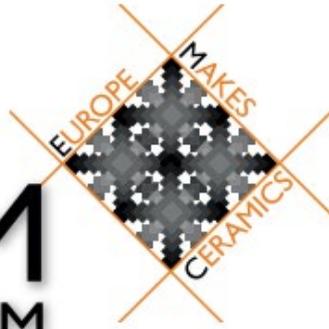


2023
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Keynote Speakers

Lithography-based ceramic manufacturing in the field of modern medical engineering

Authors

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Topic Areas

Keynote speakers

Abstract

Additive manufacturing (AM) has become an integral part of modern medical engineering. Compared to conventional shaping methods of ceramics, AM approaches offer the possibilities to create highly complex ceramic products like patient-specific medical implants. Lithography-based ceramic manufacturing (LCM), a vat polymerization technique with selective exposure of photocurable ceramic suspensions to create green parts in a layer-wise fashion, represents one of these methods. This work focuses on three promising materials for LCM in the field of medical applications, based on alumina-toughened zirconia (ATZ), hydroxyapatite (HA) and tricalcium phosphate (TCP). In the context of medical implants, a differentiation is made between permanent implants for load bearing applications and bioresorbable and bioactive to promote natural bone healing. ATZ is a very promising candidate for permanent implants as it combines the best properties of alumina and zirconia. The advantages of ATZ are good biocompatibility, chemical inertness, high strength, fracture toughness, hardness as well as good abrasion resistance. The feasibility of massive components from ATZ with a wall-thickness up to 20 mm was demonstrated, paving the way to manufacture dental implants and permanent medical inserts like femoral head implants in an industrial scale using LCM. Hot isostatic pressing of ATZ parts led to significantly increased mechanical properties with regard to bending strength, achieving values up to 1100 MPa in a 4-point bending-setup. HA and TCP on the other hand exhibit very good biocompatibility and their porosity and strength can be tuned over a broad range, allowing the fabrication of scaffolds that can be resorbed and remodeled into bone tissue upon implantation. In this context the production of bioactive and bioresorbable implants with defined porosity and patient-specific freeform shape to mimic natural bone was demonstrated, showing that LCM is an interesting alternative to conventional processing techniques for the medical industry.

The path from research to the industry standard

Authors

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Topic Areas

Keynote speakers

Abstract

Processes that are considered to work well in research are far from being running industrial processes. Using the LIS (Laser Induced Slipcasting) and LSD (Layerwise Slurry Deposition) processes as examples, the path from the research stage to the industrial process is shown. Where are the hurdles? What is the difference between a research system and an industrial system? What criteria must be met around the printing process. It does not matter how good a process is at the research stage, if it is not industrialised with full commitment, sooner or later it will disappear in the drawer. We have taken up the challenge and made the LSD and LIS process available to industry.

How Additive Manufacturing is becoming a game-changer for the production of clean hydrogen

Authors

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Topic Areas

Keynote speakers

Abstract

Additive Manufacturing has been around for decades now, but its innovation is accelerating continuously, pushing the boundaries to open up new ideas to explore for industrials as well as researchers.

In a European collaborative program starting this year called HYP3D, AM is helping to improve the standards of hydrogen production with pressurised 3D printed SOEC stacks. The challenges that come from this technology lie in 3 main domains : production, storage, and supply chain. Here, the program is focusing on improving the clean production of hydrogen. Currently, the electrode supported cells are flat in shape, produced by traditional shaping methods, and the metallic interconnects are voluminous and complex in shape.

3DCeram's SLA technology is giving the possibility to print thin corrugated ceramic cells in 8YSZ, increasing the surface area by 60% which in turn enhances the performance of the cells. It's also enabling the cells to have more functionality, reducing the size of the interconnects and therefore the stack as a whole.

This is another example of how 3D printing is bringing to life more disruptive designs, helping to enhance system performances, and creating new avenues to explore in research and development.

Material Jetting, Binder Jetting and Powder Bed Fusion

Development potential of Direct Inkjet Printing

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Topic Areas

Material Jetting, Binder Jetting and Powder Bed Fusion

Abstract

Rauschert GmbH is not only known for high-quality pressed, extruded as well as injection-molded components, but is also constantly looking for new application and development potentials. One current development approach is additive manufacturing with a focus on Direct Inkjet Printing, known as DIP. DIP is a suspension-based process that was developed at the Institute of Mineral Engineering at RWTH Aachen University and can be assigned to the material jetting category. The functional principle is based on the dropwise application of particle-filled ceramic suspensions on a substrate by thermal print heads. As a consequence of such an application method, DIP offers a high degree of design freedom. Furthermore, the DIP is characterized by a high precision and resolution of the printed structures. However, a requirement for achieving precise structures is compliance with defined, process-specific parameters, such as the particle size or viscosity of the suspension, otherwise clogging of the print head nozzles or uncontrolled suspension discharge could occur. Process-wise, the DIP permits simultaneous printing of four different suspensions, allowing not only material but also color gradients to be adjusted within a printed structure. Current materials are Y₂O₃-stabilized ZrO₂ and Al₂O₃. At the same time, the application of a so-called support suspension enables the production of complex structures, e.g. overhangs or undercuts. After the structures have been printed, the carbon black-based support suspension is thermally decomposed during the sintering process, resulting in final purely ceramic structures. Due to these numerous advantages, DIP can be used for a wide range of applications, e.g. for machine components as well as the medical and electronics sectors. In view of medical applications, a development project has been initiated between Rauschert GmbH and RWTH Aachen University as well as other project partners, whose aim is to transfer this technology from the lab to an industrial environment.

Binder Jetting 3D Printing of Potassium Sodium Niobate: sintering procedure optimisation

Authors

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Topic Areas

Material Jetting, Binder Jetting and Powder Bed Fusion

Abstract

Binder Jetting (BJT) has demonstrated potential for producing piezoelectric devices for underwater applications using ceramic powders. The most used piezoceramic (PZT) is being replaced by lead-free alternatives, due to environmental and health concerns associated with the use of this element. One of the most popular lead-free alternatives is sodium potassium niobate ($(K_{0.5}Na_{0.5})NbO_3$ / KNN), which has good piezoelectric properties, a high Curie temperature, and a high acoustic impedance. However, the sintering of this material presents challenges, due to the narrow sintering range caused by late densification processes and alkali evaporation. Additionally, Abnormal Grain Growth (AGG) can have a detrimental effect on piezoelectric and mechanical properties. There is a lack of uniformity in the literature regarding optimal sintering conditions, including parameters, atmosphere, and whether to use an atmospheric powder. Obtaining dense pieces through traditional shaping methods is already difficult, and it is even more challenging for pieces obtained through BJT that have a porous green body with low density. The aim of this study is trying to research and standardize the optimal sintering conditions for binder jetted parts, by reviewing existing literature and using a Design of Experiment (DoE) to weigh the different conditions and gain a clearer understanding of the processes related to the densification of KNN. So far, the relative density values obtained after sintering range from 67% to 80%, with an average value of 73% depending on the printing conditions. With the optimisation of the sintering procedure, the aim is to achieve even higher relative density values while keeping the grain size small so as not to degrade the functional properties.

Drying mechanism and kinetics in solvent evaporation of the cantilever method

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Topic Areas

Material Jetting, Binder Jetting and Powder Bed Fusion

Abstract

Layerwise slurry deposition (LSD) is one of the newer additive manufacturing methods, in which the powder of the broadly used powder bed binder jetting is dispersed into a slurry.

This slurry is then evenly deposited to form layers which can be printed on. With this manufacturing step, normally found in slip casting, a new problem of capillary stresses between particles develops which can lead to the formulation of cracks, pores, and bubbles. These defects will result in a lower performance in the product or to the unfeasibility of the whole process. A better understanding of the drying mechanisms and the formation of cracks in the green body is an important field in material science and in LSD-Technology.

In this work, the LSD-Technology its potential problems in the deposition step, as well as the cantilever deflection method are discussed. It will focus in particular on the cantilever method and will give a closer look into the theory of drying, the influence of kinetics in solvent evaporation and the intensity of in-plane stresses developed during drying.

Porous alumina manufacturing by binder jetting 3D printing

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Topic Areas

Material Jetting, Binder Jetting and Powder Bed Fusion

Abstract

Production of porous ceramics is of great interest for a series of applications, as catalyst supports, filtering devices, biomedical implants, EMI shields and thermal insulators. Conventional manufacturing routes (incomplete sintering, replica method, sacrificial template and direct foaming) suffer from limited shaping capabilities and could be expanded by implementing additive manufacturing techniques, especially binder jetting. In this work, this technique is employed to produce alumina components.

The study is developed in three steps: powder characterisation, printing capabilities assessment and sintering development.

Two sets of particulate feedstock were considered: the first with gas atomised particles and the second with spray-dried granules. Particle size distributions and morphologies are determined by granulation and SEM, then correlated to the flowability quantified by rheometry and dispensing rate within the printing system. Powders with high true density and minimal sub-micrometric fractions provide optimal performance due to the limited cohesiveness of particles.

Suited feedstocks were printed with a constant layer thickness of 50 μm and binder saturation varying in between 55% and 90%, while the remaining parameters were adapted to each flowability range. Green density below 25% was obtained with granules, while values went up to 50% with dense particles: the difference arises from the intrinsic porosity of granules that is summed to the voids from particle packing in each layer.

Sintering was evaluated at intermediate and high temperature to determine the onset of diffusive mechanism with respect to the primary particles size. Granulated powders provide larger densification (+20/30%) which is more limited for atomised particles. In the former case, volume diffusive mechanisms leads to intragranular densification, while consolidation by necking among particles occurs in all cases. Porosity is evaluated by microscopy and mercury intrusion porosimetry, revealing the formation of multimodal void size distribution with oriented development along the printing layer surface.

Material extrusion with inks and pastes I

Colloidal route towards novel sodium superionic conductor electrolyte (NASICON) with complex 3D structures fabricated by Direct ink writing (DIW)

Authors

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Topic Areas

Material extrusion with inks and pastes I

Abstract

Towards a sustainable energy model, safer new generation energy storage devices with high energy density and power are needed. In this sense, the improvement in terms of efficiency and sustainability has led to the interest in solid-state batteries (SSBs). Lately, sodium-ion batteries (SIBs) due to their abundance, low cost and improvements in terms of fast sodium-ion conductor solid electrolytes (SCSEs) have become an emerging alternative. Among all the SCSEs NASICON type electrolyte is one of the most well-known electrolytes, being widely developed in terms of synthesis and materials. However, the processing and manufacturing of these electrolytes have gone almost unnoticed, without considering that well-designed structures of electrodes/electrolytes are the bridge toward turning advanced energy materials into high-performance devices. This work presents the fabrication of complex 3D NASICON electrolytes by direct ink writing (DIW). Through a colloidal route fine NASICON phase powder with high pureness is obtained, empowering the obtaining of NASICON printed electrolytes in one step fabrication process. By means of the development of the ink, a dense electrolyte layer, acting as an ionic conductor and separator, was inserted between two complex porous patterns with a total height below 1.2mm. Further, the densification of the 3D electrolyte was analysed to improve the ionic conductivity and, therefore, the electrochemical performance of the electrolyte. Thus, fast ion conductor NASICON solid electrolyte with shorter diffusion pathways and have larger interfacial surface areas between electrode/electrolyte was obtained, enhancing the electrochemical properties by 3D layer-by-layer design.

