospinning, catalyst, stability test.

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OVERCOMING INVERSE DESIGN CHALLENGES: OPTIMIZING ENGINEERED STRESS PROFILES IN LAMINAR CERAMICS VIA ML-FEA

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ABSTRACT

Although producing engineered stress profiles in multi-material laminar ceramics is a solution to catastrophic failure, accurately predicting and fabricating the desired layer designs is challenging. The complex non-linear mechanics of thin ceramic layers and the vast number of design possibilities—over a million in a twenty-layer bi-ceramic laminate—make inverse design for achieving the required damage tolerance practically unfeasible. To overcome this limitation, we present a prediction and optimization technique that combines Machine Learning (ML) and Finite Element Analysis (FEA). The method comprises: (i) applying FEA to predict and capture the response of selected laminar structures to Hertzian contact damage, and (ii) employing machine learning-based Bayesian optimization using the FEA output to obtain the laminar structure with the highest damage tolerance. The optimized designs developed by the ML-FEA-based method and their performance against the empirical results were evaluated. Laminar ceramics composed of Al_2O_3-$MgAl_2O_4$ layers were prepared using the tape-casting and thermo-compression methods; subsequently, their response to Hertzian contact damage was determined. While the machine learning (ML) outcomes seemed to overestimate the damage resistance of laminar ceramics, it was discovered that defects introduced during the shaping and densification processes contributed to a decrease in damage resistance. Potential solutions for integrating the impact of these defects into the ML model or finite element analysis (FEA) prediction were critically discussed.

Keywords: Hertzian Contact, Machine Learning, Finite Element Analysis

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SYNTHESIS OF NIOBIUM CARBIDE NANOFIBERS AS NANOFILLERS FOR ADVANCED CERAMIC MATRIX COMPOSITES

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ABSTRACT
This work details the synthesis of carbon/niobium-based fibers from PAN/Nb_2O_5 pre-ceramic/polymer composite precursors via a needleless reactive electrospinning approach. This method is unique because the ceramic component forms in situ during the fiber fabrication and collection process. The electrospinning solution was prepared from polyacrylonitrile (PAN), niobium pentachloride ($NbCl_5$), and N,N-dimethylformamide (DMF). The ceramic precursor was transformed through the hydrolysis of $NbCl_5$ by atmospheric moisture, resulting in amorphous hydrated Nb_2O_5 encapsulated within the PAN fibers. The resulting amorphous precursor fibers were pyrolyzed in an argon atmosphere between 1100 and 1700 °C to study the influence of temperature on their phase composition and morphology. Pyrolysis at 1100 °C yielded carbon fibers containing NbC nanoparticles and traces of oxides. At 1300 °C, a complete carbothermal reduction of Nb_2O_5 led to the formation of pure NbC. Further increasing the temperature to 1500 °C and 1700 °C caused notable grain growth in the NbC particles, while preserving the overall fiber morphology. All fibers were characterized by DTA/TG, SEM, TEM/EDX, BET, and XRD.

Keywords: Needleless electrospinning, Ceramic nanofibers, Niobium carbide, UHTC nanofibers, Reactive electrospinning.

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PROPERTIES OF BORIDE-MATRIX COMPOSITES REACTIVELY SINTERED FROM BORON CARBIDE AND SILICIDE PRECURSORS

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ABSTRACT

Manufacturing composites with borides matrix by traditional route demands high temperature sintering to assure high density of products. Utilization of reactive sintering could decrease demanded sintering temperatures in a significant level and allows to achieve composites with fine-grained microstructures. Presented work concerns mechanical property characteristic of two types composites. One derived from boron carbide (B_4C) and titanium silicide (TiSi) and the second one from B_4C and zirconium silicide (Zr_5Si_3). Both composites after sintering were composed of homogeneously distributed boride phases (TiB_2 or ZrB_2) and silicon carbide (SiC). The mean grain size of constituent phases were about 1 micrometre. Microstructural aspects of cracking were discussed in the context of local inhomogeneity in a few micrometres range. Discussion of result is supported by measurements of interaction with synchrotron radiation.

