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98th DKG Annual Meeting



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Grußwort der Programmkommission

Liebe Mitglieder der DKG und Freunde der Keramik,

Cari membri del DKG e amici della ceramica,

hiermit laden wir Sie herzlich zur KERAMIK 2023, der 98. Jahrestagung der Deutschen Keramischen Gesellschaft (DKG), ein. Die Veranstaltung, die allen Keramikern und Keramikinteressierten offensteht, planen wir - erstmalig seit 2019 - wieder als Präsenzveranstaltung.

Gastgeber der KERAMIK 2023 / 98. DKG-Jahrestagung ist die Ernst-Abbe-Hochschule Jena (EAH Jena). Die EAH Jena wurde am 1. Oktober 1991 als Fachhochschule Jena geründet. Hervorgegangen ist sie aus der Ingenieurschule Carl Zeiss in Jena. Seit dem Sommersemester 2012 trägt sie den Namen des in Jena aktiven Wissenschaftlers, Unternehmers und Sozialreformers Ernst Abbe.

Mit 4.500 Studenten (2018) und 450 Beschäftigten fokussiert sich das Studienangebot der EAH Jena auf die Themenbereiche Technik, Wirtschaft, Soziales und Gesundheit und zeichnet sich durch einen hohen Praxisbezug aus. Der Fachbereich SciTec verbindet Naturwissenschaften „Science“ und Technik „technology“. Hier ist die Werkstofftechnik und insbesondere die Hochleistungskeramik beheimatet. Prof. Dr. Jörg Töpfer lehrt und forscht umfangreich zu funktionskeramischen Werkstoffen (Magnet-, Piezo-, Ferro- und Thermoelektrische Werkstoffe, funktionskeramische Schichten und Multilagen-Bauelemente). Auf diesen Gebieten arbeitet die EAH eng mit den Keramikunternehmen und dem Fraunhofer-Institut für Keramische Technologien und Systeme IKTS in Hermsdorf zusammen, die sich zum Netzwerk TRIDELTA CAMPUS HERMSDORF zusammengeschlossen haben. Prof. Dr. Ingolf Voigt, Standortleiter des IKTS Hermsdorf lehrt an der EAH Keramiktechnologie und ist als Co-Chair in die Organisation der Keramik 2023 eingebunden. Als dritte wissenschaftliche Einrichtung unterstützt das Otto-Schott-Institut für Materialforschung der Friedrich-Schiller-Universität Jena die Organisation der Tagung. Prof. Dr. Frank Müller forscht hier insbesondere auf den Gebieten der Oberflächen- und Grenzflächentechnologien und der Synthese keramischer Nanopulver.

Die Stadt Jena ist ein Hightech-Standort mit vielen Industrieunternehmen, darunter große Namen wie ZEISS, Jenoptik und Schott, aber auch vielen kleinen und mittelständischen Unternehmen, insbesondere in den Bereichen Optik und Photonik, Gesundheitstechnologie und Digitale Wirtschaft. Jena ist eine international bekannte Wissenschaftsstadt und beherbergt neben der traditionsreichen Friedrich-Schiller-Universität und der praxisorientierten Ernst-Abbe-Hochschule auch eine Reihe renommierter Forschungseinrichtungen, darunter Institute der Max-Planck- und Fraunhofer-Gesellschaft, der Leibniz-Gemeinschaft sowie des Deutschen Zentrums für Luft- und Raumfahrt (DLR).

Wir freuen uns, insbesondere die Italienische Republik als Konferenzpartner der KERAMIK 2023 gewonnen zu haben und begrüßen damit auch gleichzeitig alle unsere Kollegen aus den europäischen und nichteuropäischen Nachbarländern zur bedeutendsten Keramiktagung in Deutschland.

Wir freuen uns, Sie in Jena persönlich begrüßen zu dürfen

Saremo lieti di accogliervi personalmente a Jena

Im Namen des Programmausschusses



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Greeting by the Program Committee

Dear members of DKG and friends of ceramics,
Cari membri del DKG e amici della ceramica,

We hereby cordially invite you to CERAMICS 2023, the 98th Annual Conference of the Deutsche Keramische Gesellschaft (DKG). The event, which is open to all ceramists and those interested in ceramics, we are planning - for the first time since 2019 - again as a face-to-face event.

The host of KERAMIK 2023 / 98th DKG Annual Conference is the Ernst-Abbe-Hochschule Jena (EAH Jena). The EAH Jena was founded on Oct. 1st, 1991 as the Jena University of Applied Sciences. It emerged from the Carl Zeiss School of Engineering in Jena. Since the summer semester of 2012, it has borne the name of Ernst Abbe, a scientist, entrepreneur and social reformer who was active in Jena.

With 4,500 students and 450 employees, the courses offered at the EAH Jena focus on the subject areas of technology, economics, social affairs and health and are characterised by a high level of practical relevance. The SciTec faculty combines natural sciences and technology. It is home to materials technology and, in particular, advanced ceramics. Prof. Dr. Jörg Töpfer teaches and conducts extensive research on functional ceramic materials (magnetic, piezo, ferro and thermoelectric materials, functional ceramic coatings and multilayer devices). In these fields, the EAH works closely with ceramics companies and the Fraunhofer-Institut für Keramische Technologien und Systeme IKTS in Hermsdorf, which have joined forces to form the TRIDELTA CAMPUS HERMSDORF network. Prof. Dr. Ingolf Voigt, site manager of the IKTS Hermsdorf teaches ceramics technology at the EAH and is involved in the organisation of Ceramics 2023 as co-chair. The Otto-Schott-Institut für Materialforschung at Friedrich-Schiller-Universität Jena is the third scientific institution to support the organisation of the conference. Prof. Dr. Frank Müller conducts research here, particularly in the fields of surface and interface technologies and the synthesis of ceramic nanopowders.

The city of Jena is a high-tech location with many industrial companies, including big names such as ZEISS, Jenoptik and Schott, but also many small and medium-sized enterprises, especially in the fields of optics and photonics, health technology and the digital economy. Jena is an internationally renowned science city and, in addition to the tradition-steeped Friedrich Schiller University and the practice-oriented Ernst Abbe University of Applied Sciences, is also home to a number of renowned research institutions, including institutes of the Max Planck and Fraunhofer Societies, the Leibniz Association and the German Aerospace Center (DLR).

We are particularly looking forward to having won the Italian Republic as a conference partner of CERAMICS 2023 and thus also welcome all our colleagues from the European and non-European neighbouring countries to the conference.

We look forward to welcoming you in person in Jena
Saremo lieti di accogliervi personalmente a Jena

On behalf of the program committee



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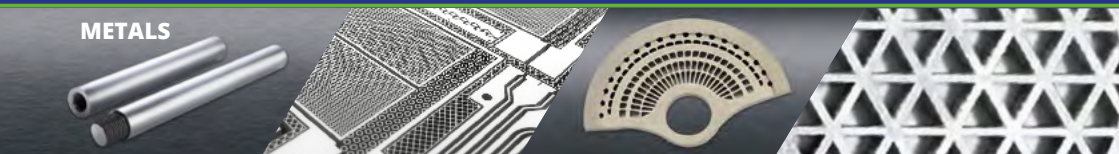
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LECTURES

Production of functional ceramic components by means of thermoplastic Additive Manufacturing and Hybridisation thereof

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Within sinter-based Additive Manufacturing (AM), thermoplastic approaches benefit from a large experience in terms of materials and processes. Therefore these technologies are under investigation in science and industry. The Multi Material Jetting (MMJ) is a droplet-based method, which allows manufacturing of components made of one or more materials simultaneously by droplet fusion, whereby Fused Filament Fabrication (FFF) is a more or less line-based process also exhibiting the possibility of multi material manufacturing. By means of MMJ LTCC substrates have been manufactured sowing an alternative to traditional Multilayer Technology (MLT). The combination of electrical conductive and insulation $\text{Si}_3\text{N}_4/\text{MoSi}_2$ shows the ability for application as spark and plasma plug. Fused Filament Fabrication (FFF) known as one of the most popular extrusion-based AM method was investigated to produce functional ceramic components made of LTCC and two component parts made of conductive and insulating $\text{Si}_3\text{N}_4/\text{MoSi}_2$ as well. For functionalizing the LTCC components were supplemented by MMJ using silver-based feedstocks in green state. This hybridizing allows not only the economic usage of novel materials by MMJ but also covering the circuit with LTCC by means of a productive extrusion process in green state. The first results show that the component exhibit an electrically conductive circuit after co-firing. The two component $\text{Si}_3\text{N}_4/\text{MoSi}_2$ parts were printed, sintered and tested also successfully opening new opportunities for heating applications. It turned out that both thermoplastic AM technologies are suitable to manufacture single material, multi material and hybridised components.

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Electrospun Vanadium Pentoxide Nanofibers as a Photocathode in a Light Rechargeable LIB

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An ever-expanding demand of more efficient energy storage systems pushes the development of next generation batteries. These include the innovative so-called "photo battery", whose electrode combines energy storage with energy harvesting. Lithium-ion batteries (LIB) are currently one of the most promising techniques to do so. The application of ceramic dual-functional materials in photo-rechargeable batteries represents a synergistic concept, using Vanadium pentoxide (V_2O_5) with a suitable band gap (2.35 eV) and good theoretical capacity value (294 mAh g^{-1} for 2 Li^+) as such. We hereby report electrospun, calcined vanadium pentoxide nanofibers (VNF) and carbon coated VNF by plasma enhanced chemical vapor deposition. The carbon coating of the ceramic fibers not only acts as a conductive sheath, but also contributes significantly to maintaining the morphological and structural stability of the V_2O_5 fibers, as shown by long-term stability tests (300 cycles at 0.75 °C) and post-mortem analysis. Under light illumination, the discharge capacity could be increased. In addition, the light-only charged materials could be used to drive an LED and showed promising overall conversion efficiencies.

Cofiring of lead-free piezoceramic KNNLT multilayer actuators with nickel electrodes

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Potassium-sodium-niobate piezoceramics ($K_{1-x_2}Na_xLi_z$) $Nb_{1-y}Ta_yO_3$ (KNNLT) are considered a promising, environment-friendly alternative for lead-zirconate-titanate $Pb(Zr_{1-x}Ti_x)O_3$. For multilayer actuator fabrication, the use of inexpensive base metal alloys (nickel or copper) is desired. This requires firing in reducing atmosphere. For the fabrication of defect-free, high-performance piezoelectric components, a thorough understanding of the complex interplay between piezoceramic oxide and metal layers during debinding, sintering and reoxidation is necessary. Sintering protocols which avoid oxidation of the metal electrodes during sintering at low oxygen partial pressure and to tailor the oxygen vacancy concentration in the piezoceramic upon reoxidation have to be developed.