Field-assisted sintering of BaTiO₃ ceramics fabricated by Direct Ink writing (DIW)

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Topic Areas

Material extrusion with inks and pastes I

Abstract

Conventionally, ceramic components are manufactured by sintering powder compacts produced by pressing, casting, or injection molding; however, structures with complex geometries are difficult to produce due to equipment and mold design limitations. A possible solution emerged with the evolution of Additive Manufacturing (AM). Additive manufacturing has the potential to fabricate complex, near-net-shaped parts. A wide variety of technologies such as vat photopolymerization with ceramic suspensions, binder jetting, material extrusion, etc. can be utilized to fabricate the desired components; the latter, and in particular direct ink writing (DIW), has drawn our interest due to its simplicity, production speed, greater flexibility in terms of feedstock, and limited amount of binder needed. Here, we report on the DIW of BaTiO₃, which is one of the most widely used functional ceramics. The appropriate shaping of BaTiO₃ allows for the tailoring of these properties to the specific application needs. An ink with very high solid loading and limited binder content was optimized with desirable rheological properties so that it can be extruded through nozzles with a diameter of 0.4 mm or more. Porous log-pile structures were printed using commercially available 3D printing equipment (Delta Wasp 2040 Turbo 2). Several sintering strategies, including non conventional ones such as ultra-fast high temperature sintering and pressure less spark plasma sintering, have been tested to produce crack-free components. Moreover, the time required to reach density comparable to those achieved with conventional sintering was reduced drastically from few hours to minutes in inert atmosphere. Coupling DIW with field-assisted sintering provides a proof-of-concept for rapid processing of ceramics and may also be used to process a wide range of shapes and compositions.

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Near-universal slicing for continuous-extrusion DIW through differential growth algorithms

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Topic Areas

Material extrusion with inks and pastes I

Abstract

One of the main advantages of Fused Deposition Modelling (FDM) over Direct Ink Writing (DIW) is the option of reliably retracting the filament during the printing process, yielding discontinuous extrusion. While this may also be feasible in DIW with the appropriate setup, the more difficult control of discontinuous flow compared to FDM may lead to critical defects in the final part and it is often best to limit it or avoid it completely. However, with commercial slicing software catering towards FDM, generation of continuous machine paths for DIW is often a manual and tedious process.

The authors propose a novel slicing strategy for continuous extrusion DIW processes by exploiting differential growth, a common phenomenon in the natural world which occurs when a confined system needs to grow while maximising its surface area. The result is complex, heavily folded 2D or 3D shapes seen, for example, in the growth pattern of corals or the corrugated structure of mammalian brains.

A custom differential growth algorithm was implemented in Rhinoceros3D/Grasshopper. After slicing the model into 2D layers, the algorithm iteratively grows a planar space-filling curve for each layer. Each iteration consists of a growth phase, where new curve segments are created in proximity to empty areas, and an equilibration phase, where the curve relaxes to achieve a homogeneous density. The process continues until the entire 2D section is filled, then it is repeated for the other layers. Each layer is thus traced with a single, non-intersecting curve which constitutes an ideal machine path for continuous-extrusion DIW. Moreover, porosity may be controlled by choosing the appropriate curve density to alternatively yield porous infill or fully dense sections.

Through this fully automated approach, slicing of objects with arbitrary shapes can be achieved without the need for discontinuous extrusion, greatly increasing the flexibility of DIW processes.

Robocasting of reaction-bonded carbides: Multi-layer and core-shell printing

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Topic Areas

Material extrusion with inks and pastes I

Abstract

Robocasting, also called Direct Ink Writing, is an Additive Manufacturing process enabling the processing of various ceramics and the production of complex structures. Ceramic pastes with suitable rheological properties are printed layer-wise to build the desired structure. Additionally, robocasting offers the possibility of printing multi-material structures. Samples consisting of different materials can be produced through the use of two nozzles, or core-shell struts are feasible using special nozzles.

In this work, reaction-bonded carbides were fabricated by combining robocasting and liquid silicon infiltration. Porous, printed preforms were infiltrated with liquid silicon to obtain different reaction-bonded carbides, depending on the used starting materials. For the first time, pastes were used to print multi-layer laminates as well as core-shell structures using a multi-material printer. The microstructure of the fabricated specimens was analyzed, and physical and mechanical properties were investigated. Varying crack patterns were observed depending on the material combination and printed structure. This crack formation is due to residual stresses during cooling, which were investigated using Raman spectroscopy. The shifting of the Si peak gives information about the stress, and these can be used to explain the crack formation.

Poster Session I

Preparation of Al₂O₃-TiB₂ composite coating by Air Plasma spraying of SHS powders on steel substrate

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Topic Areas

Poster Session I

Abstract

In the present study, Al₂O₃-TiB₂ coating was successfully produced on steel substrates by atmospheric plasma spraying using the powders obtained from self-propagating high-temperature synthesis. At first, The H₃BO₃, TiO₂, and Al mixture was milled for 10 h, and then combustion synthesis was conducted in the mode of a thermal explosion at the furnace in an inert atmosphere (Ar) at 900°C. After the granulation of obtained powder, they were deposited on a steel substrate. XRD and FESEM investigated the phase composition and microstructure of SHS powder and coating. Wear behavior was investigated by measuring hardness, wear track width, and wear rate for the coating and substrate. Microhardness and wear resistance of the coating were improved in comparison with the substrate. FESEM observations showed that the thickness of coating was about 300 μm and abrasive, delamination, and adhesive were the main wear mechanisms in Al₂O₃-TiB₂ coated specimens.

3D printing of AlN ceramic by Binder Jetting and SLA-DLP for electronic packaging

Authors

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Topic Areas

Poster Session I

Abstract

This work focuses on the development of 3D AlN ceramic packaging to reach requirements of current and future high power devices (gallium-nitride or silicon carbide semi-conductor). New designs of ceramic hoods are developed to ensure efficient cooling, optimize power density and improve compactness of those new electronic devices.

Initially, a specific process to prepare powder is developed to reach requirements for both 3D printing techniques: Binder Jetting (Innovent+, ExOne) and SLA-DLP (V6000, Prodways). Several steps including mixing, granulation, grinding and sieving are done to obtain particular powders. Binder Jetting (BJ) needs powder with good flowability. SLA-DLP requires powder that mixes with polyacrylate resins and polymerizes with UV light. 3D parts are successfully printed with both 3D techniques and sintered to densify AlN material.

Characterizations of 3D AlN materials are done, especially density, hardness and thermal conductivity and diffusivity. Then, performance of both materials, printed by BJ or SLA-DLP, is compared.

Finally, first results on metallization of AlN will be shown.

3D printing of zirconia-based ceramics by lithography-based ceramic manufacturing (LCM) for structural and functional high-performance applications

Authors

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Topic Areas

Poster Session I

Abstract

Due to its exceptional mechanical properties, chemical inertness and biocompatibility yttria-stabilized zirconia (YSZ) is a high-performance ceramic material for many technical as well as medical applications. Additive manufacturing (AM) enables the fabrication of zirconia parts for a wide range of applications as it offers a higher flexibility in design compared to conventional fabrication methods. One such AM method is lithography-based ceramic manufacturing (LCM), which is a vat photopolymerization-based technique to process photosensitive ceramic slurries to green parts by selective exposure to light.

This contribution focuses on the development of YSZ systems that provide excellent performance in terms of mechanical and functional properties.

3mol% yttria-stabilized zirconia with a 4-point-bending strength of >1000 MPa can be printed with the LCM technology, therefore these 3D printed samples are already comparable to those produced by conventional shaping methods. Further improvement will be sought by evaluating the slurry production parameters as well as adjusting the thermal post-processing routine. It was also feasible to manufacture parts with an extraordinarily high reliability, reaching a Weibull modulus of 20, while maintaining a high bending strength.

Even though 3YSZ is the more commonly used zirconia grade, lower stabilized grades have recently gained attention for their excellent mechanical properties as well as their lower sintering temperatures. These 1.5 or 2 mol%-Yttria stabilized grades also promise an interesting starting material for the LCM process and the possibility to fabricate samples with even higher bending strengths. Another application with high relevance is the use of 8mol% stabilized zirconia for the use as electrolyte in solid oxide fuel cells (SOFCs). Printing 8YSZ with the LCM technology offers novel approaches in the design of SOFCs.

Preparation and characterization of $(\text{Na}_{1/2}\text{Bi}_{1/2})\text{TiO}_3$ -based piezoceramic suspensions for vat photopolymerization

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Topic Areas

Poster Session I

Abstract

Piezoceramics enable the interconversion of electrical and mechanical signals and are therefore indispensable in robotic, energy conversion, medical technology, consumer electronics, and autonomous vehicles. While piezoceramics are traditionally being produced in continuous mass production lines, there is an increasingly large number of applications that require complex-shaped objects with minimum waste and a targeted property combination. This triggered the development of additive manufacturing technologies, whereby one of the promising techniques is vat photopolymerization. However, printing inks of piezoceramics are not readily available and their behavior during photopolymerization is not well understood. The goal of this work was therefore to design stable photoactive suspensions using the piezoceramic material $(\text{Na}_{1/2}\text{Bi}_{1/2})\text{TiO}_3\text{-BaTiO}_3$, which is being considered as a lead-free alternative for $\text{Pb}(\text{Zr,Ti})\text{O}_3$ in high-power resonance applications. The ceramic powders have been prepared by solid-state reaction and the powder size and morphology were optimized. Various acrylate-based suspensions with different ceramic solid loads were prepared and stabilized. After investigating their rheological properties to ensure a suitable viscosity, the suspensions were polymerized using UV light of different intensities. In particular, the curing depth and layer lamination have been investigated. The photopolymerized samples have been benchmarked against samples prepared by conventional forming methods and their microstructures and electrical properties were compared.

Fabrication of partially water-soluble sacrificial molds obtained by vat-photopolymerization

Authors

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Topic Areas

Poster Session I

Abstract

Ceramic additive manufacturing is growing exponentially and has been used in several applications due to its high geometrical freedom. However, it is challenging to produce parts with small inner channels, such as heat exchangers, artificial rocks, and bone scaffolds with low porosity, due to the difficulties of removing the support or residual material from the complex porous structures. One possible solution is to produce sacrificial templates by additive manufacturing combined with ceramic casting. This alternative benefits from the versatility of 3D printing for the inner channels while maintaining a cheaper and simple casting for the whole structure. For this purpose, it is essential to obtain a sacrificial mold that reproduces the internal structure, has appropriate resistance for casting, and is easily decomposed. This study presents the development of a photocurable resin used to print partially water-soluble molds through vat-photopolymerization. The components of the suspension were tetra(ethylene glycol) diacrylate, icing sugar, camphor, a dispersant, and a photoinitiator, which were selected to obtain good printability and easy decomposition. About 50 vol% of the resulting ink was water soluble, while the remaining part could be thermally decomposed at moderate temperatures without inducing cracks into the part. The viscosity and curing time were measured to characterize the suspension, and the printing resolution was evaluated using SEM. Next, printed samples were immersed in water, and the mass loss and density were measured over time to evaluate the water-dissolution speed. Then, the thermal decomposition of the resin was evaluated before and after the water dissolution. Finally, as a proof-of-concept, some molds were printed, cast with a composite of calcium carbonate and geopolymer, and decomposed. The results were discussed regarding water solubility and the geometry achieved. In conclusion, the developed resin is appropriate to satisfy the requirements for sacrificial molds for ceramic casting.

