2024 Annual Meeting of

BCERS

Belgian Ceramic Society asbl

23rd May 2024

ULiège

Program & Abstracts

PROGRAMME

8:30: **Registration - Posters hanging - Coffee and drinks**
9:00: Welcome

Invited Talk:

9:30: David Grossin - CIRIMAT-ENSIACET, Toulouse, France : "Additive manufacturing of bioceramics : Advances, challenges, and outlook"

2024 Student Speech Contest:

10:00: Damien Coibion - ULiège (GreenMat) : "Bovine bones as raw material for biomedical application by additive manufacturing"

10:15: Mia Kovač - KU Leuven : "Producing High-Density Monolithic Ceria stabilized zirconia Parts via Direct Ink Writing"

10:30: Muthusundar Kumar - UMons : "Cold Sintering Process - Progressing the consolidation of bio-active ceramics and polymer composites at low temperatures"

10:45: **Coffee Break and Poster Session**

Regular Presentations:

11:30: Dominique Hautcoeur – BCRC : "Enhancing additive manufacturing of ceramic parts by DLP: exploring supercritical extraction for improved debinding and mechanical properties"

11:50: Carmen Muñoz Ferreiro - Orbital Paradigm : "Key aspects of nanosheet incorporation to zirconia-based composites for increasing crack growth resistance"

12:10: Franklin Casarrubios – Umons : "Manufacture of cordierite parts by robocasting from recycled powders"

12:30: **Awards proclamation, Lunch and Poster session**

13:30: Bao Ta – Uliège : "Suitability tests for 3d printing with cementious mortars"

13:50: Stéphane Hocquet – BCRC : "Comparison of SLA/PAM calcium phosphate parts for bone tissue engineering"

14:10: Samanwitha Kolli - VITO/KU Leuven : "3D micro-extrusion and characterization of dense SiC structures"

14:30: Visit of the GreenMat laboratory from ULiège

15:00: **Closure**

POSTERS

1. Alberto Vettorel – KU Leuven : *"*Calcium Oxide stabilized zirconia: a new tough and strong zirconia for dental applications"
2. Berfu Göksel – KU Leuven : "Effect of Raster Pattern on the Defect Structure and Mechanical Strength of DIW ATZ Parts"
3. Carmen Muñoz-Ferreiro - Orbital Paradigm, Spain : "Additive manufacturing for reusable ceramic-based heatshields in atmospheric reentry space vehicles"
4. Damien Coibion – GREEnMat, ULiège : "Bovine bones as a source of bio-based hydroxyapatite for biomedical 3D printed implants"
5. Darya Farrokhnemoun – KU Leuven : "3D-printing of 12Ce-TZP zirconia ceramics"
6. Alexandre Marlier – UMons : "Synthesis of cordierite geopolymer composite for high temperature applications"
7. Hassen Dhifaoui – GREEnMat, ULiège : "Effect of Mesoporous TiO2 on Charge Dynamics of RbCsMAFA-Based Quadruple Cation Perovskite Solar Cells"
8. Junhui Zhang – KU Leuven : "Fabrication and characterization of laminated ZrO2-WC/ZrO2 structures"
9. Laura Manceriu – GREEnMat, ULiège : "A robust and simple method for the slip casting of Yttria-stabilized zirconia (YSZ) crucibles"
10. Oussama Zwein – UMons : "Enhancement of the preferentially orientated microstructure of polar STS glass ceramics"
11. Virendrakumar G. Deonikar – GREEnMat, ULiège : "Tailored Nanostructures of WO3: Unveiling Enhanced Electrochromic Performance through Structural and Morphological Optimization"
12. Zhenhong Xue – KU Leuven : "Anisotropic Shaped Zirconia Particles: Synthesis Mechanism and Large-batch Production"

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Member of ECERS

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2024 Student Speech Contest

Bovine bones as raw material for biomedical application by additive manufacturing

Damien Coibion, Dr. Nicolas Somers, Dr. Karl Traina, Dr. Audrey Schrijnemakers, Dr. Frédéric Boschini, Dr. Rudi Cloots

Bovine bones are composed of inorganic (60-70 w%) and organic (20-30 w%) parts, as well as a few percent of water (5-10 w%). The inorganic matrix is essentially composed of hydroxyapatite ($Ca_{10}(PO_4)_6(OH)_2$). It is therefore a compound of interest for biomedical applications, notably as a bone substitute. Several studies are looking into the possibility to dope hydroxyapatite with cations such as magnesium and sodium, or anionic groups such as carbonates, which are known to enhance the bioactivity of bone substitutes. However, all these doping agents are naturally occurring in bones. As a result, it could be interesting to valorize bovine bones from the food industry, which represents 130 billion kilograms of waste per year.