Keywords: UHTC, borides, carbides, intermetallic.

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MICROSTRUCTURAL EVOLUTION OF MULTICOMPONENT REFRACTORY CARBIDES DURING SPARK PLASMA SINTERING

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ABSTRACT

Refractory metal carbides are widely recognized for their excellent hardness, thermal, corrosion, oxidation and wear resistance. Multicomponent carbides have been employed in structural applications requiring materials that withstand extreme conditions. This work is focused on microstructural characteristics and phase evolution of binary and ternary carbides (combination of transition metal elements of group IV and V of PTP) during spark plasma sintering. These carbides were prepared using ball milling for 4 hours of starting powder of monocarbides. The samples were spark plasma sintered (two step sintering at 1800°C/5min and 2100°C/10 min) at 70MPa in argon atmosphere, respectively. For comparison of selected properties also monocarbides TiC, ZrC, HfC, VC, NbC, and TaC were sintered and characterized. Almost fully dense single phase carbides were prepared. However, the XRD confirmed low amount of secondary phases, mainly oxides in the form of ZrO_2 and HfO_2. The influence of chemical composition of carbides on microstructure, phase formation, fracture mode, density, and basic mechanical properties was analysed.

ACKNOWLEDGMENT

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THERMAL SHOCK RESISTANCE OF 3D-PRINTED ALUMINA-BASED CERAMIC COMPONENTS

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ABSTRACT

In this work, we explore the potential of the Lithography-based Ceramic Manufacturing (LCM) technology for designing thermal shock resistant alumina-based ceramic systems. The first design concept demonstrates the use of the LCM technology to print alumina-based ceramic architectures with tailored residual stresses. In this multi-material design approach, alumina layers are embedded between alumina-zirconia (ZTA)-layer regions. The corresponding tailored compressive residual stresses in the embedded alumina layer regions act as an effective barrier to crack propagation, providing a minimum strength for the 3D-printed architecture [1]. In the second design concept, the capabilities of spatially tailoring porosity in 3D-printing alumina-based ceramic systems using LCM for enhancing the damage tolerance is presented. In such architectures, porous interlayers are introduced by using a polymeric pore forming agent, which is removed after the debinding process. The (specific) strength of the architecture is governed by the outer-dense alumina regions, whereas the damage tolerance after thermal shock is enhanced through the weak porous interlayers [2]. Based on the case studies, the feasibility of designing and printing more-complex shaped demonstrator components with tailored residual stresses and porosities are represented.

Keywords: Additive Manufacturing, Alumina, Thermal shock, Multi-material.

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MECHANICAL PROPERTIES OF SiC/NbC CERAMIC COMPOSITES FABRICATED BY CONVENTIONAL AND REACTIVE PROCESSING

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ABSTRACT
Silicon carbide (SiC) and niobium carbide (NbC) are advanced ceramic materials with exceptional hardness, thermal stability, and chemical resistance, making them attractive for applications in extreme environments. SiC is a promising ceramic matrix material; however, its inherent brittleness limits its wider use, and therefore, methods to increase its fracture toughness are required. In this work, SiC/NbC ceramic composites were fabricated by two different processing methods using SPS: conventional powder consolidation and partially reactive processing via in-situ SiC formation. The secondary NbC phase was added in the form of particles in amounts of 5, 10, and 20 wt.%. The microstructural properties, phase composition, and mechanical properties of the composites were systematically investigated. X-ray diffraction and scanning electron microscopy confirmed the uniform dispersion of NbC particles in the SiC matrix, with significant differences in interfacial bonding depending on the processing method. Mechanical characterisation revealed improved hardness and fracture toughness of the composites obtained by reactive processing, which is attributed to strong SiC–NbC chemical interactions and improved microstructural features. Special emphasis was placed on the ability of the SiC matrix to chemically incorporate NbC particles, which is a critical prerequisite for the subsequent introduction of NbC fibers – an avenue of ongoing research. Such reinforcement is expected to further enhance fracture toughness and damage resistance. The present results demonstrate that tailoring the microstructure through controlled NbC addition and optimised processing enables the design of SiC-based composites with excellent mechanical performance.