The KNNLT piezoceramic was successfully cofired with nickel electrodes. Manganese dopant segregation at the interface between the ceramic layer and the nickel electrode was observed. The effect of the firing parameters on the performance of the multilayer and on the distribution of manganese in the KNNLT multilayer was investigated. In addition, different reoxidation regimes were applied to enhance the properties of the multilayers. Through optimizing the firing parameters, a multilayer actuator was obtained, which exhibits a normalized strain coefficient of $d_{33}^* = 200$ pm/V, an effective coupling factor of $k_{eff} = 0.263$ and a loss factor as low as $\tan \delta = 41 \times 10^{-3}$. However, further investigations are required to improve the performance of KNNLT multilayer actuators with nickel electrodes.

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Copper-strontium/magnesium co-doped mesoporous bioactive glass for bone tissue regeneration

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Properties of Mesoporous bioactive glass (MBG) can be tailored by introducing metallic ions such as Sr or Mg and Cu which can enhance its functionalities, including osteogenetic, angiogenetic, and antibacterial properties.

In the present study Cu and Sr/Mg ions (ratio 1:1) co-doped glasses in the system $80\text{SiO}_2-(15-2x)\text{CaO}-5\text{P}_2\text{O}_5-x\text{CuO}-x\text{SrO}/\text{MgO}$ with $x= 0.5, 1$ and 2 mol%, synthesized using sol-gel and evaporation induced self-assembly techniques. XRD confirmed the amorphous nature of glasses, while BET confirmed a high surface area for all co-doped systems, indicating the presence of mesoporosity. MBGs showed in vitro bioactivity when immersed in SBF. SEM images and XRD confirmed the onset of formation of a carbonate apatite layer on the surfaces of CuMg co-doped MBGs was faster than for CuSr MBGs.

Cytotoxicity test revealed the cell viability towards MC3T3-E1 preosteoblast cells above 50% at glass powders concentrations of 1 wt./vol.% or lower for both co-doped CuSr and CuMg powders. CuMg MBG system showed improved osteogenesis (in vitro differentiation and mineralization) activity in comparison to CuSr MBG from Alkaline phosphatase (ALP) study. VEGF release was measured after 3 days of incubation and the cellular response was slightly higher in the presence of CuMg therapeutic ions. The antibacterial study showed a more prominent bacteria inhibition zone with progressive increase of Cu concentration in co-doped MBG systems.

The prepared co-doped 1 mol.% CuMg MBG powders showed enhanced biological functionalities, including bioactivity, cytotoxicity and antibacterial properties, and could be considered as promising materials for bone tissue regeneration applications.

Sodium conducting glass ceramics (NaRSiO, R=Y, Sm, Gd) for use in solid state batteries

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New stationary energy storage systems become important because of the increasing application of renewable energy technologies. Li-ion batteries are known for their high performance but suffer from increasing material costs and use of toxic elements. Sodium-based battery concepts represent an alternative to lithium-based batteries due to the high availability of sodium, lower costs, and better environmental compatibility of suitable active materials. New and innovative sodium battery concepts require dense, solid electrolytes, which act as ion-conducting separators between anode and cathode. Different materials perform as a solid electrolyte, for example sulfides, chalcogenides and oxides. Beside NASICON and Beta-alumina also certain types of glass-ceramics are promising sodium-ion conducting electrolytes. Here sodium rare earth silicates NaRSiO with R= Y, Sm, Gd have been investigated as sintered glass ceramics. The corresponding glass frits in the system $\text{Na}_2\text{O}-\text{SiO}_2-\text{R}_2\text{O}_3$ have been molten and processed as powders. Microstructures have been characterized by SEM/EDX and the phase composition was determined by XRD. High fractions of the conductive phase $\text{Na}_5\text{RSi}_4\text{O}_{12}$ (N5-type phase) have been achieved. Different sintering strategies have been explored to achieve dense microstructures. Ionic conductivities have been analyzed by impedance spectroscopy at different temperatures to deduce activation energies between 0.3 and 0.32 eV. Total conductivities at 30°C for the Gd-samples of 2.1×10^{-3} S/cm and 8.6×10^{-4} S/cm for the Sm-samples have been measured. Thin substrates were produced via tape casting, which are showing equally high conductivities and also a good cycle stability at a current density of 0.1 mA/cm².

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Influence of calcination temperature on the photocatalytic activity of a bio-inspired C,N-TiO₂ photocatalyst for microplastic remediation

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Microplastic particles have become a persistent pollutant in the oceans, and even though the implications of microplastic pollution on human health are still not clear, studies suggest that their ingestion could potentially lead to cancer and interfere with endocrine systems. Photocatalysis has been extensively studied as an environmentally friendly and efficient solution to degrade pollutants in wastewater, including microplastics. Nonetheless, efforts are still being made to find visible-active photocatalysts obtained using low-energy synthesis procedures that use renewable sources to reduce the overall environmental impact of this process. In this work, C,N-TiO₂ photocatalysts was obtained through a hydrothermal synthesis at 150 °C using the extrapallial fluid of *Mytilus Edulis* mussels as doping source. One photocatalyst sample remained uncalcined while the others were calcined at 400, 600 and 800 °C. Their properties and efficiency in the photocatalytic degradation of microplastics under visible light were investigated.

It was found that the calcined samples were significantly more crystalline and had lower superficial area than the uncalcined sample. In terms of photocatalytic activity, results showed that after the photocatalytic treatment, the uncalcined sample achieved 70% mass loss while the calcined samples arrived up to 99% of mass loss of microplastics.

Nickel-chalcogenide thin films for sustainable energy conversion

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Nickel chalcogenides (S, Se) have recently gained popularity in the photoelectrochemical water splitting reaction as alternative materials for noble transition metals. However, the challenge remains in the manufacturing of phase pure nickel sulphide/ selenide thin films. This work provides the basis for the synthesis of a novel chalcogenide-containing nickel precursor $[\text{Ni}(\text{EC}_2\text{H}_4\text{N}(\text{Me})\text{C}_2\text{H}_4\text{E})_2]$ (E=S, Se) using a chelating ligand and a suitable nickel source for reliable material preparation. Preformed metal-chalcogenide bonds enable a direct material processing using soft decomposition parameters. The single molecular sources have been characterized by NMR, single X-Ray, mass and elemental analysis. Crystalline thin films could be obtained from solution based synthesis using spray pyrolysis methods. NiS and NiSe have been confirmed by XRD measurements. Optimized thin nickel-chalcogenide films have been used in photoelectrochemical water splitting for sustainable energy conversion.

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Metal Alkoxides Derived High Entropy Oxides: A Design Strategy For Efficient Oxygen Evolution Reaction (OER) Electrocatalysts

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High entropy materials (HEO) have gained enormous attention in recent years due to various application possibilities, based on the different attributes of each implemented metal cation and the synergy in between. The tailorable characteristic of the materials makes them a great opportunity, to choose and tune the metals and thus the properties. Therefore, different precursors and the synthesis approaches are finetuned to obtain a high entropy system, by using metal alkoxides, especially metal isopropoxides of titanium, vanadium, tin, zirconium, and hafnium. Furthermore, the synthesized HEO was analyzed with different characterization methods like X-ray photoelectron spectroscopy (XPS), X-ray absorption spectroscopy (XAS), X-ray diffraction (XRD), and scanning electron microscopy (SEM). The material was tested for oxygen evolution reaction since high entropy materials have also gained high interest in terms of catalytical activity in the field of renewable energy. Based on that, the electrochemical water splitting reaction was catalyzed by the synthesized HEO $[(V,Sn,Ti,Zr,Hf)O_2]$ and superior catalytical activity with an overpotential of 301 mV and an electrochemically active surface area of 3375 Fcm^{-2} could be obtained. Furthermore, the material is tested on stability by a chronoamperometry measurement, showing great stability over 90 hours.

Thermal stability of power losses in Mn-Zn-Ferrites for power electronics

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Mn-Zn-ferrites are used as soft magnetic components for power electronics. We have examined the aging behavior, i.e. the increase of power losses with time, of Mn-Zn ferrites in some detail. It is shown, that the power losses and permeability degrade significantly over a period of several months when subjected to higher operating temperatures of 150 °C to 200 °C. As a consequence, ferrite components (e.g. transformers) require more energy over time and finally reduce battery lifetime. It was found that Co-substituted Mn-Zn-ferrites, which exhibit a broad temperature variation of the power loss, show much larger drift of losses as compared to Co-free ferrites.

In order to clarify the origin of this degradation behavior we studied the variation of Fe²⁺ concentration vs. aging time using thermal analysis, x-ray diffraction, redox titration and Seebeck coefficient measurements. All measurements revealed that the Fe²⁺-concentration remains unchanged with time. Thermogravimetric characterization as function of T and p_{O₂} reveal the nonstoichiometry and point defect concentrations; however, no significant variations of cation vacancy concentrations between Co-containing and Co-free ferrites were found, eliminating different ion diffusivities as source of the observed aging phenomena. To reduce the drift of power losses, new Mn-Zn ferrite compositions with various concentrations of TiO₂ or SnO₂ dopants were fabricated. The effect of sintering parameters, e.g. temperature and oxygen partial pressure, on the power losses were studied. The aging behavior of these new ferrite compositions is significantly reduced and amounts to 5% as compared to 20% of the Co-containing Mn-Zn-ferrite.

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Hierarchically Structured Nano Porous Cathode Materials - Potential, Limits, Applications

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Typical cathode materials for batteries have micron sized particle diameters. In contrast, nanoscale cathode materials are able to achieve very high rate capability and significantly improved cycling stability due to short diffusion paths and high stress resistance. However, inefficient packing density and increased additive demand limit the achievable energy density. Hierarchically structured cathode materials represent an approach to combine the advantages of both worlds and allow nanoparticles to gain access as electrode materials in batteries. For this purpose, the nanoparticles are aggregated into secondary particles in the microscale, for which a comparable processing can be applied as for established materials. Characteristic of the hierarchically structured particles is the high share of open intraparticle porosity. It leads to lower energy density, since a higher residual porosity remains in the electrode despite an untypical plastic deformation behavior of the secondary particles. However, the ability to create favorable conditions for fast charge transport makes the hierarchically structured particles particularly attractive for electrode materials with low conductivity and for post-lithium systems, such as the sodium or magnesium ion batteries, where classical particle structures only lead to electrodes with significantly limited performance. The paper gives an overview of the state of development of hierarchically structured cathode materials with established electrode materials. It also addresses the potential of this particle morphology for future developments in the battery field.