Currently, the conventional method to recover hydroxyapatite from a bone matrix is to burn it at high temperature to degrade the organic part into CO_2. However, the disadvantages of this method are that it is an energy-consuming process, producing CO_2, which is a greenhouse gas. Moreover, it degrades the carbonates naturally occurring in bones. Therefore, we have developed a method that reduces CO_2 emissions by degrading the organic matrix into water-soluble molecules, while preserving the carbonates.

In addition, we considered the possibility to use the recovered hydroxyapatite powder in additive manufacturing by stereolithography, to produce parts with a pore volume similar to cancellous bone (75 vol%).

Producing High-Density Monolithic Ceria stabilized zirconia Parts via Direct Ink Writing

Mia Kovač [1], Erin Koos[2], Bart Van Meerbeek[3], Jef Vleugels[1], Annabel Braem[1]

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Ceramic dental implants are gaining ground as a solution for tooth loss. While 3Y-TZP ceramics offer excellent strength and aesthetics, their vulnerability to low-temperature degradation (LTD) in the mouth is a concern. This research proposes ceria-stabilized zirconia (12Ce-TZP), known for its superior hydrothermal stability, as an alternative material solution.

Direct ink writing (DIW) is an AM process entailing micro-extrusion of highly concentrated zirconia pastes through a narrow nozzle deposited in a continuously spatially controlled filament in a layer wise fashion. To ensure an easy flow through the nozzle during printing and sufficient yield stress to support layer stacking after deposition, DIW pastes need to have an adequate shear-thinning flow and specific viscoelastic properties. Defect-free printing followed by a proper drying procedure and adequate debinding and sintering cycles allow fully dense monolithic prints to be obtained.

In this work, a water-based hydrogel (Pluronic F-127) was explored as a carrier for ceria-stabilized zirconia powder to manipulate the paste rheology while maintaining colloidal stability. The effects of the dispersion and mixing techniques on paste properties were investigated using particle size distribution, zeta potential, and rheology analysis. Scanning electron microscopy, micro-computed tomography, and density measurements were used to evaluate the printing defects in sintered Ce TZP samples. Biaxial flexure testing and hardness measurements were performed to assess the mechanical properties of high-density printed Ce-TZP discs.

Optimizing the mixing technique significantly enhanced the mechanical properties of the DIW zirconia. These improved properties rival or even surpass those observed in conventionally processed parts.

Cold Sintering Process – Progressing the consolidation of bio-active ceramics and polymer composites at low temperatures

Muthusundar Kumar[1,2], Mohamed Aymen Ben Achour[2], Marie Lasgorceix[2], Rosica Mincheva[1], Jean-Marie Raquez[1] and Anne Leriche[2]

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Abstract

Cold sintering process (CSP) is a non-conventional, low-energy sintering technique that promotes the densification of ceramics in the presence of transient liquids under low temperatures (≤300°C) and pressures (≤500 MPa). This low-temperature consolidation additionally provides a new strategy for co-sintering ceramic and polymers into a single system, which is not feasible through conventional methods. Exploiting the advantages of cold sintering, this investigation has aimed to explore the fundamentals of consolidating the hydroxyapatite (HA) ceramics at the nanoscale and the co-integration of HA/polylactic acid (PLA) based composite for bone regeneration applications. The importance of liquid phase chemistry in cold sintering of HA was assessed using water, acetic acid, and phosphoric acid as liquids. The changes in relative density were observed concerning the nature of liquid/ionic concentrations (0.5M, 1.0M, & 2M). The cold-sintered HA samples were characterized with relevant techniques to unveil the chemical and microstructural features to understand the impact of liquids. In the case of composites, the influence of pressure, and different compatibilizers on the homogeneous integration of HA/PLA composite was examined. Interfacial bonding, dispersion, and co-sintering aspects were further evaluated with relevant techniques. Eventually, this study contributes to underscore the critical fundamental knowledge on developing dense HA ceramics and bio-active polymer composites.