Suitability of fast sintering techniques for the consolidation TCP scaffolds manufactured by Digital Light Processing

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Topic Areas

Poster Session I

Abstract

In this study porous scaffolds were fabricated via Digital Light Processing (DLP) from commercial β -TCP powder and sintered using different techniques: conventional sintering in air (*CSA*), rapid sintering in air (*RSA*) and pressure-less spark plasma sintering (*pI-SPS*) in vacuum, at four different temperatures: 1200, 1300, 1400 and 1500 °C. Although all three techniques are suitable for sintering layered structures produced by DLP, each sintering strategy resulted in scaffolds with different phase composition, microstructure and mechanical properties. Regarding their chemical composition, the results confirm, that long dwell times or high temperatures are necessary to achieve a complete $\beta \rightarrow \alpha$ transformation; and that rapid cooling rates facilitate avoiding the reverse transformation ($\alpha \rightarrow \beta$). Moreover, the presence of graphite in the sintering chamber plays a crucial role in stabilizing the α -TCP phase, explaining the prevalence of this phase in *pI-SPS* scaffolds. However, the incorporation of carbon into the microstructure hindered the densification *pI-SPS* scaffolds, although this avoided the generation of transformation-induced cracks. These were present in all *CSA* and *RSA* scaffolds sintered above 1200 °C and deteriorated their mechanical properties. Nevertheless, all scaffolds exhibited compressive strengths within the range of cancellous bone strength values, with the highest average value of 22 ± 4 MPa achieved by the *RSA* scaffolds sintered at 1300 °C, thanks to the high level of densification and fine microstructure obtained during the fast-sintering treatment.

Dimensional accuracy of zirconia dental bars fabricated by vat photopolymerization and material extrusion

Authors

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Topic Areas

Poster Session I

Abstract

Because of its ability to produce custom-tailored pieces from zirconia ceramics with complex geometries and thin margins, additive manufacturing (AM) is a promising technology for dental applications. Vat photopolymerization (VPP) is the most promising among the entire set, producing accurate and dense objects. However, multi-material AM techniques based on VPP are not yet as advanced. Thus, fabricating pieces from different materials is currently more accessible with material extrusion (MEX) techniques, which are capable of multi-material AM but lack dimensional accuracy. Dimensional accuracy is critical in producing dental constructions because these must fit passively to teeth or dental implants, not introducing harmful internal tensions that affect the construction's longevity. The study aimed to evaluate the dimensional accuracy of zirconia dental bars fabricated with VPP and MEX and compared to conventional CAD-CAM milling.

Zirconia dental bars, replicating real-time clinical pieces with complex geometry, were digitally designed (exocad Plovidiv 2.4) and manufactured by VPP (CeraFab 7500), MEX (Prusa i3 MK3S), and conventional CAD-CAM milling to serve as a control (n=10/gp). Dental bars were scanned by an optical scanning system (ATOS Q) and X-ray micro-computed tomography (Neoscan S80). Scan and CAD data were superimposed using the iterative closest point (ICP) registration algorithm (GOM Inspect 2017) and 3D deviation root mean squares (RMS) were obtained. The RMS differences between different fabrication methods were determined using one-way ANOVA followed by Tukey HSD post hoc test for pairwise comparison. The level of statistical significance was set at $P < 0.05$.

Dental bars fabricated by CAD-CAM milling and VPP presented low RMS values, indicating high dimensional accuracy and reproduction ability, whereas the RMS values of MEX were high. The dimensional accuracies of both VPP and CAD-CAM milled dental bars were within the clinically acceptable margin (150 μm), while chippings at thin margins were observed with the CAD-CAM milled zirconia bars.

Band-gap engineering studies of nano TiO₂-based 3D porous FFF parts by thermal treatments

Authors

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Topic Areas

Poster Session I

Abstract

In 2015, the United Nations General Assembly adopted the 2030 Agenda for Sustainable Development as a roadmap for international cooperation on sustainable development. Today, geopolitical developments make it essential to redouble efforts and address challenges for the rapid prototyping of advanced, environmentally friendly, low-cost and highly efficient photochemical devices.

One of the most promising technologies for water treatment today is photocatalytic membrane reactors (PMRs) for the immobilisation of nanometric semiconductors; however, their engineering is still unresolved. The challenge is related to the fabrication of porous photoactive membranes adapted to PMR configurations. In that sense, this work focuses on the prototyping of porous photoactive membranes from thermal material extrusion additive manufacturing technologies. The band gap engineering of the commercial semiconductors used will be carried out by thermal treatments at different atmospheres of the 3D printed parts.

In this regard, for water decontamination based on ceramic semiconductor (commercial TiO₂) we proposed a colloidal method to self-support nanoparticles in a polymeric matrix of polylactic acid (PLA). The fabrication of a photocatalysts material of thermoplastic nature made the composite suitable for processing by additive manufacturing techniques. The first step is surface modification using a polyelectrolyte to ensure homogeneous dispersion of the particles in the new medium. Then, after wet mixing of the matrix-particle system heat extrusion are used to process the final parts. These are thermally treated to enhance the photocatalytic activity of the nanoparticles in the matrix at different atmospheres. Catalytic activity is evaluated as a function of the degradation of a chemical indicator over time. The results indicate that the geometries printed by FFF and pre-sintered in different atmospheres reach degradation rates for methyl orange of 100% after an exposure time of 12h, reaching 50% degradation after 4 hour of testing.

Development of a partially water soluble binder for fused deposition of ceramic

Authors

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Topic Areas

Poster Session I

Abstract

In comparison to other additive manufacturing techniques of ceramic, the use of material extrusion based techniques are limited by two main factors: the staircase effect and the rough surface quality. These effects are inconvenient not only for optical reasons, but they also increase the surface roughness that may create stress concentrations.

The work focus on the development of a partially water soluble binder for fused deposition of ceramic with an alumina-PVB-PEG-plasticizer basis, which enable an easy and automated post processing through dip-coating.

In a first step, the influence of the feedstock constituents on the mechanical and thermal properties, the rheology, the debinding and sintering behavior are investigated. An optimal composition is aimed with mixture design methodology. In the center point composition of the test matrix, different filler contents, various constituents and additives are investigated to evaluate the effects on the above mentioned properties. The mechanical properties are evaluated with tensile test on rectangular extruded parts. These samples are also used for the water debinding, and the sintering shrinkage evaluations. The thermal properties of the feedstocks are investigated with DSC, whereas the thermal debinding is evaluated with TG/DTA analysis coupled with thermokinetics modelling.

In the second step, a suspension will be developed for dip-coating in order to improve the surface roughness without altering the printing resolution. The influence of the surface roughness on the mechanical properties of the sintered parts will be investigated with white light interferometry and four points bending tests on as processed and dip-coated samples printed with different angles.

The feedstock and the post-processing step developed in this research will enable to print dense ceramics parts with high surface quality and serve as basis for the development of tougher ceramics, which will allow the use of Fused deposition of ceramic for structural applications.

Structured NH₃ sorbents fabricated by DIW of SrCl₂-based composites

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Topic Areas

Poster Session I

Abstract

Reducing emissions of NO_x from internal combustion engines is crucial for improving air quality and meeting regulatory standards. One effective method is selective catalytic reduction (SCR), which uses NH₃ as a reducing agent over a suitable catalyst to convert NO_x to N₂ and H₂O. However, the use of NH₃ presents significant safety risks when stored in pressurized vessels, so commercial SCR units typically employ urea as a reducing agent, which produces CO₂ as a byproduct. Alternatively, alkali earth metal halides (AEMHs) offer a solid-state storage solution, but the volumetric expansion associated with the formation of ammine complexes limits their practicality.

Here, we propose a novel approach for shaping SrCl₂ into a structured sorbent for NH₃ storage in SCR units using Direct Ink Writing (DIW) of SrCl₂-bentonite slurries. The 3D-printed component's porous lattice design improves NH₃ access to the salt and accommodates SrCl₂'s volumetric expansion (over 400%) to preserve structural integrity. The use of bentonite allows for good mechanical properties of the printed monoliths after drying without the need for heat treatments, which can be problematic due to the poor stability of SrCl₂ at high temperature. Our SrCl₂-bentonite monoliths exhibited comparable NH₃ storage capacity to that of pure SrCl₂ and maintained performance and structural integrity during numerous absorption-desorption cycles.

In summary, our approach offers a safer and more efficient method for NH₃ storage in SCR units and demonstrates the potential of additive manufacturing for creating novel sorbent materials.

Printing microbatteries by Robocasting

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Topic Areas

Poster Session I

Abstract

The integration of microbatteries in increasingly complex (e.g. flexible or wearable electronics) and numerous electronic devices requires a paradigm shift in their fabrication, towards greater design and fabrication flexibility, lower costs, as well as a reduced environmental footprint. Additive manufacturing can meet this challenge, while allowing innovative internal structuring. This work aims to demonstrate the feasibility of microbatteries printed by Direct Ink Writing (DIW) [1]. This additive manufacturing technique allows printing with a current resolution of the order of 200 μm of anode, cathode, and even solid electrolyte materials. By optimizing both the materials and the design of their architecture a better manufacturing flexibility is targeted as well as a decrease in financial and environmental costs.

In the present work, the optimization of the formulation and the printing conditions allowed to obtain a printable and rheologically adequate graphite-based ink. This enabled the printing of anodes with good electrochemical performances on low charge cycles ($C/20$ to $C/10$).

The active material chosen as cathode is LiFePO_4 (LFP). Cathode ink formulations are not yet rheologically suitable for DIW. SEM observations of the LFP highlight the aggregation of the particles up to nearly 200 micrometers (above the printing limit). Moreover, the viscosity of these inks changes strongly with time: it decreases by 96% in less than 20 days. Despite these rheological characteristics still to be worked on, the first printed cathodes have shown very good capacity maintained even at C-rate $C/5$.

DIW of complete batteries offers a new field of possibilities in terms of battery design. The interdigitated battery is one of these still hardly explored paths [2]. To achieve this, gold collectors deposited by lithography on glass slides will allow us to realize the first interdigitated battery with ionogel electrolyte [3].

Choosing the Correct Raster Pattern and Drying Route for High-Density and High-Strength Monolithic Alumina Toughened Zirconia Parts

Authors

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Topic Areas

Poster Session I

Abstract

Direct Ink Writing, also called robocasting, is an additive manufacturing technique that allows for the production of complex ceramic parts with high density. This process involves extruding a high-solid-loading viscoelastic paste through a narrow nozzle layer-by-layer based on a computer-aided design (CAD) model, followed by drying, debinding and sintering the printed green body. As the pastes studied are high solid loading aqueous pastes, controlled humidity drying is required to avoid drying cracks and warping.

The selection of printing parameters is crucial for the mechanical properties due to the inability to remove the printing defects during the subsequent drying-debinding-sintering steps. These parameters decide the printed and sintered parts' overall dimensional accuracy, roughness, and density. The printed parts' resolution and surface quality depend mainly on nozzle shape and size. At the same time, the dimensional accuracy is primarily related to the extrusion rate, i.e. pressure and nozzle speed. Additionally, carefully selecting the raster pattern, filament spacing, and layer height is required to produce high-density parts with minimal defects.

To obtain the desired printing quality, optimization of printing parameters and the drying-debinding-sintering route is crucial. This study optimized raster pattern and layer height based on the mechanical characterization of the high-density, high-strength, and high-accuracy monolithic ATZ parts produced with optimized paste and printing parameters. Moreover, a drying study is conducted to shorten the already established drying route.

Elaboration of BaTiO₃ piezoceramics by Direct Ink Writing for acoustic applications

Authors

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Topic Areas

Poster Session I

Abstract

Due to their properties, piezoelectric ceramics allow an integration in such various systems as capacitors, actuators, and acoustic transducers. Their single-material or composite forms generate plenty of interest in maritime, biomedical, and numerous fields. The improvement of these devices and their performances involve the manufacture of more complex geometry materials. Nevertheless, conventional shaping routes require numerous machining steps that are prone to inherent constraints as the brittleness of the ceramic materials and the wastage of raw materials. These challenges can be overcome by investigating new manufacturing ways such as additive manufacturing that allows more design flexibility for 3D-shaping and a lower production cost. The shaping of ceramics by 3D printing requires a particular study when choosing organic additives and the load rate of solid particles in the formulation because the rheological properties of the ink play a major role in the development of ceramics with complex shapes. The present communication reports the production of BaTiO₃-based materials using Direct Ink Writing technology, similar in its piezoelectric and dielectric properties to those that have been made by conventional routes. The preparation of inks for which the rheological behavior ensures good printability of our ceramic slurries will be presented. Additionally, investigations on the thermal treatments as well as the electro-mechanical properties of these ceramics will be characterized and discussed in this study.