Green hydrogen - key to the energy transition

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The goals are set by the Paris Agreement for the fight against the climate change and the reduction of CO₂ emissions. These goals require not less than the de-fossilisation of all sectors of our traditionally carbon-based society.

Green hydrogen from electrolysis is considered to be the key in this de-fossilisation as it connects the renewable energy sector with a wide range of industrial applications and other sectors like transportation, power generation and building heat and power. Already today hydrogen is produced in large amounts mainly as industrial feedstock. Anyway, hydrogen produced today is 'grey' as it comes from SMR and is accordingly responsible for emissions of around 900 Mt/a CO₂. Converting all of this grey hydrogen into green hydrogen will require around 1 TW of installed electrolysis capacity. As the demand for hydrogen will expand also to other areas as mentioned above it is expected that the hydrogen market will grow sevenfold by 2050.

Following this demand, the challenges are tremendous in view of e.g. sector coupling, hydrogen infrastructure, supply chain topics, availability of material and of course for the required technologies and their ramp-up to world-scale.

Among the various technologies for water electrolysis, alkaline water electrolysis (AWE) is acknowledged to be the most mature right now. Hence AWE will play an important role in the energy transition on short and long term having its strength especially in central and large scale production of green hydrogen.

Anyway, ramping-up productions to gigawatt-scale, be resourceful with metals and noble metals and reduce the TCO (total cost of ownership) needs to be on the list for improvements of all water electrolysis technologies to enable the energy transition.

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Protective Coatings of Cathode Active Materials for Thiophosphate-based Solid-State Batteries

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Solid-state batteries (SSB) are considered as a promising concept for next-generation energy storage systems with high energy density and safety. In comparison to conventional Li-ion batteries (LIB), the liquid electrolyte is replaced by a non-flammable solid electrolyte (SE). The Li-ion conducting SE forms both the separator and a percolating system around the cathode active material (CAM). Potential solid electrolyte classes include oxides, thiophosphates (sulfides), and polymers.

Thiophosphates were chosen for SSB due to their high ionic conductivity. However, the low electrochemical stability of thiophosphates leads to decomposition reactions in contact to CAM such as $\text{LiNi}_{0.8}\text{Mn}_{0.1}\text{Co}_{0.1}\text{O}_2$ (NMC811) during cell performance. The decomposition products form a Li-ion blocking interlayer at the interface of the CAM and SE, thus limiting the power density and long-term stability of SSB. The introduction of thin protective, ion-conducting CAM coatings prevents direct contact between CAM and SE, which enhance electrochemical stability in the cathode and reduce decomposition reactions.

In this study, wet chemical spray coatings of NMC811 with ternary metal oxides (Li-M-O with $M = \text{Nb, Zr, W}$) are analyzed. The compatibility of the coated materials is tested with conventional liquid electrolytes and thiophosphate solid electrolytes. Systematic dependencies of coating thickness on electrochemical properties are discussed to identify optimal coating thickness and process parameters. The C-rate performance of NMC811 is improved by homogenous Li_2ZrO_3 coatings.

Flash Sintering of Ceramics

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Flash Sintering (FS) is a field assisted sintering technique, which enables a sustainable densification of ceramics within a very short time compared to conventional methods. Even if the application of this process is possible and various materials are densified by it, the fundamental mechanisms of the rapid densification remain partly unclear. Beside Joule heating other mechanisms, like electrochemical reduction or phase transitions are proposed by some researchers. However, their connection to FS is still under debate.

In this research various mechanisms in FS, such as electrochemical reduction, phase transition and Joule heating are investigated by DC FS of different materials. The different mechanisms occur, dependent on the material and its properties, but also because of the way FS is achieved. Because of that, a few materials with strongly varying properties, like alumina, yttria-stabilised zirconia or boron carbide, will be discussed. Another influence which is one of the main interests of this presentation is atmosphere. Various experiments showed that dependent on the atmosphere different processes can be achieved or prohibited during FS. Varying atmospheres provide another parameter for controlling FS and could bring FS one step closer to industrial application.

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Investigations on forming crystalline solids made of chemically stabilized cristobalite CSC by means of glass-ceramic process

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Aim of this work is the forming of a chemically stabilized cristobalite CSC via the glass-ceramic route based on the surface crystallisation of high SiO₂-content glasses with limited 1st order phase transition in cristobalite. Furthermore the CSC is characterized by excellent thermal shock resistance and stability up to 1,500 °C and therefore ideally suited for refractory applications. Glass-ceramic process consists of resource-saving and energy-efficient plasma melting process followed by transformation into a compact crystalline material in a crystallization process. Because of new results, the effect of dopant ratios, raw material preparation and ageing temperature on the crystallisation behaviour as well as, because of dilatometer measurements and XRD investigations, the stabilisation mechanism of the beta cristobalite phase and the resulting thermal and mechanical properties are discussed. Further investigations which should lead to a deeper knowledge of the stabilisation mechanism of the beta cristobalite phase in the material and to an optimised crystallisation are presented. As a result of the investigations, which were partly carried out within the framework of the RUBIN alliance SAPHIR High Performance Ceramics from the Tridelta Campus Hermsdorf (funded by the BMBF, FKZ:03RU1U161F), an attempt is made to identify a composition range in which a deformation-free compact CSC glass-ceramic can be produced, which has a very good thermal shock resistance.

Design of new ternary Al₂O₃-Cr₂O₃-TiO₂ material compositions for surface technologies

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Compositions of binary oxide systems, such as Al₂O₃-TiO₂, Al₂O₃-Cr₂O₃ and Cr₂O₃-TiO₂, are well established as materials for thermal spray coating solutions. It is proposed that the addition of the third oxide of the ternary Al₂O₃-Cr₂O₃-TiO₂ system to the binary systems can further improve the coating properties.

The trial-and-error approach of the development of new and alternative materials through experimental feedstock powders is cost-intensive, ineffective, and laborious, as it requires a special particle fraction with good processability and sufficient amounts. Therefore, a reliable pre-evaluation method for the development of new compositions is required. An approach consisting of the preparation of sintering bodies via classical powder processing steps is considered as an appropriate tool for such an evaluation. In case of the ternary Al₂O₃-Cr₂O₃-TiO₂ system the knowledge of the phase relationships is very limited. Thus, such sintered bulk bodies allow to study interactions between the components, their sinterability, and target properties, such as hardness, corrosion resistance in a state close to equilibria, etc. This concept also supports the interpretation of the processes during spraying and the resulting coating microstructure. This study discusses phase formation and properties of selected ternary compositions of the Al₂O₃-Cr₂O₃-TiO₂ system. Some limitations of this approach for prediction of coating properties are critically discussed as well.

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Characterization of a High Fracture Toughness YSZ Ceramic

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Yttria-stabilized Zirconia (YSZ) is used in a variety of structural and biomedical applications due to its excellent mechanical properties and its high biocompatibility. These properties are achieved by stabilizing the tetragonal Zirconia phase down to room temperature by doping with other metal oxides like Y_2O_3 .

This research investigates a newly developed material for commercial use by Treibacher Industrie AG (AuerTec® 2Y-40A). The Yttria content is reduced to 2 mol% which leads to a superior fracture toughness compared to Zirconia materials with higher Yttria content. The material is investigated in regards to its microstructure, mechanical and chemical properties. A sintering temperature of 1350°C is sufficient to reach a dense sintered body with a mean grain size of 0,25 μm and a 4-point-bending strength of 1200 MPa. Further, the Auertec® 2Y-40A shows an outstanding fracture toughness of 17 $\text{MPa m}^{1/2}$ determined by indentation fracture method. These characteristics make the ceramic suitable for demanding applications in industrial fields, cutting tools and consumer goods.

Tensile test of thin 3YSZ ceramic foils for SOC applications – effect of edge roughness on strength

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The production of hydrogen is a crucial process for the energy transition in Germany and for the independence from fossil fuels. High temperature Solid Cells (SOCs) play an important role in this process as they can produce green hydrogen with a high efficiency at $T=850\text{ °C}$ (Solid Oxide Electrolyzer Cells, SOECs) and can also work in reverse (Solid Oxide Fuel Cells, SOFCs). For the cost-optimization SOCs, an important property is the mechanical stability of the cell, which is mainly provided by the cell electrolyte. In this study, the authors investigated the tensile test method to evaluate 3YSZ ceramic foils (thickness $< 90\text{ }\mu\text{m}$) for SOC applications, which allows the evaluation of a large volume of material compared to the more common flexural test. To realize the test, dogbone-shaped samples are coupled with a hook-shaped gripping system using metallic cap stripes for the transmission of load and a swivel head for the misalignment minimization. To evaluate the influence of edge quality on the strength, the samples were cut with a laser and subsequently ground with three grinding tools with different diamond grain size. All ground samples were found to have an average strength above 1000 MPa, while the lasered ones show strengths below 500 MPa. The sample edges were analyzed via SEM to investigate the influence of roughness on strength. For a coarser diamond grain size on the grinding tool the standard deviation is higher, but the strength does not vary compared to the one ground with a finer diamond grain size. Moreover, XRD measurements were performed on the sample edges to evaluate the phase transformation (tetragonal \rightarrow monoclinic 3YSZ) during grinding.

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Inductive heating of preceramic polymers for the repair of ceramic matrix composites

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In the field of ceramic matrix composites, a change in thinking towards sustainability must take place in the near future. Currently, fiber-reinforced silicon carbide ceramics are declared as rejects and are discarded when slight damage occurs during production or in the period of use. Repair is often impossible or too costly due to the need to reprocess the entire component. Here, induction heating offers a promising solution since it allows to heat and process only a defined part of the component. We describe how preceramic polymers, e.g., phenolic resins, can be modified by specific mixing with additives such as graphite powders, silicon carbide powders, or carbon fibers to enable their inductive coupling to an electromagnetic field. Inductive heating was successfully established for crosslinking and pyrolysis of these preceramic polymers and tracked using a pyrometer and an infrared camera. A high residual carbon content of the pyrolyzed preceramic polymer and a pore-free connection between the ceramic matrix composite substrate and the pyrolyzed polymer were achieved, which was proven by thermal analysis and non-destructive testing. Induction heating will also be performed for siliconization of the carbon material for reaction to a silicon carbide matrix. The presented concept of inductive heating opens a new route for the local repair of CMCs.

Novel lead-free, ferroelectric $\text{BaCu}_{0.33}(\text{Nb}_x\text{Ta}_{1-x})_{0.67}\text{O}_3$ perovskites

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The applications of ferroelectric perovskites are versatile, ranging from PTC resistors, actuators and sensors to photovoltaics. However, these perovskites often contain non-replaceable, toxic lead (Pb) to achieve tailorable and unique properties.