Regular Presentations

Enhancing additive manufacturing of ceramic parts by DLP: exploring supercritical extraction for improved debinding and mechanical properties

D. Hautcoeura, G. Bistera, L. Boileta, N. Nurmib, E. Levänenb, E. J. Frankbergb

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Stereolithography is a proven additive manufacturing technique used for printing ceramic parts in three dimensions. However, it requires the use of organic compounds for formulating feedstocks that need to be removed before sintering to achieve dense ceramic parts. When parts are small or porous (such as scaffolds), this debinding process is typically carried out thermally, at a slow heating rate, to allow gases resulting from the decomposition of the organic material to escape. Despite relatively long debinding times, obtaining large defect-free parts remains a challenge. To address this issue, pre-debinding cycles or specific treatments could be employed before the thermal cycle.
Previous studies have suggested that using supercritical extraction with CO2 (SC) as a pre-debinding step might be advantageous compared to conventional water debinding for SLA-printed parts. The influence of both soaking time and specimen size has been evaluated by measuring mass loss and conducting TGA analysis. Considering soaking time and extraction time (limited to 6 hours due to equipment constraints), no preferential pre-debinding treatment has been clearly identified except for surface defects. A combined cycle (SC + water debinding) has been investigated and assessed for improving processing time, part integrity, and mechanical properties (3-point bending).
It is well known that the mechanical properties of ceramics depend strongly on the finish of the surface. The mechanical properties are determined on cut and polished samples, unlike the final products. In the case of components obtained by additive manufacturing (especially Stereolithography-SLA), post-processing and finishing is a difficult task which cannot usually be performed for very complex parts.
The Belgian Ceramic Research Centre (CRIBC) and Tampere University (TAU) have initiated a collaborative project on this topic. The project will focus on improving post-processing and characteristics of 3D printed ceramic oxide samples. The possibility of reducing debinding time and enhancing the mechanical strength of sintered parts will be evaluated by modifying post-printing treatments (cleaning and debinding). Two approaches have been selected to achieve these goals: proper selection of suitable monomers for paste preparation and an improved chemical debinding process involving supercritical extraction with CO2.

The European Regional Development Fund (ERDF) and Wallonia, are gratefully acknowledged for their financial support to these research projects CERAMTOP and CERAMPLUS “Iawatha” in the frame of the “Transition programme.” In addition, we acknowledge financial support from Business Finland within the project cerAM.

Keywords:
Stereolithography, debinding, supercritical extraction CO_2

Key aspects of nanosheet incorporation to zirconia-based composites for increasing crack growth resistance

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Materials are a key factor in the progress of the environmental, medical, space or energy industries, which is often limited by the material properties. Thus, new composite materials that meet the multifunctional properties required in these fields are being developed. Ceramics are particularly important because of their high strength and hardness, temperature resistance, chemical inertness and corrosion resistance, making them suitable as matrices for new composite materials. However, their fragility makes them still unreliable for many structural applications.

It was expected that by incorporating graphene-based nanomaterials (GBN) into these high-performance ceramics, composites would achieve greater structural stability, due to the outstanding mechanical properties attributed to graphene. More recently, inorganic graphene analogues (IGA) have also emerged as promising reinforcement. In particular, 2D hexagonal boron nitride (2D-hBN) rapidly stood up because of its mechanical properties comparable to those of graphene, its electrical insulating capacity, and its superior chemical stability up to 800-1000 °C. Thus, new functionalities of the composites are expected to be achieved by incorporating these IGA. However, a review of recent studies did not show a clear trend of the GBN enhancing abilities in ceramic matrix composites, and only few studies addressed the mechanical performance of ceramic composites incorporating 2D-hBN. Several authors have related the properties of the composites to the size, specific surface area and oxidation rate of the graphene nanostructures, but the factors promoting mechanical enhancement are still unclear. Therefore, a deeper understanding of the microstructure-to-properties relationship would allow to enhance and tailor the composite properties for precise applications.

In this work, the role of 2D reinforcements (graphene or 2D-hBN nanosheets) in the crack propagation resistance of 3 mol% yttria stabilized zirconia (3Y-TZP) has been addressed and compared, focusing on the nanosheet content, morphological characteristics, oxidation rate and dispersion throughout the matrix. To that end, several starting nanostructures differing in size and chemical nature, were used to prepare the composite powders, which were then homogenized with the 3Y-TZP powder either by bath sonication or planetary ball milling. Fully dense composite materials were consolidated by Spark Plasma Sintering at 1250 °C with a 75 MPa uniaxial pressure in vacuum. The fracture toughness and crack-growth resistance (R-curve) of the resulting composites were evaluated on single edge V notched beams by 3- or 4- point bending tests. The results were related to their microstructural features - analysed by scanning and transmission electron microscopy, X-ray diffraction and Raman spectroscopy -, revealing the key aspects promoting a rising R-curve.