Preparation of planetary simulant granulates with high flowability via spray drying for powder-based additive manufacturing in space

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Topic Areas

Poster Session I

Abstract

The world economy is on the verge of a second space age, with a huge increase in investment and attention to activities in space, often from the private sector. Many forward-looking proposals envision using fine regolith particles covering planetary bodies' surfaces as base materials for a future in-space economy. Most processing proposals aim to directly process the rugged-edged regolith, including their significant fine particle fraction. Such a direct utilization on Earth is typically unfeasible — for example ceramic feedstock materials for AM with a fine particle fraction are generally handled either dispersed in liquids or pre-processed to increase flowability. Here we propose spray drying for planetary regolith to increase their flowability. Our concept includes using space clay minerals as binders in the spray drying process, with clay being available from Asteroids in the cis-lunar economic sphere or directly on the surface of Mars. In detail, we milled and mixed Lunar, Martian and Astroid simulants with water, producing slurries with a small particle size ($d_{50} \sim 5 \mu\text{m}$). We investigated the rheology of such slurries with and without small amounts of additives, for example, changing the pH. We produced simulant granulates that showed excellent flowability confirmed using angle of repose, Scott bulk density, tapped density, Hausner's ratio and powder rheometer characterization. The produced spray-dried powders are ideally suited for various AM processes such as powder-bed applications (BJ and PBF) and powder spraying (DED), which we showed by generating model powder beds and measuring their density as well as transporting powders in a continuous stream through a hose using a DED powder delivery system. Further work will explore various AM methods to fuse spray-dried granules, especially focusing on prospective space resource utilization (SRU) applications.

Material extrusion with inks and pastes II

Additive manufacturing of capillary suspensions for sustainable energy harvesting

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Topic Areas

Material extrusion with inks and pastes II

Abstract

In the wake of rapid technological advancements, portable electronics and wireless sensors have driven the need for small, long-lasting power sources. Complex architected porous ceramic structures are relevant for such applications, as these architected, hierarchically organized structures can enhance the functional properties of electroceramic materials. Capillary suspensions (CapS) are a novel, innovative, and inexpensive method for creating highly open porous ceramics. Manufacturing precise porous structures with complex geometries are challenging. However, additive manufacturing of CapS has the potential to develop hierarchically complex structured functional materials with unprecedented properties. A high Figure of Merit (FOM) opens up a wide range of applications ranging from electromechanical energy harvesting over battery and fuel cell materials or thermoelectric applications to bone tissue utilizing piezoelectric stimulation of tissue growth.

This work aims to produce highly porous barium titanate (BT) ceramics via direct ink writing using CapS-type BT inks. Various geometries with self-organizing particle networks were printed and analyzed. The capillary forces acting in this ternary solid/fluid/fluid system induced the formation of a self-organized, sample-spanning particle network. Due to the distinct flow properties of the ink, it was possible to achieve printed structures with a high strut size-to-pore ratio of about 1:4, with an overall porosity in the range of 40-60%. Out of all the different geometries tested, the highly porous additive manufactured log-pile structure sintered at 1150 °C was found to show an overall porosity of 58% with the d_{33rem} value of 330 pC N⁻¹ and a remanent relative permittivity of 1197 which resulted in a high energy harvesting figure of merit (FOM₃₃) of 10.3 pm² N⁻¹, which is more than four times higher than the documented data for this particular material¹.

1. Menne, D., Lemos da Silva, L., Rotan, M., Glaum, J., Hinterstein, M. & Willenbacher, N. *ACS Appl. Mater. Interfaces* **14**, 3027–3037 (2022).

Ceramic Matrix Composites shaped by DIW of Pre-Ceramic Polymer based feedstocks

Authors

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Topic Areas

Material extrusion with inks and pastes II

Abstract

Pre-Ceramic Polymers (PCPs) are compounds that can be shaped as polymers and then converted into ceramics with thermal treatment in an inert atmosphere; the main issue of these materials is the formation of cracks due to the shrinkage that occurs in the conversion from polymer to ceramic, more evident the bulkier the object is. Additive Manufacturing (AM) technologies are helpful to reduce the problem because they enable the creation of complex geometries with thin walls.

In this work Direct Ink Writing (DIW) was used to shape, according to a python-generated geometry, the PCP-based feedstocks, that contain chopped carbon fibers and fillers, used to improve the mechanical properties and to further reduce the shrinkage, as well as to modify the rheology of the composition, the samples were then converted to Ceramic Matrix Composites (CMCs) by thermal treatment in nitrogen atmosphere.

To increase the complexity of achievable shapes, Embedded-DIW was used to fabricate geometries, generated by a different custom-made Python code. This technology uses the DIW technique to extrude the slurry in a viscous medium that supports the structures; in this way, it was possible to produce curved surfaces, continuously extruded and without supports.

Ordered mesoporous silica-based robocasting feedstock formulation: impact of the inorganic binder content on flow, mechanical and structural properties.

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Topic Areas

Material extrusion with inks and pastes II

Abstract

COK-12 is an ordered mesoporous silica that has attracted attention for its remarkably porous structure and environmentally friendlier synthesis. Their shaping through robocasting can facilitate their usage while providing the advantage of tunable geometries to suit any presented application requirements. However, the robocasting process requires certain rheological properties of their feedstocks, and the impact of the feedstock components on the flow behavior is not always clear. In this study, the influence of a widely available and sustainable clay inorganic binder on the rheological behavior of COK-12 robocasting feedstocks and the resulting mechanical and structural properties of the printed bodies is measured.

The parameters of the Herschel-Bulkley rheological model are examined at a constant solid volume content and varying feedstock COK-12/clay binder ratio. The rheology of the slurries with higher clay content is characterized by lower flow indexes (higher shear-thinning behavior) and higher yield stresses, which prove convenient for processing. Monoliths were robocasted with a modified fused deposition modelling 3D printer in a woodpile structure and calcined at 600°C. This temperature conserved the amorphous, mesoporous structure of the COK-12 and generated sufficient mechanical resistance to permit handling.

Micro-computer tomography measurements showed linear shrinkage independence from the COK-12/clay ratio. The printed structures exhibited up to 40% higher specific mesopore volumes, and up to 36% increased average mesopore size when compared to the calcined COK-12 when examined by gas sorption analyses. Small-angle X-ray scattering (SAXS) analysis showed an increased lattice parameter and reduced wall thickness in the OMS fraction of the monoliths. Mechanical characterization showed a 5-fold increase in uniaxial compressive strength of the prints when doubling the binder/solids ratio.

These findings highlight the importance of an adequate feedstock formulation, and its significant impact not only on the flow properties of the feedstock but also on the structure of the final material.

Dynamic Molding as a new opportunity for large size ceramics and ceramic-based composites additive manufacturing: two case studies

Authors

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Topic Areas

Emerging, hybrid and multimaterial AM technologies

Abstract

Despite the significant progresses of the last decades, ceramic Additive Manufacturing (AM) most performing technologies (*i.e.*, direct ink writing or stereolithography) still present limitations: the size of printable objects, the need of supports in case of complex/heavy geometries (affecting surface and mechanical performance) and the opening to ceramic-based composites.

Our goal in this project is the evaluation of ceramic materials and ceramic-loaded composites printability by Dynamic Molding, in order to overcome the limitations of existing AM technologies. Dynamic Molding is an extrusion-based AM technology, whose building environment is composed of a granular phase (powder), behaving like a dynamic molding system, in which manufacturing materials (inks) are dispensed. Two case studies of silicone-ceramic composites and pure alumina printing by Dynamic Molding are presented.

In the case of ceramic-loaded composites, a silicone ink is extruded and act as the matrix, opening the way to ceramic soft composites. The integration of advanced ceramic powders into the silicone matrix, achieved by Dynamic Molding, allows for elastomeric 3D objects with improved properties as thermal insulation and fire resistance. The charge rate is up to 45 vol.%. Furthermore, hardness gradients and modular mechanical properties are achieved by controlling the printing parameters of the technology.

For pure ceramic AM, the hypothesis is that the support given to printed parts by the powder allows for heavier and larger objects without using extra support structures. The compressive force acting during the printing process – due to the powder weight – can compact and integrate the powder in the printed green part and result in denser objects. The results obtained for alumina-based inks and powders demonstrate the printability of large-size 3D models (>150 cm³) with relative density values >85% on sintered samples. Complex geometries, including overhanging structures (>45°), were successfully printed without using support structures.

Mechanical testing

Strength and failure of AM ceramics: what fractography can tell us

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Topic Areas

Mechanical testing

Abstract

No matter which for application a ceramic part is designed and no matter which additive manufacturing method is used to make it, in order to perform adequately, it has to maintain its structural integrity. This can be achieved if it has a sufficient strength and life-time. In ceramics the strength is governed by material inhomogeneities that act like cracks. The mechanical strength as well as the life-time of ceramic components can be improved to a great extent, if these defects are eliminated or if their size and frequency is reduced.

Fractography of ceramics – after strength tests or after component failure - is used to detect the fracture initiating defects. The nature of these defects can be related to processing steps which are typical for certain aspects of a given additive manufacturing method. Thus, valuable hints can be obtained to improve and control the additive manufacturing processes. Knowledge of defect types in specimens which were tested in order to obtain strength distributions can help to interpret the strength scattering behaviour.

Examples for such characteristic defects and their formation in ceramics made by material extrusion and vat based photo polymerization of various ceramics will be shown to demonstrate strategies to discover typical defects and illustrate how the nature of defects can be traced back to processing details. The consequences of the existence of different defect types on strength distributions will be exemplified.

Design concepts for 3D-printing alumina-based multi-material ceramics with exceptional mechanical properties

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Topic Areas

Mechanical testing

Abstract

In this work, the potential of the Lithography-based Ceramic Manufacturing (LCM) technology to design novel alumina-based ceramic systems is explored. The first approach demonstrates the use of the LCM technology to print alumina-based ceramic parts with superior strength. The combination of alumina (outer regions) with alumina-zirconia (ZTA) layers introduces compressive residual stresses in the surface layers. A characteristic biaxial strength as high as 1 GPa is measured on the alumina-based multilayers corresponding to the magnitude of in-plane residual stresses in the external alumina layers. The second approach shows the effectiveness of the multi-material design to enhance the thermal shock resistance of 3D-printed alumina-based ceramics. In this damage tolerant design, the alumina layers are embedded between 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 architecture. The retained strength after thermal shock of the multi-material ceramic is significantly higher than that of the monoliths, owed to the crack arrest capability of the embedded layers. In the last step, the feasibility of designing and printing a more-complex shaped component with tailored residual stresses is represented, which may open new application fields in the future.

A novel strength testing method for additively manufactured ceramics

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Topic Areas

Mechanical testing

Abstract

3D-printed components exhibit textured surfaces as a consequence of the additive manufacturing process. For the stereolithographic manufacturing process, the texture's geometry and periodicity depend on the printing direction, i.e. the angle between the building direction and the specimen surface. Such structures stem from over-polymerization and from the aliasing effect, which is a result of the pixel-based nature of the stereolithographic manufacturing process. Therefore, the strength of ceramic 3D-printed components may depend on the orientation in which tensile stresses act with respect to the printing direction. Other aspects of the manufacturing method, such as insufficient layer bonding or delamination, might further increase the observed variation in strength.