This environmental and health risk encourages research into novel lead-free ferroelectric perovskite oxides with tailor-made material properties. In this contribution, we demonstrate how the substitution of the B-site by more than two different cations enables to tune the characteristics of Barium-based ABO_3 -perovskites. Therefore, the optical and electrical characteristics as well as the ferroelectric phase transition temperature are analyzed considering the synthesis conditions.

$\text{BaCu}_{0.33}(\text{Nb}_x\text{Ta}_{1-x})_{0.67}\text{O}_3$ ($0 < x < 1$) is synthesized by solid-state reaction of mixed oxides. Fundamental material properties are characterized by temperature-dependent Rietveld refinement of diffraction data, UV-VIS-NIR spectroscopy, as well as SEM and EDX

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Green synthesis of anatase TiO₂-based powder nanoparticles for microplastic photocatalytic removal

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Microplastics (MPs) are present in aquatic ecosystems and are consumed by their biota. Strategies to fight MPs pollution include photocatalysis using TiO₂ nanoparticles. Photocatalysis is based on the interaction of TiO₂ with photons with E higher than E_g, and the generation of hydroxyl (OH[•]) and hydroperoxyl/superoxide anion radicals (HO₂[•]/O₂^{-•}). Those species mineralise MPs into CO₂ and H₂O or degrade them into less toxic substances. Here, N-TiO₂, C,N-TiO₂ and C,N-TiO₂/SiO₂ nanoparticles obtained from 3 different renewable raw materials were prepared, characterized and tested for removing MPs. Two green syntheses were used: a bio-inspired route where proteins from *Mytilus edulis* and *Mytilus galloprovincialis* mussels were used as renewable raw materials and a biomineralization route using *Pteria sterna* oysters. The structural, chemical, optical, and textural properties of the powders were investigated by XRD, XPS, DRS, FTIR, nitrogen adsorption, and FEG-SEM. Photocatalysis of MPs was monitored by gravimetry, FTIR, carbonyl index, MO and SEM-EDS.

It was found that the mussel's derived nanoparticles have a better ability to degrade MPs than the biomineralized powders. The C,N-TiO₂ derived from *M. edulis* mussels can degrade up to 70% PE MPs and the C,N-TiO₂/SiO₂ derived from both mussels can degrade PET MPs. It was demonstrated that the photocatalytic degradation of MPs in an aqueous medium using TiO₂-based advanced ceramics is possible and can be used to fight MP marine litter.

Optical inspection – a solution for inline inspection of ceramic products and processes

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Advanced ceramic components are frequently used in industrial applications. Despite optimized production technologies, defects still occur in the manufacture of ceramics due to the complexity of the processes. Since ceramic is a brittle material, the appearance of defects leads to rapid crack growth followed by spontaneous destruction, which results in a function failure of ceramic components. Therefore, the inspection of ceramic products is particularly important especially at early stage and process of manufacturing.

Time-resolved Laser Speckle Photometry (LSP) is an optical inspection method based on the evaluation of either temporal change of speckle or static speckle patterns. Speckle pattern is an interference pattern which is determined by surface structure of objects. In this method, the characteristics of speckle pattern will be extracted to correlate the quality state of ceramic products. In this case, LSP is an appropriate technology which can be applied to real time inline inspection for ceramic manufacturing.

A developed concept for quality monitoring of ceramic materials based on the LSP will be presented in current presentation. The LSP implementation will be carried out using the example of dense and porous ceramics. A robot-guided measurement principle for the detection of various defects will be shown, which can be used for defect detection, porosity determination and surface characterization. In addition, the LSP is also valid to monitor the quality state of the roll-to-roll process in the manufacturing of battery electrode.

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Rheological investigation on thermoplastic Al₂O₃-feedstocks

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Aluminum oxides are frequently used to investigate routes or analyze parameters for ceramics processing methods. One reason is the cheap price in comparison to other ceramics like ZrO₂. In our studies, alumina thermoplastic feedstocks based on ethylene vinyl acetate and stearic acid were prepared and shaped by a commercial 3D filament printer. In addition, the rheological properties of the feedstocks compositions were investigated using plate-plate configuration. The effect of the stearic acid content on the rheological behavior was investigated in detail and compared with existing rheological models. The results show that at a low amount of stearic acid, a viscosity plateau (cross model) could be observed. At higher stearic acid content, a yield point (Herschel-Bulkley model) occurs, as the stearic acid content surpasses the amount needed to cover the powder surface. Stearic acid also influences the mechanical properties of the filament. A higher content resulted in a more brittle and less flexible behavior of the filaments. Thin wall structures were printed, debonded and sintered to demonstrate the shape stability and fusion between the layers. Complex ceramic tetrahedron structures, which are challenging to produce by more conventional methods such as injection molding have been printed and the thermal debinding process was investigated in more details. The shrinkage of the parts was found to be anisotropic, depending on the orientation of the printing pattern. The alignment of the ceramic particle orientations introduced by FDM printing was identified as a potential cause of the anisotropy. This study further demonstrates that using a powder bed during the thermal debinding process yields sintered structures that can withstand twice the compressive force.

Additive manufacturing of ceramics from liquid feedstocks

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

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Recycling the product of thermal inertization of man-made vitreous fibres for the manufacture of porcelain stoneware tiles

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This work investigated the employ of a material deriving from the thermal inertization of exhaust hazardous mineral wool as secondary raw material in the production of porcelain stoneware tiles. The aims were to promote the recovery and recycling of a material not served by the supply chain and therefore destined for landfill, as well as to promote the use of alternative raw materials by encouraging an efficient use of resources. The glassy material deriving from the inertization of the mineral wool was fully characterized and, on the basis of its properties, added to a batch for the production of porcelain stoneware tiles in substitution of the fluxing component. The characterization of semi-finished products did not highlight the presence of bottleneck in the production cycle. The final products were fully characterized in terms of technological properties (linear firing shrinkage, water absorption, bulk density, flexural strength, colorimetry). The final product properties resulted satisfactory, since the values of all the technological parameters recorded for waste-bearing bodies match the standard requirements for porcelain stoneware (ISO13600). Moreover, the introduction of inertized waste allowed to lower the firing temperature up to 40 °C with respect to the benchmark. The only drawback observed is related to the colour of the ceramic bodies: the amount of Fe intrinsically present in the inertized glass led to a darker final colour.

Mixed-mode fracture behaviour of refractories with asymmetric wedge splitting test: numerical and experimental study

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The thermo-mechanical stresses introduced by the industrial operation processes are one of the most critical failure features. Despite its importance, the pure Mode I loading is very rare and seldom represents a realistic loading condition. Most damage cases are caused by combined stress states. Shear stress is very frequently co-existing. Different from the most applied mixed-mode test methods such as centrally cracked Brazilian test, the three-point bending test with off-center notch and so on, an asymmetric wedge splitting test (WST) for mode I/II mixed-loading was developed in this work. Firstly, the suitability of asymmetric WST was validated by FE simulation with a three-dimensional heterogeneous continuum FE model based on the stress/strain distribution and the fracture process related-energy dissipation. Secondly, the influences of various levels of mode II loading on damage behaviour of refractories with different brittleness were experimentally investigated by comparing mode I and mixed-mode fractures under symmetric and asymmetric WST loading with seven different wedge angles. The digital image correlation technique was also used for strain maps visualization as well as the deformation parameters acquisition. The results show that the fracture behaviour becomes unstable with the increase of asymmetric wedge angle, more instantaneous energy release and restrained fracture process zone development. The in-plane shear loading contributes to the accelerated extension of the crack tip and its deviation from central plane. Meanwhile, the co-existing local shear stresses caused by the refractory's heterogeneity lead to crack path deflection as well.

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Manipulation of calcium phosphate slurries and pastes in the field of bone regeneration

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A wide range of calcium phosphates have been studied in the last decades as promising candidate to promote the regeneration of bone tissue. However, the design and preparation of large bioceramic scaffolds provided with tailored porosity still represents a remarkable challenge. In this respect, their porosity, pore size distribution and interconnection strongly reflect their bioactivity and osteoconductivity *in vivo*.

Several methods have been reported to produce porous scaffolds, including replica, sacrificial template, direct foaming and 3D Printing technology.

The issue comes around as all these techniques are based on the manipulation of slurries, highly concentrated water-based suspensions with critical processing. In this context, a precise rheological characterization of bioceramic slurries is demanded. It was reported that the thermal calcination of the powder and the amount of dispersant agents significantly affect both the viscoelasticity of the slurry and the final properties of the scaffold. In this work, hydroxyapatite (HA)-based slurries were prepared, while evaluating the effect of calcination treatment, powder amount and dispersing agent on the rheological properties. The stability of the slurries was monitored by pH and ζ -potential measurements, while both viscosity and viscoelasticity tests were performed.

The stability of the suspensions was mainly affected by the calcination temperature, while the effect of the dispersant becomes significant with increasing the powder concentration. Furthermore, the picture was complicated by also analysing the dynamic rheology of self-hardening calcium phosphate cements, their setting kinetics and phase transformation towards the control of their extrusion.

Mechanically stable and crack-free SiOC screens for gas-liquid phase separation

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A macroporous SiOC pore structure for phase-separation requires balanced mechanical and mass transport properties that can be created via unidirectional solution-based freeze-casting of a preceramic polymer. In this work, cyclohexane and tert-butyl alcohol or a mixture of both were used as a template media in freeze-casting to achieve monoliths with a permeable pore morphology with high mechanical stability. The results show that the dendritic pore structure has the highest compressive strength (39 MPa) due to its smallest pore sizes (16-20 μm) and secondary dendrites, and was therefore selected to fabricate crack-free porous screens for air-hydrofluoroether phase-separation. The pore window size of the screens varied between 6 μm and 35 μm depending on the freezing temperature (-20 °C, -80 °C or -120 °C). These screens have already been used in initial experiments and show promising properties for applications in the field of gas-liquid phase separation.