Manufacture of cordierite parts by robocasting from recycled powders

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Abstract

Advanced ceramics exhibit remarkable properties, such as high mechanical strength, thermal conductivity, or wear resistance. Therefore, they are used as critical components for specific applications in the fields of aerospace, automotive, energy production or cutting tools. Silicate ceramics usually present less efficient properties. However, they can be an economically advantageous alternative to technical ceramics in many applications operating at room or moderate temperatures (< 1000 °C). Moreover, regarding the environmental aspect, silicate ceramics are processed from natural abundant mineral resources, show a high recyclability, and require moderate sintering temperatures. One potential technical use of silicate ceramics is the manufacture of gas cleaning devices, such as catalyst substrates or particulate filters, as it is the case for cordierite in the automotive industry. The expansion of the use of these devices in other sectors goes through the development of low-cost flexible manufacturing technologies and the improvement of the efficiency of the devices through the design of components with complex architectures.

On that purpose, the aim of this study is to demonstrate the possibility to manufacture complex cordierite parts by robocasting. Moreover, this work focuses on the use of an already-formed cordierite powder recycled from industrial wastes. First, the sinterability of recycled cordierite is studied and compared with a commercial cordierite. Next, the preparation and characterization of a stable ink of cordierite powder with a suitable rheological behaviour for the robocasting process is achieved. Finally, a comparison is made, regarding the final properties (density/porosity, coefficient of thermal expansion and Young's modulus), between printed parts and parts obtained by uniaxial pressing, after sintering at 1300 °C for 4 hours.

SUITABILITY TESTS FOR 3D PRINTING WITH CEMENTIOUS MORTARS

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Additive manufacturing has found widespread application across various industrial sectors including the construction industry, which presents significant potential. Nonetheless, the utilization of materials for 3D-printed construction encounters challenges due to their unique properties, particularly their setting time. Hence, a thorough investigation into the rheology of 3D-printed mortar becomes imperative.

The material used for this process must exhibit a delicate balance, being sufficiently fluid to facilitate smooth transportation through the pump pipe while also possessing the requisite viscosity to support multiple layers when applied. Furthermore, the 3D-printed filament must feature a smooth surface and continuity to ensure optimal cohesion between layers. To assess these properties, three key criteria are established: pumpability, extrudability, and buildability.

This study aims to investigate the methodology for measuring these characteristics. Two complementary testing methods are employed: simple standard tests for construction materials providing an estimation of rheological characteristics and rheometer tests ensuring precise and reliable measurements of yield stress and viscosity. The primary objective lies in identifying a suitable test methodology for assessing the rheological traits of materials on the construction site. Various tests, including the flow table test, fall cone test, V funnel test and pistol test, are compared to determine the optimal approach for predicting the suitability of cementitious material for 3D printing. Experimental assessments conducted by means of the RheoCAD rheometer are indispensable for comprehending material behaviors across diverse testing conditions.

Comparison of SLA/PAM calcium phosphate parts for bone tissue engineering

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Natural bone is an organ of the human body capable of regenerating in the event of injury or fracture. But when the bone defect to be repaired is too large (greater than 1mm), it is necessary to use a bone substitute, of which the current gold standard is the autologous bone graft. However, this type of graft has major shortcomings: it is available in limited quantities, requires a second surgical operation (thus increasing time and health costs, as well as the risk of infection) with general anaesthesia of the patient, and is in any case not an option for large defects.
Calcium phosphate bioceramics have several advantages: they are highly biocompatible, they have osteo-inductive and osteo-conductive properties, they are chemically similar to natural bone tissue and, if necessary, they can be bioresorbable. To become an 'ideal' bone substitute, however, they must meet other criteria: they must be able to be shaped with a complex external geometry and have a micro- and macro-porous architecture that encourages cell recolonisation.
In this work, two additive manufacturing technologies are compared, demonstrating their interest in the manufacture of macroporous calcium phosphate-based structures that best meet all the criteria of the "ideal bone substitute". These two technologies are Digital Light Processing (DLP) and Pellets Additive Manufacturing (PAM). The presentation highlights the advantages, shortcomings and opportunities for improvement/development of the two technologies, as well as an initial in vivo characterisation result.