In order to provide reliable material data for the design of components, the strength characteristics have to be assessed routinely for various processing conditions and/or as means for quality control. Such investigations are laborious and costly if standardized strength specimens and methods for ceramics are used. In the project CharAM (ZIM & IraSME), an innovative test specimen is designed and investigated. It allows manufacturing of a sample set of sufficient size (2 x 48 specimens) for statistical strength evaluation within a single print job with the LCM-procedure (CerAM VPP). Additionally, suitable test equipment is developed to enable rapid and simple test execution.

In order to assess the practical applicability of this testing method and its accuracy, a thorough investigation of possible influences on the measured strength has been conducted. Supported by practical observations, Monte-Carlo analysis, analytical considerations, and Finite-Element-Analysis have been employed. Based on Weibull theory, the influence of surface structures on the measured strength was determined through both empirical strength testing and Finite-Element-Analysis.

Validation of the CharAM test methodology in two round robin tests

Authors

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Topic Areas

Mechanical testing

Abstract

Additive manufacturing (AM) technologies are tool-less manufacturing processes that increase freedom in component design and make it possible to design and construct according to function rather than production. However, these new technologies also present new challenges, e.g., with respect to the mechanical characterization of filigree structural components that could not be manufactured previously. Another challenge is the consideration of AM-specific properties, such as the cascading surfaces created by the layer-by-layer manufacturing process.

Based on these challenges, the CharAM characterization method was developed, which is based on a unilaterally fixed bending beam with a varied cross-section to ensure a constant maximum bending stress. The effort required to manufacture and characterize a large number of test specimens has been significantly reduced. In addition, differences in surface quality resulting from different orientations of the components in the construction volume can be simulated and taken into account.

As part of the IraSME project "CharAM", the methodology was further optimized and validated in two round robin tests with a total of six partners. The test bodies were all additively manufactured using CerAM VPP technology and equipment as well as a suspension (Lithalox 350) from Lithoz, Vienna. The geometry and the orientation of the rods were varied and the results differed significantly.

During the presentation we will describe and discuss in detail the obtained results of the interlaboratory tests. In the first round robin test, the manufacturing and sintering was done at the respective partners and only the mechanical characterization was done at IKTS with different testing equipment. In the second round robin test, only the shaping was performed at the partners and the thermal processing and characterization at IKTS.

Vat photopolymerization I

Computational Modelling for Ceramic Stereolithography Process

Authors

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Topic Areas

Vat photopolymerization I

Abstract

In ceramic stereolithography, ceramic particles are dispersed in a photosensitive resin to create a thixotropic paste suitable for processing. This work demonstrates the use of the Discrete Element Method to model the behaviour of ceramic particles, such as Alumina, Yttria Stabilised Zirconia, and glass/alumina, in a resin matrix. This method was used to predict the maximum achievable packing density for stereolithography paste optimisation, and optimal particle size distribution of ceramic particles in order to reduce the time required for post-process sintering of ceramic parts. Ray Tracing was also used to further analyse the particle models to determine the required laser processing parameters to achieve sufficient material curing by stereolithography. Utilising these two methods reduced the number of experimental iterations required for material preparation and parameter optimisation, reducing the time and resource cost of optimisation. Additionally, complex geometries were evaluated by Computational Fluid Dynamics to aid the design of parts for specialised components such as complex flame nozzles, solid electrolytes, and thermoacoustic stacks. These methods will be valuable in industry and research to improve ceramic stereolithography process optimisation and component performance.

Proper selection of solvent for a 3Y-TZP zirconia suspension decreases viscosity and retaining low post-processing shrinkage.

Authors

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Topic Areas

Vat photopolymerization I

Abstract

Obtaining homogeneous and stable ceramic suspensions with high solid loading and low viscosity represents a challenge in vat photopolymerization additive manufacturing (AM) because solid particles increase the viscosity, resulting in non-uniform deposition layers. Solvents added to the formulations may reduce viscosity and decrease warping and delamination of the green parts. However, the greater the amount of organic matter in the suspension, the greater the post-sintering shrinkage. In this study, we evaluate the effect of different solvents in a 3Y-TZP zirconia formulation for vat-photopolymerization concerning the suspension viscosity and post-sintering shrinkage. The reference formulation comprised 20 g of the monomer PEGDA (poly(ethylene glycol) diacrylate 575 Mw) and 0.4 g of a photoinitiator. Eight other formulations included one of the following solvents: methyl alcohol, benzyl alcohol, camphor, ethylene glycol, ethyl alcohol, isopropyl alcohol, n-methyl-2-pyrrolidone, and polyethylene glycol. These eight formulations were tested to identify which would present the best behavior regarding the consolidation of layers with 25 μm thickness. The better performance was achieved by the suspension containing n-methyl-2-pyrrolidone. Then, a suspension composed of PEGDA + the dispersant DISPERBYK-111 (3 wt.% of the monomer) + zirconia powder (40 vol.%) + n-methyl-2-pyrrolidone (10 vol.%) + photoinitiator (2% wt.% of the monomer) was prepared. The addition of solvent allowed for a 22.6% decrease in the viscosity of the suspension, facilitating the formation of uniform layers. Average post-sintering shrinkage was 26.11% in all three dimensions. Sintered pieces had an average density of 5.95 g/cm^3 (97.14% of the theoretical density of zirconia) and an average flexural strength of 256.34 ± 48.65 MPa. It was concluded that adding a small amount of solvent does not impair the mechanical properties. In addition, it improved the viscosity of the suspension without compromising post-sintering shrinkage compared to existing data in the literature for solvent-free suspensions.

Nanostructuring approach for the fabrication of carbides via digital light processing

Authors

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Topic Areas

Vat photopolymerization I

Abstract

Carbide materials possess exceptional hardness and strength, combined with high melting points and low coefficients of thermal expansion; their thermal stability enables them to withstand extreme temperatures without significant degradation. Such properties can be enhanced by structuring carbide components via additive manufacturing into architectures with previously unattainable degrees of complexity. However, the limitations for powder-laden inks, such as nozzle clogging, rheological constraints, particle sedimentation, light-scattering and absorbing phenomena narrow the range of available processes. To address this hurdle, we have developed an innovative synthesis protocol for the preparation of sol-gel-based UV-photocurable formulation aiming at the fabrication of carbide/carbon nanocomposites by means of digital light processing (DLP), pointing to applications in the field of nuclear physics. Indeed, carbides have attracted an increasing interest as target materials for the production of radioisotopes in the Isotope Separation On-Line (ISOL) facilities; however, the release of radioisotopes relies on the presence of open porous structures, thus enhancing the diffusion from the target. Through our approach, not only we can overcome powder-related issues, but we can also fabricate hierarchical porous structures; in fact, the control of the positioning of the building blocks and the supramolecular interactions during the inorganic polymerization of the molecular precursors allow for a multiscale structuration of the network, providing a fine control over the local arrangement. The molecular structure of the printed carbides was investigated by means of Fourier transform infrared spectroscopy, while Raman spectroscopy technique was used to characterize the degree of structural order of the carbon phase. X-ray diffraction was employed to unravel the phase identification, and a complete investigation of the textural properties was carried out by means of N₂ physisorption analysis. Our “nanostructuring approach” has thus proven to be a flexible one-pot synthetic route for the design and tuneability of the porous network architecture of several carbide-based materials.

Creep behavior of 2PP-printed technical ceramics

Authors

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Topic Areas

Vat photopolymerization I

Abstract

Vat-Polymerization promises the highest accuracy of all Additive Manufacturing technologies. This also applies for ceramic additive manufacturing. But, ceramic slurries for powder processing routes require ceramic particles, to sinter them into strong ceramic specimen after the printing process. Those ceramic particles turn slurries opaque, which is contradictory to light-based vat polymerization techniques. Choosing nano-particles from ceramic-suspensions with a mean size of 5nm reduces the scattering at the particles sites sufficiently to create a mixture suitable for Two-Photon-Polymerization (2PP) with a ceramic particle fraction of up to 80wt%. This enables the fabrication of yttria stabilized zirconia (YSZ) parts with a resolution of 500nm, the smallest for ceramic-AM so far.

The nanoparticles, due to their higher sintering activity, allow to vary sintering regimes and create stable ceramic architectures already at sintering temperatures of 800°C, compared to 1450°C, the standard temperature for zirconia ceramics. The variation of sintering temperatures results in untypical particle sizes. Together with the high resolution of 2PP architectures are created with mechanical properties different from monolithic zirconia. 2PP-structures show high mechanical strength even at lower densities and creep at room temperature was observed, depending on the sintering temperature, chosen geometry and building resolution. A ceramic meta-material is created.

Vat photopolymerization II

Holographic photopolymerization combined to microfluidics for the fabrication of lab-in-lab microdevices and complex 3D micro-objects

Authors

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Topic Areas

Vat photopolymerization II

Abstract

We will present brand-new possibilities of lab-in-lab fabrication while combining holographic photopolymerization and microfluidics. One shot real-time 3D-printing can produce 3D architected microchannels, or free-standing complex micro-objects eventually in flow. The methodology is very versatile and can be applied to e.g., acrylate resins or hydrogels.

Development of high solid-load slurry for the SLA 3D Printing of yttria-stabilized zirconia electrolytes for SOFCs application

Authors

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Topic Areas

Vat photopolymerization II

Abstract

Additive manufacturing (3D-printing technology) allows shaping ceramic materials with limitless opportunities in terms of design freedom, improving the processing speed while reducing cost and waste. In this work, a stereolithography-based (SLA) method of 3D printing was successfully used to fabricate complex-shaped ceramic components. In the case of the SLA printing technique, the quality of a final product depends on many factors such as the 3D design of the model, preparation process and stability of the ceramic slurry, rheological properties, curing process, debinding, and final sintering conditions. The homogeneous slurries of UV-curable resins with added yttria powder stabilized by 8 mol.% of ZrO₂ with different particle sizes (0.2 and 1.1 μm), additives (PEG), and different solid content in the range of 40-70 vol.% and with appropriate rheological properties were prepared. The cure depth of the suspensions was suitable to print the objects with 30 μm of layer thickness, good interlayers connection, and surface finishing, regardless of solid loading and particle size distribution. All samples were sintered at an optimized sintering rate in the temperature range of 1300-1400 °C for various sintering times, achieving a high relative density of up to 99.0 %TD. The high solid loading and homogeneity of the suspension promote the uniform shrinkage of the printed samples. These results indicate the potential of 3D printing technology by the SLA technique to prepare a dense electrolyte for SOFCs.

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 739566. This work was created in the frame of the project: Advancement and support of R&D for "Centre for diagnostics and quality testing of materials" in the domains of the RIS3 SK specialization, ITMS2014+:313011W442, based on the Operational Programme Integrated Infrastructure and funded from the European Regional Development Fund.

Fabrication of calcium carbonate parts with multiscale porosity: comparing vat-photopolymerization and geopolymer casting.