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Combination of extremely low thermal and high electrical conductivity of sustainable carbon-ceramic electrospun nonwovens

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Electrospinning is a processing method for polymer fibers with diameters in the nanometer scale from solutions. Polymer fibers can later be converted to ceramic fibers by pyrolysis using preceramic polymers. For example, the resulting fiber mats can be used as catalysts or as filtration media. In this study, various nonwovens composed of polymer blends of polyacrylonitrile (PAN) and different amounts of a commercially available organosilazane were processed. Subsequent stabilization and pyrolysis led to ceramic C/SiCON fiber mats. The characterization of the microstructure and elemental distribution of the fibers with high-angle annular dark-field imaging (HAADF) and scanning transmission electron microscope x-ray energy dispersive spectroscopy (STEM-EDS) confirmed the transformation to a “sea-island” nanostructure. Various analytical methods such as FTIR, WAXS and solid state NMR, revealed the structure of the resulting material. It is composed of a mainly amorphous carbon phase, where amorphous SiC_xO_y , SiN_xO_y , and SiC_xN_y ceramic nanophases are embedded. Thanks to this structure, the material achieved unique properties having simultaneously a high electrical conductivity of 4.2 S/cm and a low thermal conductivity of 19.8 mW/m.K. Whereas the carbon phase caused the high electrical conductivity, the ceramic phases are responsible for the low thermal conductivity by boundary scattering. Furthermore, the obtained nonwovens had an excellent foldability, mechanical flexibility and oxidation stability, making them attractive for applications such as smart textiles and heating systems.

Na-beta"-alumina – novel concepts for an established ceramic electrolyte

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Ceramic electrolytes made from Na-beta"-alumina are commercially utilized in Na/NiCl₂ and Na/S-batteries since the 1990s. [1,2] They offer a variety of benefits like a high ionic conductivity (about 0.2 S cm⁻¹ at 300 °C), a neglectable electronic conductivity, stability towards Na and a low toxicity. [1,3] The Fraunhofer Institute for Ceramic Technologies and Systems (IKTS) worked for the last years on the next steps to pave the way towards novel fields of application and production methods for Na-beta"-alumina electrolytes.

Highlights that will be presented within the contribution are: The mechanical and electrochemical material properties of Na-beta"-alumina were modified by transition metal doping. The observed effects were explained by changes of the microstructure.

[3] The shaping of green bodies is one of the critical processes of ceramic production. This process was mastered at Fraunhofer IKTS by several techniques: Besides the common production route of isostatic pressing, methods like extruding, slip casting, or tape casting were successfully investigated. Especially extrusion and tape casting were efficient ways to produce Na-beta"-alumina electrolytes.

[4] To utilize Na-beta"-alumina in room temperature cells, hybrid electrolytes of polymers and Na-beta"-alumina particles were prepared and tested. They unite the advantages of Na-beta"-alumina (ionic conductivity) with the advantages of polymers (flexibility).

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Ceramic membrane-based technologies for oilfield produced water treatment

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“Produced water” (PW) is any fossil water that is brought to the surface along with crude oil or natural gas. By far, PW is the largest waste stream by volume associated with oil and gas production operations. Due to the increasing volume of waste all over the world in the current decade, the outcome and effect of discharging PW on the environment has lately become a significant issue of environmental concerns. Therefore, there is a need for new technologies for PW treatment due to increase focus on water conservation and environmental regulation.

The use of membrane processes for treatment of PW has several advantages over many of the traditional separation techniques. In oilfield produced water treatment with ceramic membranes, process efficiency is characterized by the specific permeate flux and by the oil separation performance. Apart from the membrane properties, the permeate flux during filtration of oily wastewaters is known to be strongly dependent on the constituents of the feed solution, as well as on process conditions, e.g. trans-membrane pressure (TMP) and cross-flow velocity (CFV). The research project presented in these report describes the application of different ceramic membrane filtration technologies for the efficient treatment of oil-field produced water and different model oily solutions.

Silicone-assisted Advanced Additive Manufacturing of Glass-Ceramic Scaffolds

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Silicone resins are attractive both as precursors of silicate bioceramics and as feedstock for additive manufacturing technologies, including masked stereolithography. The two aspects may be successfully combined when engineered blends, as binders for ceramic powders, consisting of a silicone polymer are mixed with photocurable acrylates. The first case study concerns scaffolds with a composition resembling well-established Biosilicate® glass-ceramics, from the direct thermal transformation of silicone into silica, reacting with sodium and calcium salts (carbonates and phosphates). The technology enables the obtainment of novel composites, with the silicone yielding also pyrolytic carbon, by firing in nitrogen. The latter phase provides extra functionalities, such as intensive heating by absorption of IR light, useful for disinfection purposes. The second case study regards wollastonite-diopside glass-ceramics, in which the final phase assemblage relies on the chemical interaction, upon firing, between binder-derived silica and softened glass. Compared to glass-ceramic scaffolds from stereolithography with fully sacrificial acrylate binders, with the same overall oxide formulation, the new methodology enables a distinctive topological control.

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Optimization challenges of KNN-based piezo ceramics: temperature stability of strain, sintering temperature and scalable processability

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(K, Na)NbO₃ (KNN) based ceramics evolved to the most promising candidates to replace lead containing Pb(Zr,Ti)O₃ based ceramics in various application. Over the past decades, phase composition design and domain engineering by chemical modifications and sophisticated synthesizing led to excellent piezoelectricity. One of the most challenging applications are actuators e.g., for precise positioning. In this technical environment the strain must be unaffected by the temperature over a broad range to achieve the needed accuracy and reliability. In recent years, attempts were undertaken that focus on a diffuse phase transition with increasing temperatures and at the same time a multiphase co-existence (Huang et al. 2018; Lv et al. 2020; Yao et al. 2016; Cen et al. 2018). This leads to a multiphase co-existence over a wide temperature range. Thus, an abrupt degradation of the strain behavior is prevented. All referred investigations were performed in laboratory environment and the important assessment of their relevance for a possible industrial processing is lacking.

In this contribution we investigate the sintering behavior, strain performance and limitations due to scaling attempts for different phase engineered compositions. A moderately increased sample size results e.g., in tremendous porosity and degradation of piezoelectric performance. In many cases, the ideal sintering temperatures for best performance are much higher than expected and too high for multilayer processing. Thus, the impact of different sintering additives on the performance of the complex materials is also investigated.

Optical 3D profilometry for characterising and digitising ceramic surfaces

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The texture of a ceramic surface, e.g. a floor tile, plays a decisive role with regard to its properties in interaction with environmental factors. Slip resistance, cleanability and durability, as well as antibacterial and easy to clean properties - all this is strongly defined by the surface (e.g. shape, topography, roughness). A tile with a surface profile characterised by deep, narrow valleys or grooves will be more difficult to clean than a completely smooth surface, but might, dependent on the different aspects of its profile, exhibit a different slip behaviour than expected. This makes it even more important to be able to precisely characterise a surface using state of the art 3D optical profilometry. Thus, explanation for and predictions about performance or a precise analysis of defective behaviour and defects are possible. Furthermore 3D profilometry is a suitable tool for optimising ceramic surfaces with regard to their applications by developing new products through targeted surface design, e.g. realised by appropriately designed pressing tools. With a mobile optical profilometer, even large, non-transportable samples as well as floor coverings can be non-destructively characterised on site, shifting the decision point on site instead of after laboratory measurements or duplicates or extracted samples.

The project "DigiKerO - Digitisation of Ceramic Surfaces", which is aimed at the enhancement of the FGK competence on surface characterisation is funded by the state of Rhineland-Palatinate in the Ministry of Economics, Transport, Agriculture and Viticulture. The lecture shows the diverse application possibilities of optical 3D profilometry for (ceramic) surfaces and presents the first project results.

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Porous carbon-rich transition metal modified SiCN ceramic fibers for electrochemical water splitting

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The global energy demand and environmental pollution drives the need for the augmentation of the existing sustainable energy devices. Electrocatalytic devices such as fuel cells have raised considerable interest due to their high energy and power density. On the other hand, electrocatalytic water splitting offers an attractive route to produce high-purity hydrogen and platinum based electrocatalysts have proven to be the best in terms of its oxidation/reduction reactions. Currently, platinum on a carbon support is extensively used for water splitting reactions. However, the use of noble metals increases the overall cost of electrocatalytic devices and platinum based electrocatalysts are also known to be unstable in fuel cell working conditions. Hence, the development of efficient and durable catalysts is critical for the commercialization of fuel cells, as the catalysts' reactivity and durability dictate their ultimate activity and lifetime.

In this work, carbon-rich cobalt modified SiCN ceramic fibers were produced from a precursor derived ceramics (PDC) approach. The role of the pyrolysis temperature on the structure of the ceramics and its electrocatalytic activity were investigated. The phase evolution, structural and textural properties of the developed catalytic supports were evaluated with the aid of X-ray diffraction, scanning electron microscopy and nitrogen adsorption analysis. The ceramic fibres synthesized with a combinatorial approach using PDC and electrospinning had mesoporous structure. The electrocatalytic performance was determined using rotating disk electrode method under alkaline conditions.

Electromechanical properties of the lead-free piezoelectric $\text{Bi}_{1/2}\text{K}_{1/2}\text{TiO}_3$ as a function of sintering parameters

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Processing parameters play a crucial role in the achievement of optimal properties in functional ceramics. $\text{Bi}_{1/2}\text{K}_{1/2}\text{TiO}_3$ (BKT), a critical end member for lead-free piezoceramic solid solutions, is a promising candidate for high temperature applications owing to their elevated ferroelectric-to-paraelectric transition temperature, $T_c \sim 380$ °C as well as a high depolarization temperature, $T_d \sim 300$ °C. However, the processing of BKT is challenging due to sintering difficulties, *i.e.*, low melting point of BKT (1070 °C) and volatilization of A-sites cations (Bi^{3+} and K^+), which limits phase purity and reduces the functional properties. In this study, the electromechanical behavior as a function of sintering temperature and dwell time in solid-state processed BKT was investigated. The findings were directly correlated to a change in crystal structure and microstructure, where higher internal stresses in smaller grains resulted in the inhibition of relaxor-to-ferroelectric phase transition (RE-FE) as well as a diminution of dielectric, piezoelectric, and ferroelectric properties. This work comprehensively demonstrates the microstructure/structure–piezoelectric property relationships in solid-state processed BKT related to an enhancement of dielectric and electromechanical response for dense ceramics. Moreover, importantly Rayleigh analysis revealed a significantly higher extent of intrinsic contributions (94%) to the piezoelectric coefficient in BKT.

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Ca-substituted perovskites as promising materials for H₂ production in Oxygen Transport Membrane Reactors

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The affordable production of pure hydrogen on a large scale is crucial to fulfill a transition to a decarbonized energy system. Water, as an abundant and carbon-free material, is the ideal H₂ source, however the splitting reaction of the water molecule into O₂ and H₂ has a very low equilibrium constant and therefore poor efficiency. This limitation can be overcome by performing the process in a membrane reactor, where the produced oxygen is continuously removed by an Oxygen Transport Membrane (OTM) and the reaction equilibrium is shifted towards the products. The diffusion of such technology is still hampered by the poor stability of the OTM materials in the reactor operating conditions, namely high temperatures (> 800 °C) and harsh environments (presence of steam and reducing atmospheres).