Keywords: carbides; ceramic composite materials; sintering; silicon carbide; niobium carbide.

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THE IMPACT OF DISLOCATIONS ON THE MACROSCOPIC STRENGTH AND FRACTURE OF OXIDES WITH MgO AS A MODEL MATERIAL

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ABSTRACT

The targeted introduction of dislocations in ceramics has the potential to enhance mechanical performance as well as to improve their functional properties. Typically, an increased dislocation density could only be achieved in small material volumes. In recent years, tailoring the dislocation density (up to $\sim 10^{15}$ m^{-2}) of ceramics in a larger volume at meso/macroscale at room temperature has been made available through a surface scratching technique. So far, the effects of such a treatment on the material's hardness, crack-initiation/propagation behaviour and the microscopic strength have been investigated, and a toughening effect through increased dislocation density has been observed [1].

In this work, the effect of an increased dislocation density on the macroscopic strength of MgO single crystals has been investigated. Specimens were commercially polished, cut into square plates, and subsequently scratched to purposely introduce dislocations. They were tested in an unaltered polished state ($<10^{12}$ m^{-2}), with a slightly increased dislocation density ($\sim 10^{14}$ m^{-2}) and with a dislocation density comparable to metallic materials ($>10^{15}$ m^{-2}). Ball-on-Three-Balls-tests as well as Ball-on-Ring-tests were performed to assess the biaxial strength. Finite element analysis was performed to assess the influence of anisotropic material properties on the applied stress field as well as the susceptibility of such loading configurations to specimen edge failures. Extensive fractographic analysis and chemical etching was conducted to discern different types of failure and to explain the observed difference in strength.

Keywords: Magnesia, Dislocations, Single Crystals, Mechanical Strength, Macroscopic testing.

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CONTACT STRENGTH OF SILICON NITRIDE BEARING BALLS – IS THERE A LINK TO TENSILE STRENGTH?

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ABSTRACT

Ceramic bearing balls are essential components for so-called hybrid bearings, used as electro-mechanical systems that require electrical insulation, like bearings in modern wind turbines. Silicon nitride ceramics is favored for this application due to its high hardness, stiffness, and strength combined with low density. However, premature failure of ceramic bearing balls may occur due to crack initiation and propagation under contact stresses. Therefore, assessment of relevant mechanical strength properties is mandatory.

Generally, the tensile strength of ceramics can be understood within the framework of linear elastic fracture mechanics. The scatter of strength arises from a size distribution of fracture origins and is conventionally described by Weibull statistics. This encompasses a size effect on strength. A standardized tensile strength test for balls is the "notched ball test" [1]. Contact loads as they occur in use induce high compressive stresses below the contact zone and tensile stresses immediately outside of the compression zone. Exceeding a critical load typically ring cracks are initiated in the tensile loaded region. To investigate the contact strength of ceramic balls a symmetrical setup of a ball-on-ball contact test has been developed. These tests involve pressing two balls together with increasing load until cracks are formed. An attempt was made to predict the contact strength distribution on the basis of notched ball test results using Weibullian size extrapolation. Experiments showed that predictions significantly underestimate the empirical contact strength data, which exhibit greater strength and much lower scatter than anticipated. Different concepts to resolve this discrepancy are discussed and a more sophisticated statistical evaluation of defect distributions will be presented.

Keywords: silicon nitride balls, notched ball test, Hertzian contact test.