Authors

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Topic Areas

Vat photopolymerization II

Abstract

Fabricating calcium carbonate parts is fundamental to replicating rocks found in petroleum and gas reservoirs. Such artificial rocks would allow better calibration of petrophysical measurements and the validation of numerical models. However, natural rocks have a multiscale porosity, which is challenging to reproduce using a single manufacturing method. For this reason, using hybrid methods that combine different strategies is necessary to achieve this goal. In this study, we compare two different hybrid strategies to produce calcium carbonate parts with multiscale porosity: Additive manufacturing and casting of a geopolymer composite. 1) The first strategy was the vat-photopolymerization of photocurable acrylate resin loaded with calcium carbonate and a water-soluble pore former. After printing, the porogen was partially removed by water dissolution. Then, all the organics were removed by debinding at 500°C. Finally, sintering at 850°C required a CO₂ atmosphere to avoid the decomposition of the calcium carbonate into calcium oxide. 2) The second strategy was casting a composite of geopolymer (based on metakaolin and a sodium aluminum-silicate solution), calcium carbonate, and a pore former. The composite was cast in an organic sacrificial mold made partially soluble in water. The sacrificial mold and the pore formers were removed by water dissolution and thermal burn-out below 500°C. The resultant parts from both strategies were compared concerning the feasible geometries, mechanical properties, microstructure, density, porosity, and chemical constitution. Among the benefits of the 3D printing strategy, it should be highlighted the final constitution of 100% calcium carbonate. On the other hand, the advantages of geopolymer casting included allowing a larger sample size and not requiring sintering or thermal treatment in a CO₂ atmosphere. Concluding, we discuss how the selection of the most suited strategy should consider the application requirements, such as the geometry, target porosity, mechanical properties, and chemical constitution.

Poster Session II

Application of Potassium-Based Geopolymer in Medical Waste Treatment: Adsorption of Clenbuterol and Comparative Analysis with PAC

Authors

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Topic Areas

Poster Session II

Abstract

This conference presentation focuses on the application of a potassium-based geopolymer in the treatment of medical waste, specifically targeting the adsorption of Clenbuterol, a common pharmaceutical pollutant. The geopolymer samples were manufactured using the robocasting technique, providing precise control over their structure and composition.

To evaluate the geopolymer's effectiveness in adsorbing Clenbuterol, equilibrium tests were conducted. Aliquots of the Clenbuterol solution were collected at various time intervals to assess the adsorption kinetics. Comparative analysis was performed using powdered activated carbon (PAC), a widely utilized adsorbent material, as a benchmark for comparison.

The results reveal that the potassium-based geopolymer exhibits significant adsorption capabilities for Clenbuterol at equilibrium. The geopolymer effectively immobilizes and removes Clenbuterol from the solution, leading to a significant reduction in its concentration. Comparative analysis with PAC demonstrates that the geopolymer performs similarly, if not better, to a commercial adsorbent material in terms of adsorption efficiency.

This study highlights the potential of potassium-based geopolymer as a viable solution for medical waste treatment. The utilization of the robocasting technique allows for precise fabrication, ensuring optimal structural characteristics for enhanced adsorption capacity. The successful adsorption of Clenbuterol by the geopolymer suggests its applicability in removing pharmaceutical waste from medical settings.

The findings presented in this conference presentation contribute to advancing sustainable waste management practices in the medical field. The application of potassium-based geopolymer, produced through robocasting, offers a promising solution for the efficient and environmentally friendly treatment of medical waste. The comparative analysis with PAC emphasizes the geopolymer's potential as a cost-effective alternative for adsorption-based waste treatment processes.

This research opens avenues for further investigation into the broader applications of geopolymer in medical waste treatment, showcasing its potential to address environmental concerns while promoting a circular economy mindset.

Direct Ink Writing of Filter Geometries from Capillary Suspension-based Inks

Authors

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Topic Areas

Poster Session II

Abstract

The capillary suspension concept enables the manufacturing of sintered filters with high open porosity and a more homogeneous microstructure when compared to other traditional means of producing porous ceramics, leading to a better filtering performance. Capillary suspensions are ternary liquid-liquid-solid systems, in which the addition of a secondary fluid, immiscible to the main phase, changes the flow properties of the suspension drastically. This is due to the formation of a sample spanning particle network controlled by strong attractive capillary forces coming with the addition of the second immiscible liquid. Capillary suspensions display a high yield stress and strong shear thinning behavior, which makes them good candidates for extrusion-based 3D printing processes, with superior shape accuracy. The network structure also reduces shrinkage and suppresses crack formation, making capillary suspensions ideal precursors for manufacturing of highly porous bodies, with the possibility to tune porosity and pore size, simply by varying particle size and volume fraction. In this work, we will present possible filter geometries, not achievable by traditional fabrication processes, 3D printed using capillary suspension-based inks as feedstock. Porosity and pore size will be analyzed and their influence on permeability will also be addressed.

Influence of Various Mixing Procedures for Direct Ink Writing of Ceria Stabilized Zirconia

Authors

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Topic Areas

Poster Session II

Abstract

Ceramic dental implants are becoming an increasingly viable treatment option for tooth loss in adults. The most promising ceramic material is 3Y-TZP due to its mechanical and aesthetic properties. Still, low-temperature degradation (LTD) remains a potential disadvantage, which is even more severe in the wet oral environment. To prevent failure due to LTD, the use of ceria-stabilized zirconia is proposed as a hydrothermally stable alternative material.

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

In this work, a water-based hydrogel (Pluronic F-127) was used as a carrier for ceria-stabilized zirconia-ceramic powders, allowing for fine adjustment of the suspension rheology while maintaining paste stability. Dispersion amount and technique affect the stability and viscoelastic behaviour of the aforementioned paste. Particle size distribution, zeta potential, and rheology analysis were used to study the impact of various dispersion and mixing procedures on paste properties and for subsequent procedure optimization. Additionally, SEM, μ CT characterization, and density measurements were performed on sintered samples in order to evaluate printing defects.

Filament-Based Additive Manufacturing of Refractory Products for Demanding Thermo-Mechanical Conditions

Authors

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Topic Areas

Poster Session II

Abstract

The development of filament system for manufacturing of ceramics and steel-ceramic composites has been carried out. Elaborated filaments consisted of approx. 52 vol% of solid phase and 48 vol% of polyethylene-based binder, with addition of cellulose, lignin sulfonate and stearic acid. The binder composition was adapted to enable solvent-free debinding process. For this purpose, in-depth DSC/TG thermal analyses focusing on the thermal stability of the filament over the whole temperature range of thermal treatment were carried out. The production of the filament was performed via winding aided filament extrusion setup.

Selected filaments based on the AR78 spinel were manufactured and subsequently printed into form of casting nozzles using Xerion Fusion Factory Printer. Sintered nozzle prototypes were subjected to thermal shock at 850 °C. For the evaluation of the impact of the thermal shock on their mechanical and structural properties using splitting tensile and compression strength tests, SEM analyses and computed tomography were performed. The final appliance test was carried out in the steel casting simulator via pouring of approx. 100 kg of steel at 1620 °C through the preheated to 600 °C nozzle.

Elaborated FFF-technique with internal layer-to-layer structure allows the production of thin-walled refractory products with an excellent thermal shock resistance and capability of withstanding refractory working conditions due to purposeful incorporation of functional porosities. The filament-based additive manufacturing of complex refractory parts gives them an exceptional lightweight potential. Developed filament enables successful utilization of broad selection of fine-grained ceramic and metal powders for production of multi-functional refractory parts intended to work in demanding metallurgical and electrochemical processes.

Experimental approach to analyze the processing conditions for cement-based materials in binder jetting 3D printing process

Authors

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Topic Areas

Poster Session II

Abstract

Cement-based materials can be produced using the promising 3D printing technique known as binder jetting (BJ3DP). However, there is a lack of knowledge regarding the processing conditions that influence the characteristics of printed parts. This study aims at investigating how different processing parameters affect the final properties of cement-based products in BJ3DP. Commercially available quick setting cement and siliceous sand were used. The curing medium, water-to-cement ratio, and aggregate sizes were among the factors that were considered. For different processing conditions, flowability, compressive and flexural strength, and dimensional accuracy were measured. The research showed that the properties of printed parts are significantly influenced by the processing conditions. The results show that larger water-to-cement ratio improves the mechanical performances, the improvement being clearer using coarser quartz sand. The use of finer quartz sand also results in limited dimensional accuracy.

Hydroxyapatite and Tricalcium Phosphate Sinter-Joined with Zirconia to Selectively Enhance Large Bone Implants Manufactured by Digital Light Processing-based Vat Polymerization

Authors

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Topic Areas

Poster Session II

Abstract

Natural bone has the capacity to self-repair as long as the defect is small. However, once the defect exceeds a critical size, the body needs artificial support to restore the missing structure. Artificial bone replacement is designed to regenerate damaged material by mimicking natural bone tissue in structure and mechanics. The implant serves as a replacement for the missing endogenous bone and must withstand applied loads, including handling of the implant before and during surgery, as well as the stress needed to keep the implant in place. Structural similarities such as a specific degree of porosity facilitate bone ingrowth and vascularisation.

Since every patient, and therefore every bone defect, is unique, additive manufacturing (AM) has great potential for personalized and customized solutions in implantology. We use digital light processing (DLP) based vat polymerisation, which selectively cures photopolymers filled with ceramic powders layer by layer. The excellent geometric resolution of the process enables precise replication of the patient's complex anatomy.

Hydroxyapatite or tricalcium phosphate ceramics have impressive properties, such as osteoconductivity or osteoinductivity, and bioresorbability. However, their poor mechanical properties restrict their use to small bone replacement implants. Zirconia, on the other hand, has high compressive and flexural strength and is also biocompatible. Therefore, we fabricated pieces of hydroxyapatite and tricalcium phosphate joined with zirconia during the sintering process to selectively enhance specific areas. We investigated different designs, sintering temperatures, and holding times.

Shaping of dense silicon carbide by paste-based 3D micro-extrusion

Authors

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Topic Areas

Poster Session II

Abstract

Silicon carbide (SiC) is one of the most promising materials for thermal management devices due to its superior thermophysical properties. However, conventional powder processing methods impose restrictions on design flexibility thus limiting the ultimate performance of thermal components. Additive manufacturing (AM) is emerging as a solution to overcome these constraints for applications demanding complex geometries, making it an excellent approach for producing high-performance SiC thermal management devices. However, AM of SiC by laser powder bed fusion and stereolithography, the most commonly employed AM technologies, is highly challenging due to its high melting temperature and opacity to UV light, respectively. On the other hand, 3D micro-extrusion is a versatile extrusion-based AM technology that offers great potential in coupling the conventional colloidal processing of SiC and AM. 3D micro-extrusion involves layer-by-layer deposition of powder-loaded feedstock paste extruded through a fine nozzle followed by debinding and pressureless sintering.

The present work aims to develop a complete processing route for 3D micro-extrusion of highly dense silicon carbide structures with superior functional and mechanical properties. This involves the development of the paste along with the optimization of the printing and thermal treatment processes. The development of an optimized paste entails the selection of the appropriate starting materials (SiC powder, sintering aids, binder, and dispersants), optimization of powder loading, and mixing procedures. For printing by 3D micro-extrusion, the rheology of the paste is of utmost importance. Furthermore, producing dense parts requires optimization of the printing parameters to obtain minimal printing defects in the green state. Lastly, densification of the printed parts is performed using two conventional routes, i.e. liquid phase sintering and liquid silicon infiltration, to comprehend the feasibility and efficiency of the methods in producing high-quality dense silicon carbide. The fabricated parts are characterized for structural, microstructural, mechanical properties, and dimensional accuracy through shrinkage evaluation.

Multi-material metal-ceramic components manufactured by Selective Powder Deposition followed by pressure-assisted sintering

Authors

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Topic Areas

Poster Session II

Abstract

The high efficiency targeted in new electrical machines demands superior fabrication freedom compared to conventional manufacturing processes in terms of design and materials selection. Electrical machine cores are traditionally manufactured by stacking laminated electrical steel (FeSi) sheets with an insulating coating which impedes the flow of eddy currents. This manufacturing route restricts the Si content in the soft magnetic material to less than 4wt% in order to maintain minimum workability that is necessary for conventional steel sheet processing. However, electrical steel with higher Si content, i.e. Fe-6.5wt%Si, represents the optimized grade due to its zero magnetostriction and higher electrical resistivity, which further reduces the eddy current losses.