In this context, we propose innovative Calcium-substituted perovskite oxides that can serve as materials for OTMs. Starting from the material system SrTi_{1-x}Fe_xO_{3-d} (STF), which already proved its applicability for oxygen separation membranes, we synthesized powders where the perovskite A-site is partially substituted with different amounts of Ca (Ca_ySr_{1-y}Ti_{1-x}Fe_xO_{3-d}, CSTF), aiming to improve the CO₂ tolerance. The CSTF powders were used to realize dense OTMs, whose properties were preliminarily assessed. The results obtained for the different compounds will be presented and discussed in relation to possible employment in a membrane reactor for water splitting.

Joining and integration issues of ceramics and CMC

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Innovation in processing and characterization of ceramic- and CMC-based joined components developed at GLANCE-Glasses, Ceramics and Composites research group at Politecnico di Torino, Italy (www.composites.polito.it) will be presented and discussed.

Joined components for energy storage and transformation have been designed, fabricated and characterized in simulated working conditions: recent results will be briefly reviewed.

The combination of advanced design of interfaces and joining materials/technologies, selective matrix removal from the composite surface, laser structuring and mechanical machining of the composite/metal surfaces will be discussed and compared to existing solutions.

The work done with the aim of developing reliable and user-friendly international standard test to measure the shear strength of joined components will also be reviewed.

Finally, J-TECH@PoliTO (<https://www.j-tech.polito.it/>) , Advanced Joining Technology research center at Politecnico di Torino, will be described together with collaboration actions and opportunities for common research activity on joining.

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Characterization and simulation of porous ceramics

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Cellular materials offer a wide spectrum of applications such as catalyst support structures, lightweight materials, energy adsorption or energy storage materials. Due to several ways of processing and different materials, a wide range of material properties e.g. thermal conductivity, mechanical strength or damping can be adjusted, measured and verified, with regard to the expected properties. Especially in heterogeneous and homogeneous porous structures and their composites, only global effective material properties can be determined and measured. For example, the knowledge on the predominating influence of the microstructure on the global properties is the key for designing materials with desired properties. To fill this gap and enable a "look-in" a microstructure model derived from μ -CT measurements carried out at certain processing steps can be used as model for FEM-calculations. In this context, the representative volume of interest (REVOI) in particular plays a decisive role in order to be able to determine the global and not only local characteristics. By combining Minkowski and structural parameters, the REVOI can be determined as a function of the cellular structure.

Combining estimated material properties by experiment with microstructure models offers the possibility to carry out different simulations over different hierarchical levels in order to design the structures for future applications of porous ceramics.

Intrinsic and Extrinsic Control in Gas Phase Deposition Processes for Functional Ceramics

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Gas phase deposition processes like chemical vapor deposition (CVD), atomic layer deposition (ALD) or atomic layer epitaxy (ALE) rely on molecular precursors with defined composition and reaction profile, enabling thin-film deposition with desired properties regarding coverage, crystallinity, composition, phase, as well as functionality (i.e. conductivity, barrier or catalytic properties, etc.). Precise process control is mandatory to achieve reliable and reproducible materials synthesis as well as to realize eco-friendly and sustainable manufacturing techniques. Therefore, both intrinsic as well as extrinsic measures should be exploited complementarily in order to achieve best process control. In addition to temperature, pressure and electromagnetic radiation, electric and magnetic fields can be employed as external stimuli in materials processing to influence structure, morphology and phase formation. While the influence of electric fields has been investigated more thoroughly, reports of magnetic fields, especially in CVD remain scarce and limited to few examples, where for example magnetic metal catalysts guide the directional growth of CNTs. Here we report the influence of external magnetic fields on the decomposition and phase and morphology evolution of transition metal oxides and nitrides based on molecular single source precursors. The precursor composition has direct influence on the thermochemical as well as magnetic properties and thus directly affect the growth kinetics under varying deposition temperatures, field strengths and deposition times, magnetic field CVD (mfCVD) offers a versatile additional degree of freedom in optimizing gas phase deposition processes.

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Size effect of strength for ceramic matrix composites

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Ceramic matrix composites (CMCs) exhibit a peculiar combination of damage tolerance combined with typical properties of ceramics like thermal stability, wear resistance, and low density. CMCs overcome especially the brittleness of monolithic ceramics. Even so, CMCs also show a dependency of strength as a function of the tested size or volume of the sample. Up to now, the mechanisms, models, and even existence of a size effect of strength for CMCs are still unclear. This study analyzed the influence of the sample size and testing method on the strength of carbon fiber-reinforced silicon carbide (C/C-SiC). Hence, C/C-SiC samples with varying sample geometries were tested under bending and tensile loads. A testing device was developed to ensure minimally biased tensile testing due to a self-alignment and centering of samples. It was found that a size effect of strength exists for C/C-SiC. It was further shown that the Weibull modulus was not a material constant for C/C-SiC and the classical probabilistic aspects of brittle fracture with the application of Weibull statistics is unsuitable to describe the size effect of strength. The size effect of C/C-SiC is discussed regarding the concepts of load sharing, the weakest link approach, and the energetic size effect of quasi-brittle materials.

Development of an efficient production technology for the synthesis of highly pure, stoichiometrically and morphologically defined oxide nanopowders

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The production of transparent and optoceramics but also active materials for battery storage or oxide ceramics for medical technology products increasingly requires powder materials that are characterized by nanoscale grain sizes, high purity and a defined phase composition.

Fraunhofer IKTS and Glatt Ingenieurtechnik are pursuing the synthesis of oxide nanopowders based on pulsed spray pyrolysis in the project "PulsON". Among other things, the new synthesis plant can realize a process gas temperature of up to 1300° C, a frequency of 0-400 Hz and a pressure amplitude of up to 60 mbar. A major goal in this project is to determine the interrelationship between the precursors, the process parameters, and the powder properties. The material system selected for this purpose is Mg-Al spinel. For the first time, the process engineering advantages of high-temperature synthesis can be exploited, since the target stoichiometries and phases of the powders can be precisely controlled on the basis of the very short heating times, the highly effective, homogeneous heat input and controllable residence times in the synthesis plant. In addition, models are developed on the basis of computer-based simulations, which are validated by extensive experimental investigations, from which, in perspective, highly scaled energy efficient plants with significantly higher throughput quantities are possible.

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Dislocation-based Plastic Deformation and Fracture Behavior of Single-Crystal CaF₂

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Ceramics are often considered inherently brittle, especially at room temperature. There is, however, a wide variety of ceramic materials that are actually plastically deformable by dislocation activities at room temperature. Compared to the advanced studies and characterization of dislocations in metallic materials, the understanding of dislocations in ceramic is less developed. The recent upsurge in research interest for dislocation-tuned functional properties of ceramics has also triggered more attention to the dislocation-based mechanical properties. With their role in plasticity, work hardening, and toughening, dislocations have the potential to greatly improve the damage tolerance of ceramic parts. By introduction of dislocations, it was already possible to significantly toughen SrTiO₃ single crystals. Recently, the method of cyclic Brinell indentation was established to introduce a high dislocation density up to 10¹³/m² in areas larger than 100 μm in diameter in single-crystal SrTiO₃ without crack formation. To further extend the dislocation studies in other ceramics, here we applied this cyclic indentation approach on CaF₂, which allows for an in-depth analysis of the dislocation structure evolution during cyclic indentation, such as the dislocation multiplication mechanisms and the interactions between dislocations. We tested the micro-hardness and compared the differences in the crack patterns around the Vickers indents between the pristine and the dislocation-enriched regions. A detailed analysis of the interactions between dislocations, their influence on crack nucleation, and the overall dislocation toughening effect in CaF₂ is also conducted.

Cooperation as competitive advantage on the example of international norming of additive manufacturing of ceramics

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New processes face a lot of entry barriers before they can succeed. Scientists and engineers tend to overestimate the technological challenges and are surprised that new production processes do not catch on the way they expected. The simple fact is that customers have to be convinced of the advantages of the product and rules for the use must be established.

This is probably the hardest lesson for ceramic additive manufacturing. Another lesson learned is that teaming up is a clever idea.

Faced with this, the experts in the field in Germany teamed up on different topics. On the research side the "Szene Additive" was founded within the DKG with prominent members of the scientific community. This has now evolved into the User Circle for Additive Ceramic Manufacturing "Anwenderkreis Additive Keramische Fertigung" (AKF) within the DKG that is representing the interests of industrial partners and universities.

The VDI (Association of German Engineers) took on the approach to solve the market acceptance challenge. Therefore machine producers and machine users were making guidelines for designing and testing.

A technology is not really established if it does not have the basic rules for its use and applications. Here it is noteworthy that in a rare case of collaboration, the ASTM International and the ISO are teaming up to harmonize all norms concerning additive manufacturing.

Here is where it all comes together: the norming group of the AKF is internationalizing the VDI guidelines for additive manufacturing for ceramics parts within the frame of the ASTM/ISO working groups, a truly international cooperation.

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Inorganic membranes for dewatering of liquid solvents and process mixtures

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In chemical processes the desired products are often obtained as mixtures that contain impurities resulting from the reaction. Water is one of the most frequently present components since it occurs as starting material, intermediate or by-product in chemical reactions as well as due to intrinsic properties of certain substances such as hygroscopicity or azeotrope formation. Membranes can be tailored to selectively separate water from these mixtures. However, only a few membrane techniques have been established so far. Those were applied mainly in gas phase application like biogas production whereas in liquid phase only very few examples (solvent recovery, azeotrope separation) are considered for downstream purification, yet. In a recent project founded by the German Ministry for Economic Affairs and Climate Action (BMWK) various inorganic membranes were screened in dewatering by pervaporation of solvents (acetone, ethyl acetate), alcohols (methanol, ethanol), synthetic fuels (DME, OME, butanol), ammonia and amines. Thus, a broad range of operating conditions (T , p , pH value) were examined. Depending on their surface properties (porosity, hydrophily) the suitability of the respective membranes was compared. The most promising ones were used in an upscaled pilot plant to investigate their feasibility as well as longevity. Furthermore, a comparison with established distillation processes regarding energy consumption and CO₂ emissions will be drawn and potential applications concluded.

The Advantages of Ceramic Ultrafiltration Membranes for Pre Treatment of SWRO & waste water streams

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Reverse Osmosis (RO) is the current preferred technology for seawater desalination. When provided water free of suspended solids and low in dissolved organic carbon (DOC) RO membranes can last for 1-2 years between cleans and a lifetime up to 10 years. Conventional RO pretreatment has struggled when faced with challenging influent water quality resulting in: Capacity loss, High cost of RO cleaning, Frequent RO element replacement. UF membrane pretreatment is expected to address these issues. Another topic is the treatment of municipal waste water as the 4th cleaning step to reduce the intake of critical chemicals like PFAS/ PFOS, micro- & nano plastic as well as pharmaceuticals to natural waterways upstream. In this presentation the results of several pilot trials under realistic raw water conditions for both topics are presented.