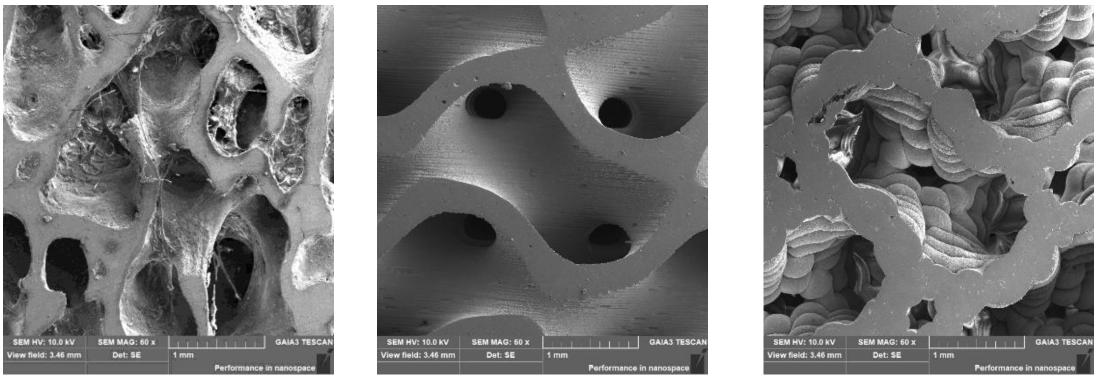

Figure : SEM pictures of (on the left) a natural bone tissue, (in the middle) a gyroidal microporous structure obtained by DLP and (on the right), a gyroidal microporous structure obtained by PAM.

3D micro-extrusion and characterization of dense SiC structures

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Due to its exceptional thermophysical characteristics, Silicon carbide (SiC) is recognized as a highly promising material for thermal management devices. However, traditional powder processing methods impose limitations on design flexibility, consequently constraining the ultimate performance of components. Addressing these challenges, additive manufacturing (AM) emerges as a viable solution, particularly for applications requiring intricate geometries, thereby proving to be an excellent approach for crafting high-performance SiC thermal management devices. 3D micro-extrusion is one of the most versatile AM technologies that offers processability of a wide range of materials, including SiC.

In 3D micro-extrusion, the powder-loaded paste is extruded through a fine nozzle and deposited layer-by-layer followed by debinding and pressureless sintering. The objective of this study is to establish a comprehensive processing route for 3D micro-extrusion of highly dense silicon carbide structures with exceptional functional and mechanical properties. The work presents a detailed analysis of feedstock paste preparation including characteristics of the starting powders, the optimal amount of sintering aids, binders, and additives. Secondly, shaping of green parts using 3D micro-extrusion with minimal printing defects. Lastly, the densification of the printed components by liquid phase sintering using an optimized sintering cycle. The resulting parts undergo a thorough characterization, evaluating structural, microstructural, and mechanical properties, and dimensional accuracy. The technology enabled the fabrication of highly dense SiC parts with a relative density of 97% TD while exhibiting mechanical properties i.e. hardness of 22 ± 1 GPa, and indentation toughness of ~6.4 MPa·m$^{1/2}$ which are on par with conventionally manufactured SiC parts.

Posters

"Calcium Oxide stabilized zirconia: a new tough and strong zirconia for dental applications"

Alberto Vettorel [1,2], Jef Vleugels [1], Bart Van Merbeek [2], Fei Zhang [1,2]

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Zirconia are characterized by excellent mechanical and chemical properties along with high aesthetics and biocompatibility, which make them interesting to make various dental prostheses. Although different types of zirconia ceramics stabilized by yttria or ceria have been widely studied or introduced in the dental market, there are still lack of zirconia that can combine high toughness, strength, damage tolerance and translucency. In this work, the performances of a novel calcium oxide (4.5 mol %) stabilized zirconia (Ca-TZP) were studied. Ceramics were prepared by pressing and sintering approach at three sintering temperature (1200°C, 1250°C and 1300°C). Key mechanical and physical properties including density, hardness, biaxial strength, grain size distribution and phase characterization were characterized.