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PROCESSING, STRUCTURE AND FRACTURE BEHAVIOUR OF $VNbTaXWC_5$ (X=Cr, Mo) HIGH-ENTROPY CARBIDES

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ABSTRACT
The improvement of room temperature fracture toughness of brittle transition metal carbides is demanding for the development of next-generation ultra-high temperature ceramics. Experiments have revealed that the increase of effective valence electron concentration (VEC>9) in high-entropy carbides (HECs) promotes the brittle to ductile transition. Recent calculations have predicted the synthesizability of $VNbTaXWC_5$ (X=Cr, Mo) compositions, belonging to the highest deformability HECs with a VEC of 9.4, which are addressed to be investigated in the present work. The compositions were synthesised from both oxide and carbide precursors using carbothermal reduction and spark plasma sintering. Structural characterisation (XRD, SEM and EDS) revealed the formation of single-phase rock salt structures with homogenous distribution of metal elements. The samples were subjected to micro/nanoindentation to determine hardness and elastic modulus. The fracture behaviour of the compositions was studied using three-point bending of Single-Edge V-Notched Beams (SEVNB). The fracture toughness values were found to be similar for both compositions, with values in the range of K_{IC}=2.9-3.4 $MPam^{0.5}$, depending on the precursor quality and processing routes. These values are in the range of data reported for other HEC compositions measured by indentation methods, which generally overestimate the real fracture toughness.

Keywords: High-entropy carbides, fracture toughness, carbothermal reduction, spark plasma sintering, indentation.

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SCRATCH BEHAVIOR OF OXYGEN-DOPED CrN COATINGS

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ABSTRACT

A single-layer CrN coating, widely used in toolmaking, was modified with different amounts of oxygen additions. The coatings were prepared industrially by balanced DC magnetron sputtering assisted by an independent microwave plasma source. The modified coatings have a multilayer, sandwich-like architecture in which a gradient layer connects the top and base layers of different compositions (Table 1). The unmodified CrN and four types of modified (Cr-N-O) coatings were characterized by instrumented hardness testing. The chemical composition was characterized by energy-dispersive spectroscopy. Scratch tests were performed with progressive loading force (F = 2-100 N). With increasing oxygen content, the critical load (F_{crit}) causing coating delamination exhibits a decreasing trend as the nitrogen-to-oxygen ratio increases up to a 1:1 ratio. At the same time, it increases monotonically as the introduced oxygen ratio is further increased. It is found that oxygen-doping changes the damage mechanism in the gradient layer region. At higher oxygen ratios (75 and 100%), the initial direction of the subcritical cracking network at the edges of the scratch groove points in the scratching direction, which gradually becomes perpendicular to the scratch groove axis in the load range at which the scratch tool passes through the gradient layer.

Table 1. Technological parameters and some characteristics of the investigated coatings

Sample	Gas flow (sccm)			Chemical composition (at%)			Functional layer		
	Ar	N_2	O_2	Cr	N	O	Thickness (nm)	Deposition time, (s)	Deposition rate (nm/s)
CrN	60	40	0	60	32	7	3400	10800	0.315
Cr25	60	30	10	61	29	10	1200	3600	0.333
Cr50	60	20	20	61	20	18	1600	3600	0.444
Cr75	60	10	30	63	12	24	1900	3600	0.528
Cr100	60	0	40	67	5	28	3400	3600	0.555

Keywords: PVD coating, CrN layer, microwave plasma source, scratch test, damage mode.

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MECHANICAL PERFORMANCE OF JOINED HIGH-ENTROPY CERAMICS: FRACTURE AND STRENGTH ANALYSIS

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ABSTRACT

The objective of this research was to evaluate the mechanical performance of three distinct techniques for joining of two different High-Entropy Carbides (HECs), such as (HfZrTaNbTi)C and (MoNbTaVW)C. While both materials were developed using Field Assisted Sintering Technology, different sintering conditions were used. (HfZrTaNbTi)C was prepared at the temperature of 2050°C and a pressure of 70 MPa with a dwell time of 7 min, while (MoNbTaVW)C was prepared at 1600 °C for 15 minutes under 70 MPa in. Three different joining methodologies were investigated: direct joining, brazing with a metallic interlayer, and solid-state diffusion bonding. The most successful approach yielded crack-free joints incorporating a ceramic/metal composite interlayer, which demonstrated a maximum flexural strength of 340 MPa following a joining process at 1800 °C. The observed high joint strength and the consistent failure of samples outside the joined interface confirmed the potential of these techniques for HEC integration.

Keywords: high entropy ceramics, wetting, joining, flexural strength.