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MAX phases: from synthesis to applications

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MAX phases have attracted a considerable attention in the last years due to their unique combination of properties, bridging the gap between ceramics and metals. However, despite the potential of some compositions such as Ti_2AlC and Cr_2AlC to operate under aggressive environments, their transfer to applications is limited by three main factors: i) complexity of this large family of materials, ii) unavailability of highly pure commercial powders, and iii) extensive time to license products in strategic fields. In this work, reasons and potential solutions of these three points are discussed and correlated to the main properties of MAX phases and their synthesis routes. Emphasis is given to processing routes for developing different structures such as dense bulk samples, ceramic matrix composites, foams with controlled porosity, coatings, and near-net shaping. Well-known and novel potential applications are described as well as future challenges to facilitate the transfer to the market.

Integration of functional materials in ceramic multilayer technology: ZnO-based varistor screen printing paste development

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Zinc oxide-based varistors have been used since the 1970s as voltage-dependent resistors to protect electronic circuits from voltage spikes. They behave like insulators that switch reversibly to a conductive state at high voltages, a result of the electrostatic potential barriers formed at the grain boundaries of the ceramic by doping, similar to PTC ceramics.

Ceramic-based substrates and packages for electronic components in LTCC (low temperature cofired ceramics) technology find broad commercial application due to the embedding of passive components such as coils, capacitors, and resistors. To expand their application area and thus their added value, it is desirable to integrate additional functional elements via established manufacturing methods. Specifically, this contribution discusses the integration of ZnO-based varistors into LTCC multilayers.

Thus, the composition of the varistor ceramics were adapted for LTCC technology. To develop screen-printing pastes, the powder was mixed with organic solvents, binders, and additives to influence the rheology, thixotropy, and drying behavior. The effect of postfiring on alumina or cofiring on LTCC substrates on the varistor properties will be compared to bulk ceramics and discussed considering the microstructures, interfaces, and reactivity of the multimaterial systems. Finally, the major challenges of varistor integration into LTCC and some ideas for solving those will be presented.

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DMD-DLP based Additive Manufacturing of Ceramics – inspired by Colloid Chemistry

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The efficient stabilisation of ceramics based nanopowders is a prerequisite for the achievement of highly reliable ceramic materials and for the development of three-dimensional printing technologies, recently known as Additive Manufacturing.

Agglomeration due to Van der Waals forces can be avoided reducing the effective Hamaker values on the basis of refractive index matching [1,2] and more readily via electrostatic and/or steric stabilization using different formulation strategies to increase the (repulsive) barrier for agglomeration.

We recently performed studies to stabilize alumina, magnesium-spinel and zirconia submicron/nanoparticles. Anion and cation type comb copolymers were applied as a promising dispersants in case of titania stabilisation [3,4]. Highly charged counterions were furthermore used to modify the electric-steric double layer [5] of mixed oxides. These slurries were successfully applied to the stereolithographic shaping of ceramics using a digital light processing (DMD-DLP) [7-11].

Influence of the test parameter on the thermal shock behavior of ceramic foams

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Ceramic foam filters contribute to improving the cast quality in the foundry by removing inclusions from the melt during casting. As casting is done at high temperatures, the thermomechanical properties are very important in terms of ceramic foam filter material selection. It is therefore necessary to have a procedure for the characterization of thermomechanical properties enabling comparison between the different filter materials. There are already few standards for testing such as the German standard DIN 51068 and the European Standard DIN EN 993-11. The aim of this study is to evaluate the effect of different thermal shock procedures (including but not limited to the methods described in DIN 51068 and DIN EN 993-11) on the mechanical properties of alumina foam filters. To estimate the contribution of macroporosity, two kinds of samples were utilized- macroporous foams and bulk samples. Alumina ceramic foams were prepared by means of the Schwartzwalder process while bulk samples were made by pressing alumina powder. Prior to thermal shock, the Young's modulus, crushing strength and, bending strength of the ceramic foam and bulk samples were determined. The samples were subjected to thermal shock at 950 °C or 1200 °C and subsequently quenched either with water or compressed air. A different sample subset was immersed in molten aluminum for 1s or 10s. After thermal shock, the mechanical properties were again determined at room temperature. The residual mechanical properties were determined and evaluated. Results show significant differences in the samples' residual mechanical properties after the different thermal shock and quenching procedures and, different immersion times in molten aluminum.

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Two-Photon-Polymerization for Ceramics Powder Processing

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Manipulating ceramic powder compacts and ceramic suspensions (slurries) within their volume with light requires a minimum transparency of the materials. Compared to polymers and metals, ceramic materials are unique as they offer a wide electronic band gap and thus a wide optical window of transparency. The optical window typically ranges from below 0.3 μm up to 5 μm wavelength. Hence, to penetrate with laser light into the volume of a ceramic powder compound its light scattering properties need to be investigated and tailored. In the present study we introduce the physical background and material development strategies to apply two-photon-polymerization (2PP) for the additive manufacture of filigree structures within the volume of ceramic slurries.

Material and process hybridisation through in-mould labeling of metallic nonwovens in ceramic substrates

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The research aims not only at the hybridisation of metal and ceramic structures via an innovative textile interface, but also at the hybridisation of conventional and additive technologies. In this way, multifunctional components with a geometric complexity are producible. The focus of attention is on powder injection moulding in combination with in-mould labeling as a production process suitable for large series. Composite components are bonded to each other by means of a metallic textile nonwoven. Due to the inherent properties of the nonwoven, like non-directional fibre structure, isotropic compressibility, thin fibre diameters, it can partially be embedded in the ceramic substrate and sintered with the ceramic without damaging the composite due to the different coefficients of thermal expansion (CTE) of the fibres and the shrinkage of the ceramic. The sintering pressure of the ceramic component compresses the nonwoven and permanently binds it in a surface area. The metallic nonwoven is prefabricated and inserted into the open mould of an injection moulding machine. After closing, thermoplastic material - the ceramic feedstock - is inserted into the mould, injects and infiltrates the non-woven to a certain depth. After cooling, the component is ejected, debonded and sintered. In this way, a metallic textile anchor structure between the ceramic and the metal is produced. Further metal components can be added to the free surface of the nonwoven. Combinations between a ceramic (e.g. zirconium oxide) and a lower-melting metal with a significantly higher CTE (e.g. copper or aluminium), without being affected by thermal stresses or the different sintering temperatures of the composite can be manufactured.

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Ceramic membrane contactors with additively manufactured flow distributors for separation applications

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Inopor® is currently the only supplier who is able to deliver ceramic nanofiltration membranes with low cut-offs up to 200 Dalton. These are successfully used in a broad range of applications for example in the food and beverage filtration (wine, beer, fruit juice), water and wastewater treatment or for other different issues in the chemical and biotechnology industry.

The team of Rauschert/Inopor® and Fraunhofer IKTS is participating in different development projects. The results and potential applications of the project "KeramAD" – Program EFRE (Thüringer Aufbaubank) funded by the European Union will be shown.

As part of the project, a ceramic honeycomb element with a new flow principle using additively manufactured flow adapters is to be developed and tested in membrane extraction and membrane distillation.

Different parts of the innovation will be presented:

Design of the flow adapters and current limitations in the structure

Extrusion of ceramic honeycomb structures for aimed applications

Tests in different applications:

Microfiltration – water fluxes and dextrane retention

Membrane extraction and membrane distillation

Solid state polymer electrolyte for highly stable solid state batteries

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All-solid-state batteries (ASSB) have gained significant attention as promising candidates to replace liquid electrolyte in lithium-ion batteries for high safety, energy storage performance, and stability under elevated temperature conditions. ASSB has huge potential to be the core of future energy storage devices, especially in applications where energy density is key to the technology. However, the low ionic conductivity and unsuitability of lithium metal in solid polymer electrolytes is a critical problem. To resolve this, we used a cubic garnet oxide electrolyte ($\text{Li}_7\text{La}_3\text{Zr}_2\text{O}_{12}$ – LLZO) and ionic liquid in combination with a polymer electrolyte to produce a composite electrolyte membrane. By applying a solid polymer electrolyte on symmetric stainless steel, the composite electrolyte membrane shows high ionic conductivity at elevated temperatures. The effect of LLZO in suppressing lithium dendrite growth within the composite electrolyte is confirmed through symmetric lithium stripping/plating tests under various current densities showing small polarisation voltages. Guidelines and aspects to process alternative materials and impact the interface design towards fast lithium charge transfer between the metalloid and the Li-garnet electrolyte are formulated. The full cell with lithium nickel manganese oxide as the cathode active material has been investigated in detail. This research highlights the capability of solid polymer electrolytes to suppress the evolution of lithium dendrites and enhance the performance of all-solid-state batteries.

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How to improve ATZ ceramics for the use of highly loaded implants?

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The work presented focuses on ideas for improving ATZ ceramics for use as a material for implants in high-load applications, such as knee implants and hip implants for early intervention.

Up to date, Mathys' ATZ ceramic ceramys[®] has very good mechanical and tribological properties with a biaxial bending strength up to 1000 MPa, a fracture toughness of 7,9 MPam^(1/2) and wear rates of 0,06 mm³/million cycle in the hip simulator under edge loading conditions. Ceramys[®] is currently only used for femoral heads and inserts for hip and shoulder arthroplasty. The general challenge with ceramics is their brittle fracture behavior, so that ceramics have so far played a subordinate role in highly stressed applications where complicated or thin-walled geometries are required. Regarding implants, this is the case in the area of the knee joint, as well as in the area of early bone-saving implant technology. Current ceramic implant materials are not yet sufficient in terms of strength and fracture toughness to solve this problem. Systems available on the market are very thick-walled and do not allow a bone-saving solution.

First promising results of a preliminary project for hip implants were continued with a series of experiments by varying the composition and preparation parameters. The results are to be presented. Furthermore, possibilities to modify the ceramic with respect to dopants in order to optimize the mechanical properties and to achieve a very high hydrothermal aging resistance will be discussed. The results were obtained within the framework of the RUBIN alliance "SAPHIR high-performance ceramics" from the Tridelta Campus Hermsdorf (funded by BMBF, FKZ: 03RU1U161D).