The results showed that all the batches had a density above 99% of theoretical density. A sub-micron grain size distribution was observed in every group and the average grain size increased as a function of the sintering temperature. Rietveld refinement showed that the monoclinic phase after sintering decreased as a function of the sintering temperature passing from 10 wt% for zirconia sintered at 1200°C to 3% for zirconia sintered at 1300°C. Zirconia sintered at 1200°C was also characterized by the highest cubic phase which was indeed confirmed by the large cubic grains observed during SEM imaging. Due to the high toughness of Ca-TZP, no cracks were observed after Vickers hardness indentation while using loads up to 300 N. Zirconia sintered at all three temperatures showed transformation bands after indentation. In particular, Ca-TZP sintered at 1200°C was characterized by thicker and spread out bands while samples sintered at 1250-1300°C had thinner and less spread transformation bands. Regarding the biaxial strength, Ca-TZP sintered at 1250°C had both the highest Weibull modulus of 7.8 and characteristic strength of 1502 MPa. Interestingly, it was possible to observe transformation bands after failure only in samples sintered at 1300°C.

Effect of Raster Pattern on the Defect Structure and Mechanical Strength of DIW ATZ Parts

Berfu Göksel

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Direct Ink Writing (DIW) is a paste-based additive manufacturing technique where objects are built layer by layer by extruding a filament through a narrow nozzle under applied pressure, enabling the production of technical ceramics with high density. The resulting green parts require appropriate drying, debinding, and sintering processes.

Critical to the mechanical properties is the selection of printing parameters, given that it is not possible to remove printing defects in subsequent processing stages. The choice of raster pattern is crucial for achieving high-density parts with minimal defects, considering the variation in defect size and shape across different patterns. Therefore, raster pattern optimization is necessary to attain the desired mechanical strength.

This study focuses on optimizing the raster pattern through mechanical characterization and fracture analysis of high-density alumina-toughened zirconia (ATZ) parts produced with optimized paste and printing parameters. Through systematic investigation, this research aims to explain the relationship between raster pattern, defect structure, and mechanical strength, thereby advancing the understanding and optimization of DIW-based ceramic manufacturing processes.

Additive manufacturing for reusable ceramic-based heatshields in atmospheric reentry space vehicles

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In recent years the need for reusable space vehicles has gained interest to make this technology more affordable and decrease waste production. However, the reusability of the vehicle is restricted because of the extremely high temperatures reached at its surface during reentry into the Earth's atmosphere (up to 2000 ºC in some parts). In fact, a heatshield or thermal protection system (TPS) is necessary to protect the vehicle structure and its interior. The different TPS developed may be classified in two main groups: ablative or reusable TPS. The latter consists of a heatshield made from materials that withstand those temperatures. Ceramic based materials, such as SiC composites or silica fibers, are often proposed as reusable TPS due to their lightweight and their thermal and chemical stability. However, two main challenges still need to be faced to reduce manufacturing time and costs:

1. The difficulty of machining curved or complex parts.
2. The attachment to the vehicle surface, due to the uncoupled thermal expansion with the vehicle structure.

To overcome these difficulties through conventional procedures, TPS are manufactured in polygonal flat tiles which need to be afterward assembled with adhesives and further isolation. The implementation of additive manufacturing for this application would allow to produce more complex parts or even monolithic heatshields for small vehicles that could be mechanically attached to the surface.

In this sense, our work is devoted to producing test samples that can be mechanically mounted in a layered structured from SiO_2 and Al_2O_3 fibers and SiC. Different manufacturing technologies are proposed, among which slip casting or additive manufacturing, to compare their results and determine whether additive manufacturing is suitable for this new application.

Bovine bones as a source of bio-based hydroxyapatite for biomedical 3D printed implants

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The advent of 3D printing has offered the possibility to produce custom implants based on medical images. Among the materials used for bone grafts, hydroxyapatite (HAP) is increasingly being studied for its mechanical properties and biocompatibility. Indeed, HAP is the mineral component of bone.

A large number of studies have considered the possibility of synthesizing HAP doped with various ions known to promote osteogenesis and thus improving osseointegration. However, the HAP naturally in bones already contains these ions. Therefore, the present study considers the possibility of extracting HAP from bovine bones, which are waste products of food industry, in order to be used in additive manufacturing by stereolithography.