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FRACTURE STRENGTH OF CeO_2 DOPED ZIRCONIA PREPARED BY CONVENTIONAL AND SPARK PLASMA SINTERING

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ABSTRACT
CeO_2 doped zirconia powders (ZrO_2–10Ce or ZrO_2–20Ce) were used for preparation of the samples. The samples were prepared either by conventional sintering or spark plasma sintering (SPS). The sample preparation procedure is given in the table 1. On the samples micro-cantilevers of pentagonal shape were prepared using SEM/FIB technique. Subsequently the samples were subjected to bending test using nanoindentor with flat punch. Example of prepared micro-cantilever before and after bending test is presented in figure 1. The average values of measured fracture strength for the prepared samples are given in Table 2.

Table 1: The sample preparation procedure.

Sample	Composition	Sintering	Temp. [°C]	Time [min.]	Temp. [°C]	Time [min.]
I	ZrO_2-10Ce	conventional	1600	1200		
J	ZrO_2-20Ce	conventional	1600	1200		
B	ZrO_2-10Ce	SPS	2000	10		
B1	ZrO_2-10Ce	SPS + conv.	2000	10	1400	240

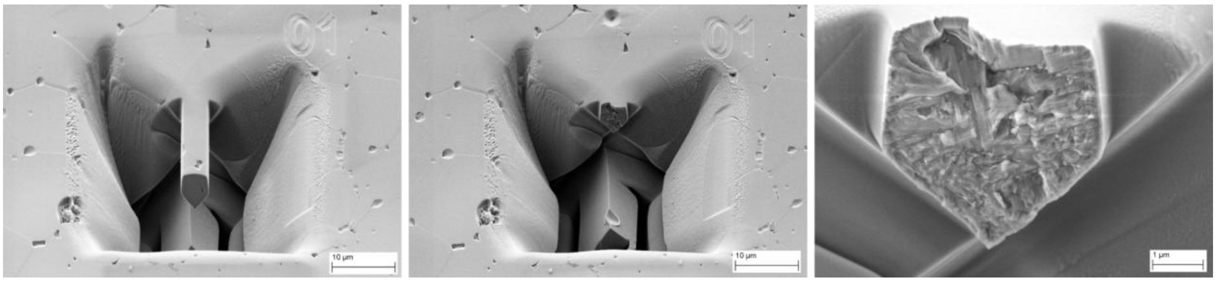

Figure 1: Micro-cantilever before and after bending test.

Table 2: Fracture strength of samples.

Sample	I	J	B	B1
Fracture strength	2.53	2.90	4.15	3.46

Keywords: CeO_2 doped zirconia, sintering, fracture strength

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COMPOSITE FILAMENTS CONTAINING COVALENT PHASES DEDICATED FOR REACTIVE SINTERING OF UHTC MATERIALS

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ABSTRACT

The study analysed the fabrication of ceramic polylactide (PLA)-based composite filaments for 3D printing by fused deposition (FDM) and the development of boron carbide (B_4C) suspensions for incremental fabrication by direct ink-writing (DIW). In the FDM process, boron carbide (B_4C) and silicon carbide (SiC) were incorporated into PLA at different weight fractions (1–40 wt% for B–C and 1–20 wt% for SiC) to produce filaments. Printing was achieved with SiC contents of up to 8% by weight using single extruded filaments and up to 20% by weight using double extruded filaments. Rheological tests showed that the low-ceramic-content filaments exhibited shear-thinning behaviour, whereas higher loadings led to Newtonian behaviour. Thermal analysis revealed a temperature range of 200-270 ° C. The DIW technique was used to produce ceramic green bodies using B–C paste. Formulations of B_4C paste were prepared by adjusting the powder content and organic compounds used to produce the paste. The DIW parameters were adjusted for each formulation. The produced bodies were characterised in terms of their density, morphology, and mechanical properties. The materials exhibited high thermal and mechanical resistance, making them suitable for applications such as wear-resistant parts, cutting tools, and protective armor.

Keywords: additive manufacturing, UHTC composites, ceramics paste.

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