Additive Manufacturing (AM) technologies offer the possibility to fabricate complex geometries and to process high Si content steels. The manufacturing of these cores structures with insulation layers implies the co-printing of two different materials (metal and insulating material). However, multi-material printing using the most pronounced direct AM process, laser powder bed fusion, is challenging if a ceramic material is chosen. On the other hand, indirect AM technologies allow to print a broad range (including FeSi) and combinations of materials.

The present work shows the manufacturing of multi-material components using an indirect powder bed-based AM technology named Selective Powder Deposition (SPD) followed by pressure-assisted sintering. Layers of FeSi and ceramic powder were alternatively deposited in a graphite die with controlled thicknesses in order to build a multi-layered metal-ceramic part. Dense and crack-free laminates were obtained upon consolidation using spark plasma sintering (SPS). Good adhesion between the FeSi and ceramic layers was realized thanks to the mechanical interlocking given by the powder deposition method. Moreover, complete insulation of the metal layers was achieved. The resolution and compatibility of the layers along with the crystallographic texture of the magnetic material were evaluated using microstructural characterization techniques.

Adding a low-cost heating system to a DLP printer reduces the viscosity and increases the printability of ceramic suspensions.

Authors

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Topic Areas

Poster Session II

Abstract

The research on ceramic vat-photopolymerization has grown exponentially in the last few years. The high resolution, great geometry freedom, and excellent mechanical properties after post-processing are the major benefits of this technic compared to other additive manufacturing technologies. Additionally, some printers with this technology have simple layouts with few moving parts and are commercially available at a low-cost, especially the bottom-up mask projection types (also known as DLP). However, these ordinary 3D printers usually have difficulty handling very viscous suspensions due to the excessive detachment forces generated during the separation stage (between the last cured layer and transparent window) along with the insufficient resin flow to fill in the new gap for subsequent layers. Consequently, to provide low viscosity, the amount of ceramic powder that can be loaded into the resins is limited, especially when nanopowders are present. As a solution, we upgraded a DLP 3D printer by adding a heating system device, composed of low-cost components widely available: a 12 V DC supply, a PTC heater with a fan, a temperature controller with a relay, a pair of digital thermometers, and 3D-printed supports. In the poster, we present the layout of the system and its simple operation. Additionally, as a demonstration, we present the results related to a suspension loaded with 40 vol% of calcium carbonate. In this case, increasing the temperature from 20°C to 50°C resulted in a viscosity reduction of about 80%, following an Arrhenius equation. The viscosity reduction increased the printability of the material and facilitated the removal of uncured material from channels and holes, enhancing the printing resolution significantly. The proposed system was easy to set up and efficient in reducing the viscosity of the ceramic suspension. Therefore, we envision that other researchers could benefit from this idea and adapt it to their 3D printers.

Direct Ink Writing of inorganic glasses out of aqueous inks based on PEO-PPO-PEO copolymer

Authors

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Topic Areas

Poster Session II

Abstract

While the research on additive manufacturing techniques focuses mostly on processing materials such as metals, ceramics and polymers, there is still a need to increase efforts in printing inorganic glass materials, especially elements with high optical quality intended for photonic applications.

In this work we investigated a concept of 3D printing of glass materials using the Direct Ink Writing method with the aim to obtain glass samples with high optical quality. The research was focused on exploring aqueous suspensions based on the water - glass powder - Pluronic F - 127 system. Depending on the pH of the suspension and use of different additives, Pluronic F - 127 may act differently. One way is formation of the hydrogel using high loading of the polymer. Inner structure of formed hydrogel depends on the temperature and the concentration of the polymer. This two parameters helps to tailor the rheological properties of the inks. On the other hand, Pluronic F - 127 may also act as a crosslinking agent, depending on the functional groups on material's surface. Both paths gives a possibility to suspend different glass powders, forming stable inks for 3D printing. In the presented study, commercial fumed silica and self - prepared silicate glass powders were used to prepare aqueous suspensions. Firstly, the rheological properties of the hydrogels were investigated using rotational and oscillatory measurements. Afterwards, different inks containing both types of amorphous powders were prepared. Obtained materials were processed into samples, and their rheological properties were investigated to find the correlation between their printability and rheological properties. 3D printed samples were characterized using differential scanning calorimetry to investigate thermal decomposition of organic components. Based on calorimetric measurements processes of firing samples were proposed. Finally, all materials prepared were investigated in terms of their structure (XRD) and microstructure.

Morphological comparison between thermoelectric films deposited by aerosol jet printing (AJP) and spin coating

Authors

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Topic Areas

Poster Session II

Abstract

Nowadays energy and pollution crisis raised interest towards alternative ways to harvest energy, like thermoelectric conversion of waste heat into electrical energy (i.e., thermoelectric generators, TEGs). The most interesting one is the direct conversion of energy by employing lightweight and quiet devices, without moving parts, therefore at zero green-house gases emissions. Owing to their thermoelectric properties, the most used and studied materials are bismuth telluride and its alloys (p-type $\text{Bi}_x\text{Sb}_{2-x}\text{Te}_3$ and n type $\text{Bi}_2\text{Se}_{3-x}\text{Te}_x$). Spin coating is a well-known technology that allows to easily deposit a uniform film by dispensing a colloidal suspension on any spinning substrate. Aerosol jet printing (AJP) is based on atomization and deposition of a colloidal suspension of a powder with a particles size (d_{50}) lower than 500 nm. The deposition by AJP of thermoelectric films starting from a milled powder is unprecedented, and in this preliminary study the morphology of the deposited films was compared with the product of a well-established technology such as spin coating. N-type and p-type bismuth telluride suspensions in organic carriers were prepared; the Bi_2Te_3 -based sub-micrometric powders were obtained by ball milling of the respective ingots. The carriers were composed by ethanol, and by a mixture of ethanol, ethylene glycol and glycerol, respectively. Different stabilizers were tested for the n and p-type powders. The films were characterized by means of optical profilometry, SEM and EDS analysis, and optical microscopy. The homogeneity and uniformity of the aerosol jet printed films was lower than for the spin coated films. This study highlighted the need to design the rheological properties of the suspensions for AJP, since the inhomogeneities of the films were due to the agglomeration of the powder, and to accumulation of the carrier (i.e., in search of a more efficient stabilizer, and higher surface tension).

Thermal shock behavior of 3D-printed alumina ceramics with spatially tailored porosity

Authors

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Topic Areas

Poster Session II

Abstract

Ceramic materials are widely known for their great thermal and chemical properties, such as high-temperature stability and corrosion resistance. However, ceramics are inherently brittle and exhibit large scatter in mechanical strength. The initiation of damage depends on the type of loading, components' geometry, among other factors. In particular, thermal shock associated with rapid temperature changes is one of the main critical loading scenarios for ceramic components.

Finding alternative strategies to increase the thermal shock resistance in ceramic parts is crucial for industrial applications, where components may be subjected to rapid temperature changes. Introducing porosity in a ceramic material has been proven as a positive way to improve its thermal shock resistance. A strategy to increase the structural integrity of porous ceramics may be thorough designing with gradients. Porosity-graded alumina ceramics have shown promise in applications such as filters, membranes, coatings, and others. Lithography-based ceramic manufacturing (LCM) is an up-and-coming technology that enables multi-material printing with discrete and smooth gradual change of materials and/or material properties within the volume of ceramic components.

Novel alumina-based laminates with embedded porous layer regions as well as porosity-graded alumina samples are investigated. The layered samples with varied designs together with the (reference) monolithic samples are fabricated using the LCM -technology. The approach combines the porosity and multi-material printing approaches (i.e. layer-by-layer and within-layer) to achieve a new 3D-printed alumina-based material with tailored porosity. The different sample designs are tested under biaxial bending and the strength parameters (i.e. characteristic strength and Weibull modulus) are determined. Thermal shock tests are performed at selected temperature differences and the corresponding strength degradation of the layered porous designs are investigated and compared to the monolithic samples. The advantages of the LCM-technology together with the capability of tailoring the porosities may open new application fields especially for alumina ceramics.

Preparation of BaTiO₃ suspensions containing platelet particles for textured ceramics using digital light processing

Authors

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Topic Areas

Poster Session II

Abstract

Textured piezoceramics with a crystallographically oriented structure exhibit piezoelectric properties that approach the properties of single crystals. However, the optimal ceramic structure orientation providing the ferroelectric domain configuration with the maximum piezoelectric response must be found for each particular material. 3D printing techniques can be used to prepare textured piezoelectric materials with tunable texture orientation. The work focuses on preparing photosensitive suspensions suitable for 3D printing using digital light processing (DLP) method. The BaTiO₃ suspensions containing isometrical BaTiO₃ particles and platelet BaTiO₃ particles were prepared and tested as a proof-of-concept system for further study on the 3D printing of textured piezoelectric materials. The studied system is based on a mixture of acrylates containing a photoinitiator system and additives, enabling the preparation of ceramic suspension with a high solid loading of BaTiO₃. The thermal debinding process of the proposed system(s) and curing characteristics were studied, and the effect of the curing process and thermal debinding on the final quality of sintered samples was described.

Ceramic composite filament for wide-spread commercial application

Authors

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Topic Areas

Poster Session II

Abstract

The Fused Filament Fabrication method has become increasingly popular since the introduction of the RepRap concept in 2004 and the expiration of the original FDM Stratasys patent in 2009. This has led to development of some other derived methods, such as Fused filament fabrication of ceramics, which uses composite ceramic filament as the feedstock for 3D printing. Although the average 3D printer user can currently purchase one of several available filaments for ceramic production, multiple available materials require either modifications of the used 3D printer or specialized debinding/sintering equipment - including, but not limited to the use of refractory ballast material and very slow heating rates - ca. 60 K/hour or less and/or the use of solvents for two-stage debinding process,

In this work we demonstrate preparation of ceramic bodies from filaments that do not require ballast material for debinding/sintering process. The used material also allows for relatively high debinding heating rates of approx. 300K/h for samples with 60% rectilinear infill.

The prepared ceramic also requires sintering temperatures of less than 1350 degree celsius, making it viable for wide-spread application - the common ceramic sintering kilns usually do not allow use of temperatures above 1350 degree celsius.

3D-printing of SiC by Digital Light Processing

Authors

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Topic Areas

Poster Session II

Abstract

SiC is a covalent ceramic with a high refractoriness and decomposes to volatile Si-species and graphite at temperatures above 2300°C. There are challenges with forming and densifying SiC, and to obtain the final geometry for a given application. Time consuming green machining and post sinter treatment are often required. To reduce the time related to production of green bodies and surface treatment needed during processing of technical ceramics, 3D-printing with Digital Light Processing (DLP) has been successfully applied for alumina.[1]. Our aim is to develop a DLP-process for SiC to produce sinterable green bodies with appropriate surface finish.

DLP-printing depends on successful UV-triggered polymerization of the added photomonomers. The UV penetration depth, and therefore the rate of polymerization, is determined by optical properties such as the transmittance of the ceramic particles. Optical properties are strongly correlated with the purity of the starting powder. In this work 99.99% SiC with particle size 500-700 nm has been chosen for its purity and high sinterability.

Dispersions were prepared by fist mixing water, dispersant (amine) and the SiC-powder, and to this dispersion, the photomonomer HDDA (1,6-hexanediol diacrylate) was added and thoroughly mixed. Solid lading was 70 wt.% to ensure sinterability of the printed green body. Stability and rheology properties depend on deagglomeration of the SiC-powder and an optimized recipe was determined using different types of polyester-based dispersants.