3D-printing of 12Ce-TZP zirconia ceramics

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Abstract

Various factors influencing the paste composition and printing parameters for the direct ink writing (DIW) process were investigated allowing to successfully 3D-print a 12 mol% CeO_2-stabilised ZrO_2 nanopowder by means of combined Direct Ink Writing and UV-Curing. Paste optimization containing ceramic powder, dispersant, monomers, diluents and photo-initiator was done by speedmixing and supported by rheological measurements. The DIW and UV-curing parameters were optimized for the printing of disc shaped samples, which were thermally debinded and sintered. The layer thickness has the most significant role in inter-filament gap filling and curing of paste. Moreover, the optimal UV-light focal point that ensures an efficient and uniform curing of the printed layers was experimentally determined.

Synthesis of cordierite geopolymer composite for high temperature applications

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This work is part of a research project aiming at producing ceramic-like materials of high thermomechanical properties up to a temperature of 1000 °C, without the need for an initial sintering. Cordierite – metakaolin-based geopolymer composites have been synthesized. The alkalinization has been made by mixing the metakaolin with a potassium silicate solution. Two sources of metakaolin are used to observe the influence of the amorphous content and the presence of quartz. The consolidation has been made at a temperature below 100 °C. The cordierite powder comes from the recycling of automotive industrial waste. The microstructure and properties of the composite are characterized after the geopolymerization reaction and after heat treatment at 1000 °C. After geopolymerization, the geopolymer is in an amorphous state. After the heat treatment, the geopolymer crystallize in leucite. Furthermore, a significant part of the potassium seems to diffuse in the structure of the cordierite, leading to the stabilization of the indialite, the hexagonal structure of the cordierite at high temperature. The material exhibits a coefficient of thermal expansion of 2.5 10-6 K -1 . With a porosity of 35% and an elastic modulus of 16.7 GPa, this composite could be interesting to produce substrate for catalytic applications or filters.

Effect of Mesoporous TiO_2 on Charge Dynamics of RbCsMAFA-Based Quadruple Cation Perovskite Solar Cells

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Abstract

The electron transport layer of mesoporous TiO_2 (mp-TiO_2) plays a pivotal role in the performance and development of perovskite solar cells (PSCs). In order to understand the charge kinetics occurring in the bulk and at the selective interfaces, we studied quadruple cation RbCsMAFA-based PSCs deposited by spin-coating, with and without mp-TiO_2. The mp-TiO_2-free PSCs demonstrated a power conversion efficiency (PCE) of 12.10% with low short circuit current (J_{sc}) whereas a higher PCE of 16.12% with increment in J_{sc} was measured for the PSCs with mp-TiO_2. The interfacial charge kinetics of PSCs was investigated by bias-dependent electrochemical impedance spectroscopy (EIS). The PSCs fabricated with mp-TiO_2 showed a lower recombination and higher charge carrier lifetime. The results demonstrated an intimate correlation between the charge recombination and the charge interaction at the interface of electron transport layer and perovskite. Further, by performing Mott-Schottky measurements, the built-in potential and doping density of immobile ions were evaluated.

KEYWORDS: mesoporous TiO_2, quadruple cation RbCsMAFA perovskite solar cells, impedance spectroscopy, charge recombination, Mott-Schottky.

Fabrication and characterization of laminated ZrO_2-WC/ZrO_2 structures

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Zirconia (ZrO_2)-based ceramics are one of the most important engineering and structural materials due to their excellent physical and mechanical properties. However, their inherent brittleness can induce catastrophic failure, limiting their applications. Ceramic laminates have gained attention to increase the crack propagation resistance and fracture energy. Additionally, ceramic laminates can integrate a conductive phase and provide potential functionalities, such as self-monitor crack extensions. In this study, we present the strategy to prepare laminate ceramics by combining a conductive phase (ZrO_2-WC) with zirconia ceramics. ZrO_2-WC and zirconia laminated composites are prepared by alternately stacking ZrO_2-WC and zirconia powder and then sintering using hot-pressing. Laminated composites have shown thermal residual stress after sintering as a result of the different coefficients of thermal expansion. The presence of compressive residual stresses in ZrO_2-WC layers can deflect cracks propagating through the layers and even arrest the crack at the interface.

Keywords: Laminated structures, ZrO_2-WC, Zirconia, Hot-pressing sintering

A robust and simple method for the slip casting of Yttria-stabilized zirconia (YSZ) crucibles

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This study was aimed to develop a robust slip casting procedure for the fabrication of yttria stabilized zirconia (YSZ) crucibles. The commercially available YSZ powder was directly used to prepare an aqueous slurry to which a dispersant was added. The mixture homogenisation and powder de-agglomeration were realized in a single step using ultrasound treatment. The slurry was casted in plaster moulds and the recovered green pieces were sintered at 1450 °C.

We studied the effect of the solid loading on the rheological properties and correlated them with the final parts density before and after sintering.

Within few minutes of ultrasound treatment particle de-agglomeration was attained with minimal material loss. The slip casting method was proven to be very robust with crucible final density exceeding 99 % for dry mass contents in the range of 73- 81%.

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Enhancement of the preferentially orientated microstructure of polar STS glass ceramics

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Abstract

The crystallisation of Strontium titanium silicate $Sr_2TiSi_2O_8$ (STS) in a parent glass of suitable composition, can lead to polar glass-ceramics that can be used for the design of surface acoustic wave (SAW) devices able to work up to temperatures around 1000°C [1-2]. However, to obtain a high electromechanical response of material, the polarisation, that results of the preferentially orientated growth of the STS crystals, must be high and constant over a thickness depending on the work frequency.

The objective of this work is to enhance the performance of an STS glass-ceramic family that has already demonstrate its potential as high temperature SAW substate. On that purpose we investigated the main parameters influencing the crystallization and microstructure from parent glasses of compositions STS + x ($1.3SiO_2 – 0.2K_2O – 0.1Al_2O_3$), where x = 0.75, 1, and 1.5, representing varying amounts of residual glass.

A comprehensive study of the glass crystallization was conducted using Differential Scanning Calorimetry, X-Ray Diffraction, Scanning Electron Microscopy, and Optical Microscopy. Based on the study results, heat treatments were applied to the glass.

The influence of crystallization temperature and glass surface roughness on the oriented surface crystallization mechanism was examined. It was demonstrated that employing a specific thermal cycle promotes a high orientation of the (002) STS planes parallel to the substrate's surface to a depth of at least 1 mm, regardless of the quantity of residual glass.

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Tailored Nanostructures of WO_3: Unveiling Enhanced Electrochromic Performance through Structural and Morphological Optimization

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Tungsten oxide (WO_3) nanostructures have gained significant attention for their potential applications in electrochromic devices due to their unique structural, morphological, and optoelectronic properties. In this study, we present a comprehensive investigation into the tailored synthesis of WO_3 nanostructures with controlled structural features and morphological properties. The hydrothermal method was used to synthesize WO_3 with different strategies, to engineer the nanostructure growth and morphology of WO_3. Characterization techniques such as scanning electron microscopy (SEM), X-ray diffraction (XRD) are employed to elucidate the structural and morphological properties of the synthesized nanostructures. Moreover, electrochemical analysis is used to evaluate the electrochromic performances of the WO_3 nanostructures to get a deeper understanding of their optical modulation, coloration/bleaching capabilities, etc. Overall, our findings highlight the importance of tuning the structural and morphological properties of WO_3 nanostructures for achieving enhanced electrochromic performance, hence revealing the way for their potential usage in displays, smart windows, printable QR codes and other optoelectronic devices.

Keywords: Tungsten oxide (WO_3); Structural properties; Morphological properties; Electrochromic performance.

Acknowledgment: Project PECLABEL was selected in the Joint Transnational Call 2022 of M-ERA.NET 3, which is an EU-funded network of about 49 funding organisations (Horizon 2020 grant agreement No 958174). The project is funded by the :

- Fonds de la Recherche Scientifique – FNRS, Belgium, under Grant n°R8002.23,
- Agence Nationale de la Recherche – ANR, France, under Grant n°ANR-23-MER3-004-01,
- Agencia Estatal Investigación - AEI, Spain, under Grant PCI2023-143366 funded by MCIN/AEI/10.13039/501100011033 and, the European Union NextGenerationEU/PRTR,
- Business Finland, Finland, under grants n°1956/31/2023 & 2888/31/2023.

Anisotropic Shaped Zirconia Particles: Synthesis Mechanism and Large-batch Production

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Anisotropic ceramic particles play a key role in developing bio-inspired ceramic composites that combine high strength with high toughness. However, developing a simple and efficient method to produce these anisotropic ceramic particles presents challenges. Traditional synthesis methods often require templates or surfactants to achieve anisotropic morphologies, which complicates the process due to the subsequent removal of these additives. In this study, we synthesized rod-shaped ZrO_2 particles using a facile and reproducible hydrothermal treatment, eliminating the need for templates and surfactants. We elucidated the formation mechanism that results in a pure *m*-ZrO_2 phase. Additionally, this synthesis method shows promise for scaling up.