Results from the optimized dispersions printed with DLP will be presented and discussed with respect to rheology and green body properties.

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Material extrusion with ceramic filaments

Mechanical properties of dental zirconia ceramics fabricated by additive manufacturing

Authors

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Topic Areas

Material extrusion with ceramic filaments

Abstract

Since the established computer-aided milling (CAM) of dental prostheses from zirconia ceramics produces a lot of waste material and lacks the possibility of forming complex-shaped dental parts, emerging additive manufacturing (AM) brings new opportunities to fabricate dental prostheses with different designs and minimal thicknesses complying with the concepts of minimally invasive dentistry. Although with high expectations, due to the field's novelty, the AM fabrication of dense bulk ceramics suitable for dental applications confronts some challenges, such as pore formation and layer delaminations, affecting the materials' integrity. The study aimed to evaluate the effects of different AM fabrication methods, namely vat photopolymerization (VPP) and material extrusion (MEX), on the mechanical properties of dental zirconia ceramics.

Disk-shaped specimens (n=30 per group) were fabricated from 3 mol.% yttria-zirconia by VPP (CeraFab 7500), MEX (Prusa i3 MK3S), and uniaxially dry pressing (DP) (147 MPa, 30 s) that served as a control. Specimens were analyzed for density, porosity, grain size (SEM), and biaxial flexural strength (ball-on-three-balls method; 1 mm/min) coupled with the Weibull analysis and fractography (SEM). ANOVA with Tukey's post hoc was used to determine the differences between different fabrication methods ($p < 0.05$).

DP zirconia exhibited the highest characteristic strength (σ_0) and reliability (m), followed by VPP and MEX. The prevailing critical flaws for VPP, MEX, and DP were process-related; flaws were substantially larger in specimens fabricated with VPP and MEX.

Compared to DP, AM zirconia performed mechanically inferiorly. However, according to international standards, AM zirconia still reached clinically-acceptable strength.

Silicon carbide additive manufacturing for space mirrors

Authors

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Topic Areas

Material extrusion with ceramic filaments

Abstract

Silicon carbide (SiC) has become a commonly used technical ceramic in various fields such as space mirrors applications [1]. This material is chosen due to its low density, stiffness, good mechanical and thermal properties and excellent oxidation stability.

The traditional fabrication of SiC ceramic includes the forming and machining of a green body, sintering (pressureless, hot pressing or reaction...), grinding and polishing [2]. The emergence of additive manufacturing (AM) technologies has provided new fabrication opportunities. These technologies are able to achieve new designs, which are of great interest for the conception of complex-shaped space mirrors [3].

This paper presents an evaluation of the possibility to form SiC space mirrors using a filament based direct AM method: Fused Deposition Modeling (FDM). A polymer filament loaded with ceramic powder is extruded from a heated nozzle on the tooling platform layer by layer [4]. The formed ceramic green body is chemically debinded to extract the polymer and then sintered at high temperatures (>2000°C).

Different heat treatments cycles have been tested. SiC components microstructural and mechanical properties – such as density, hardness, Young modulus, bending strength – have been measured. These results show promising results with grain size around a few micrometers, density above 94% and very high hardness. The measured properties are comparable with those of traditional sintered SiC ceramic parts.

Elaboration of Metal-Ceramic Multi-Material Refractory Components manufactured via Fused Filament Fabrication Technique for Corrosive Working Conditions

Authors

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Topic Areas

Material extrusion with ceramic filaments

Abstract

Fused Filament Fabrication (FFF) based manufacturing of refractory ceramics and metal-ceramics composites as well as the approach to their combination into multi-material functional part has been investigated.

Two filaments differing from utilized solid phase were prepared: the AR78 alumina rich spinel with addition of 3 vol.% TiO₂ powder and the 60/40 vol.% 316L/AR78 steel-ceramic composite. The binder system of the filament was based on the polyethylene mixture with addition of steric acid, lignin sulfonate and cellulose powders. The debinding of the filaments was performed without any solvent based pre-debinding steps and was based on the DSC/TG analyses. The work presents detailed manufacturing process including the production of filaments via multiplied extrusion, selection of most favorable 3D-printing parameters as well as discussion on the most adequate thermal treatment routes for debinding and sintering.

For investigation of mechanical properties of printed samples compressive, three-point bending and splitting tensile strength tests were performed. The results revealed very good mechanical properties of samples printed from AR78-ceramic filament sintered at 1400 °C – common sintering temperature of steel and steel-based composites. The effectiveness of the debinding and sintering processes were additionally evaluated by the measurement of residual carbon content after each thermal treatment process.

The materials were predestinated for demanding thermal conditions and very corrosive alkali salt-based atmospheres. The corrosion test of multi-material part was performed by the immersion of the sample in BaSO₄ salt at 850 °C for 2 h. The analysis of the sample prior and posterior to the corrosion test was performed using Computed Tomography based X-Ray imaging and SEM microscopy.

The results shown successful combination of two dissimilar materials into one multi-material part with metal-ceramic core and corrosion resistant ceramic shell for future development of high performance

functional refractory products.

Space

3D-printing of lunar regolith ceramics with high mechanical properties via a stereolithography-based approach

Authors

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Topic Areas

Space

Abstract

Lunar regolith, a material that is ubiquitously present across the surface of the Moon and has been formed over billions of years through space weathering of the lunar surface, holds immense potential as a valuable resource for the sustainable development of lunar infrastructure. Its abundance on the lunar surface makes it an ideal raw material for the implementation of the In-situ Fabrication and Repair (ISFR) strategy, particularly for long-duration crewed lunar exploration missions. In turn, additive manufacturing with lunar regolith enables future astronauts to efficiently produce a wide range of items, including spare parts, instruments, and essential infrastructure elements, on demand and at a rapid pace. This paper presents a method for producing dense ceramic components with high mechanical strength and good precision, out of LHS-1 lunar regolith simulant, using laser stereolithography. The study demonstrates that through the use of suitable grinding and sintering parameters, precise and dense ceramic parts with intricate designs can be produced via 3D printing of lunar regolith. It was found that obtained samples pose a relative density of 97% and a micro-hardness of more than 730HV. It was found that the manufactured regolith ceramics yield up to 380 MPa of compressive, 88 MPa of tensile, and 152 MPa of flexural strength, making it a perspective ceramic material for a wide range of applications on the lunar surface.

Habitat construction on Mars using material extrusion AM of dried clay adobe structures under simulated Martian conditions

Authors

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Topic Areas

Space

Abstract

NASA's Artemis program is currently paving the road toward a human settlement on the Moon and Mars after that. With spaceflight's physical and technical constraints, long-duration flights and prolonged stays on the red planet should be expected. In-Situ Resource Utilization (ISRU) is thus of utmost importance for feasible and sustainable space exploration. On ISRU for AM, material extrusion is the preferred AM technology for building infrastructure in space because of its great scalability, ease of use, wide range of material processing capabilities, and flexibility for harsh circumstances, including vacuum and low gravity scenarios [1]. We will discuss a novel clay simulant approach that combines highly specialized clays from the Source Clays Repository with pre-existing planetary simulants, such as those from the MGS-1 family with nontronite, the dominant clay species on Mars [2], and other clay species found in the space environment. With spectral evidence of hydrated minerals on Mars suggesting the presence of liquid brines near the surface [3], we developed a material system using Martian brine water for material extrusion, employing a brine simulant from Nuding *et al.* [4]. The stability of salt clay pastes was assessed, and the effect of clay species and brine on rheology was characterized. Extrusion of non-brine and brine pastes produced adobe structures with compressive strengths typically >10 MPa, more than twice as strong as traditional adobe bricks used to construct clay structures on Earth [5]. Finally, the effect of various-sized particulate regolith fillers on the compressive strength of adobe will be discussed.

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Emerging, hybrid and multimaterial AM technologies

The potential of liquid feedstock for the additive manufacturing of ceramics

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Topic Areas

Emerging, hybrid and multimaterial AM technologies

Abstract

Additive manufacturing of polymeric materials has reached a far greater maturity with respect to ceramics, the latter being limited by their high melting temperatures and conventional sintering from powder feedstock. Processing slurry-based feedstock, in which powders are present, poses additional challenges: a high amount of powder is required to promote densification, and results in high viscosity, scattering and sedimentation phenomena in vat photopolymerization processes, as well as clogging problems at the nozzle for extrusion-based processes. Our research activities has therefore been focusing on additive manufacturing of ceramics from liquid feedstock; in particular, we investigated the use of preceramic polymers as well as geopolymers and sol-gel solutions. Despite the many advantages related to their liquid nature, there are also some challenges related to the reactivity of such systems and to the removal of solvents and binders usually present. Here, we present our strategies for producing high quality ceramic components using a variety of liquid feedstock and different additive manufacturing techniques, from direct ink writing and digital light processing to robotic and volumetric additive manufacturing.

Tailoring structural and functional properties of AM ceramics through rapid sintering

Authors

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Topic Areas

Emerging, hybrid and multimaterial AM technologies

Abstract

The thermal post processing (TPP) of AM ceramics fabricated through vat photopolymerization (VPP) is time and energy consuming. TPP may be divided in two steps of (i) binder removal and (ii) sintering. The latter is mainly performed in conventional sintering furnaces or in some cases through hot isostatic pressing. Here the sintering step takes typically several hours or days. In this work the aim was to investigate a pressure-less spark plasma sintering set-up (PL-SPS) to densify 3D-printed ceramics within minutes and tailor its microstructures regarding structural and functional properties.

The VPP process was used to additive manufacture pure alumina as well as a composite of alumina and a MAX phase. The samples were rapidly sintered in vacuum in the PL-SPS set-up.

In the case of alumina, the rapid heating was performed at 450°C/min to promote a fine-grained microstructure. Dwell times between 2 and 8 min were applied to investigate the microstructure evolution. Optimal conditions were found at 1500 °C with 8 min dwell time, leading to grain sizes below 1 μm, with a mechanical strength improvement of > 200 MPa compared to conventionally sintered samples. In the case of alumina-MAX phase composites, different compositions were sintered to achieve materials with electrical conductivity and enhanced mechanical response.

This investigation has demonstrated that a pressure-less spark plasma sintering set-up can be used to densify and optimize the microstructures of AM ceramics within minutes. This opens the path for reducing the processing time as well as energy costs for sintering ceramics of complex shapes.

Defect-free Hybrid Manufacturing of Advanced Ceramics

Authors

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Topic Areas

Emerging, hybrid and multimaterial AM technologies

Abstract

Hybrid manufacturing combines two or more distinct manufacturing processes, leveraging the advantages of each process whilst mitigating their limitations. We use this approach to enable the production of functional parts from advanced ceramic materials. Combining material extrusion with subtractive processes allows for the geometric complexity of freeform fabrication with the surface finish and precision characteristic of conventional manufacturing. However, the extrusion process is susceptible to defects that are hard to predict and detrimental to a parts mechanical properties. We propose to address this through automated in-situ monitoring, in line with a general shift towards data-driven manufacturing coined 'Industry 4.0'. Mounting an optical camera and laser on the machine, we capture images and scans of each layer in a part. By comparing this data to the digital model, we aim to evaluate the geometrical accuracy of the part. Defects can be identified, located and measured through image processing. Using closed-loop control, and the flexibility of our hybrid platform, this information can be passed back into the control software, and material can be deposited or removed to correct defects, theoretically enabling defect-free manufacturing. This work demonstrates the reworking of defective layers during hybrid manufacturing, proving the viability of this technology for the creation of defect-free advanced ceramic components for high performance applications.