Hexagonal ferrite thick films for self-biasing circulators integrated in LTCC microwave modules

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Future satellite communication technologies used for low-, mid- and geostationary orbit for high-throughput satellite inter access in the K_a -band (26-40 GHz) require LTCC multilayer modules with integrated magnetic microwave components. Sc-, and (ZnSn)-substituted Ba ferrites $BaMe_xFe_{12-x}O_{19}$ exhibit significant potential for self-biasing microwave components, e.g. for circulators. Static magnetic measurements show that both the saturation magnetization and coercivity decrease with concentration of Me substituents indicating a reduction of the anisotropy field with substituent concentration. The ferromagnetic resonance frequency of the ferrites, determined by reflectance measurements in U- and K-band (30-60 GHz) waveguide, shows systematic shifts towards lower frequency with x. For Sc-substitution, a resonance in the K_a -band is observed for $x = 0.5$. Sintered ferrite samples were integrated into LTCC multilayer modules as drop-in bulk components into LTCC cavities. Circulators designs were fabricated and tested. The sintering behavior of the ferrites was adapted using sintering additives to reduce their sintering temperature and allow for cofiring with LTCC materials at 900°C. Anisotropic ferrite films were obtained through screen-printing, drying in a magnetic field and firing at 900°C. Cofiring of ferrite pastes with LTCC tapes at 900°C results in LTCC architectures with integrated ferrite layers. The potential of oriented hexagonal ferrite layers as integrated self-biased ferrite layers in microwave components is evaluated.

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High strength 3D-printed alumina ceramics through rapid sintering

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In the processing of ceramic materials rapid sintering technologies, such as FAST or SPS, have been recognized as game-changer that can densify ceramics within minutes. Rapid sintering may lead to fine-grained microstructures, aiming to enhance mechanical, electrical or optical properties. The mentioned rapid sintering technologies are limited to simple geometries due to the commonly associated applied pressure. Nowadays, since numerous technologies for additive manufacturing (AM) ceramics are being developed, the question raises whether it is feasible to rapid sinter ceramic components of high geometric accuracy and complexity. In this work, a pressure-less rapid sintering protocol was used to explore the possibility of rapid sintering 3D-printed ceramic parts with complex shapes and controlled microstructure. Stereolithographic 3D-printed alumina ceramics were sintered in a hollow cylindrical graphite die through radiation heat transfer within less than 20 minutes. It was found how maximum sintering temperature and dwelling time affects microstructural, physical and mechanical properties. Dense, pure alumina with a grain size below $\sim 0.5 \mu\text{m}$ could be manufactured, resulting in a biaxial strength ($\sigma_0=860\text{MPa}$), i.e. 30% higher than that of conventionally sintered alumina parts. This increase may be associated with the fine-grained microstructure and low internal micro-residual stresses, opening the path to sinter complex 3D-printed alumina parts in very short times.

Modular lead-free Piezoceramic/Polymer Composites with locally adjustable piezoelectric properties

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Piezoceramic polymer composites combine the piezoelectric sensitivity of brittle ceramics with the low density of flexible polymers and gain novel properties, but have typically the same piezoelectric properties in the entire material. We designed a modular composite of ceramic building blocks and polymer-matrix to control locally the piezoelectric properties with individual polarization of each block, while maintaining the benefits of each phase. The lead-free barium titanate (BT) building blocks were prepared by injection molding and were assembled into 2-2-layered and modular structures, which were bonded with BT-filled epoxy resin. The piezoelectric response was determined using direct excitation via Berlincourt method, and the influence of the matrix thickness as well as the different polarization directions of individual building blocks was investigated.

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Rietveld characterization of graded 3-5 mol% Y2O3-stabilized zirconia for dental application

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Y₂O₃-stabilized zirconia (YSZ) is applied in prosthetic and implant dentistry. In previous work [1], two tetragonal ZrO₂(Y) phases differing in their lattice parameters were identified and quantified by Rietveld refinement in commercial YSZ samples. The Y₂O₃ content of these phases, assigned as Y-lean t and Y-rich t^{''}, was derived from tetragonality c/a. The fracture toughness of the materials increased with increasing weight fraction of phase t. In the recent study, the materials KATANA Zirconia YML (Kuraray/Noritake) and IPS e.max ZirCAD Prime (Ivoclar Vivadent) were characterized. According to the manufacturers, both materials show a gradient in Y₂O₃ content from 5 – 3 mol% from surface to bottom to combine favourable optical appearance of Y-rich and better mechanical performance of Y-lean ZrO₂(Y). In order to assess the gradient profile, 10 slices with thickness of 1 mm each were sawed from both samples. Each slice was analysed as in [1]. All slices contained the phases t and t^{''}. In e.max ZirCAD Prime, constant decrease of t^{''} fraction occurred between depths of 2 and 5 mm, a plateau was reached thereafter. In KATANA Zirconia YML, the decrease in t^{''} started at 4 mm depth, reaching an intermediate level at 6-7 mm and then a constant plateau after 8 mm. The bulk Y₂O₃ content followed the same scheme. Hence, the proposed decrease of Y₂O₃ content from surface to bottom, resulting in decrease of Y-rich t^{''}, was confirmed for both samples, but in different profiles.

P-type thermoelectric oxides for low-power transverse thermoelectric generators TTEGs

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Thermoelectric generators (TEGs) based on oxide materials are non-toxic, cost-efficient devices that convert heat directly into electricity employing the Seebeck effect. In this work, two different *p*-type oxide thermoelectric (TE) materials were prepared by solid state reaction for building transverse thermoelectric devices with enhanced output power for low-power applications. The TE materials are lanthanum copper oxide La_2CuO_4 (LCO) and calcium cobalt oxide $\text{Ca}_3\text{Co}_4\text{O}_9$ (CCO).

A transverse thermoelectric generator (TTEG) consists of alternating layers of a thermoelectric material and a metal, these layers are tilted at an angle α , with respect to the temperature gradient direction. For building a TTEG, analytical calculations of the maximum figure-of-merit $Z_{\text{tr}}T$ and the maximum power factor PF as a function of TTEG tilt angle α and the metal-to-ceramic thickness ratio (r_{t}) were accomplished. In addition, 2D and 3D FEM simulations of TTEGs and transverse multilayer TEGs were carried out via COMSOL-Multiphysics software to overcome the limitations of the analytical calculations. In order to verify these analytical calculations and COMSOL simulations, TTEGs based on strontium doped LCO (LSCO) with screen printed Ag layers were built and measured in our laboratory. The preliminary results of a 5-layer TTEG with the dimensions of $16.4 \times 10.3 \times 3.6 \text{ mm}^3$, a silver layer thickness of $58 \mu\text{m}$, hence metal-to-ceramic volume ratio of 3%, and a tilt angle of 66° , show an output power of 2.1 mW at $dT = 150^\circ\text{C}$. Moreover, for a TTEG with a silver layer thickness of $144 \mu\text{m}$, and metal-to-ceramic volume ratio of 8%, the output power is 3.3 mW at $dT = 150^\circ\text{C}$. These experimental results are consistent with the analytical calculations and COMSOL simulations.

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Chemically Engineered Mesoporous Silica Nanocarriers for Tumor-Specific Localization and Drug Delivery

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Cancers or some subtypes of cancer show heterogeneous tumor biology with a high risk of reoccurrence and metastasis and, therefore, therapeutic options for patients are limited. In addition, the low solubility of the molecular drugs fetches poor efficacy and systemic toxicity. Wrapping of therapeutic molecules in nanocarriers such as mesoporous silica, and liposomes can alter their solubility and bioavailability in the bloodstream via various mechanisms. The fusion of ligands on the surface of the drug nanocarrier facilitates their biodistribution overcoming the intrinsic physiological barriers of clearance through the innate immune system and ensuring the safe delivery of therapeutics to tumor sites. We developed nanocarriers of hydrophilic and hydrophobic drugs for tumor targeting and dual therapeutic responses by integrating complementary functionalities and payloads (anticancer drugs, and radionuclides ¹⁷⁷Lu) into one nanocarrier through smart covalent functionalization strategies. In our study surface grafted ligands and stimuli-responsive polymers assist drug storage and controlled drug release. Experimental data confirmed the preferential uptake and accumulation of nanocarriers to the tumor site without affecting the healthy organs. The cellular targeting and site-selective drug delivery can be further enhanced by co-conjugating anticancer drugs on chemically engineered nanocarriers, which is an important determinant of the therapeutic index. The minimization of off-target effects and effective ligand-driven in-vivo delivery of anticancer drugs to the mice tumors illustrate the therapeutic potential of our site-specific drug delivery approach.

Mesoporous silica nanocarrier as the carrier for targeted hydrophobic and hydrophilic anticancer drug delivery

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Poor bioavailability of hydrophobic drugs is challenging and there are limited nanocarriers available for their delivery and suffers from drug degradation and poor circulation in blood. In this study the surface properties and high specific surface area of mesoporous silica nanoparticles were utilized for encapsulation of hydrophobic drugs. Electrostatic interactions were used to enhance the encapsulation of hydrophobic drugs on mesoporous silica nanoparticles. Surface of the mesoporous silica nanocarrier was modified with amine groups which were further converted to quaternary amine to enhance the positive charge on the surface of nanocarriers. For the estimation of active amine groups, embedded on nanocarriers surface, fluorescence-based approach using fluorescamine was conducted. Amine-functionalized silica nanocarrier offered a coverage of 9.4×10^{18} molecules of amines which lowers to 3.38×10^{18} amine molecules after target ligand conjugation such as folic acid. The as-prepared nanocarriers were evaluated for their drug loading capacity and release profile evaluation. 80% of the hydrophobic drug was encapsulated with the continuous release for 24h.

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Development in digital printing of ceramic surfaces

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Development in digital printing of ceramic surfaces

The presentation examines the current developments in digital printing with a focus on the tile industry.

The given paper contains an overview of the currently available ink systems, on the one hand, and an outlook on future opportunities for the industry, on the other. In particular it focus on the use of water-based ink systems for decoration as well as for the application of engobes and glazes.

Understanding processing conditions in cold sintering to enhance densification and structural integrity

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Among the different alternative low temperature sintering techniques, the cold sintering process (CSP) enables densification of ceramics at temperatures below 300°C. The process is driven by a chemo-mechanical effect activated by a compatible transient liquid phase and high pressure. This study aims at optimizing the processing conditions in CSP, taking into consideration the effect of heating rate and homogeneity of applied pressure on the density and structural integrity of cold sintered parts. A combination of non-destructive testing followed by biaxial bending and fractographic analyses were employed to identify macroscopic strength-limiting flaws. It was found that the strength of cold sintered samples can be remarkably compromised by improper processing conditions associated with temperature and/or pressure gradients related to insufficient tooling quality, resulting in delamination effects. The prevention of such defects is demonstrated by appropriate selection of the processing parameters in CSP, resulting in structurally reliable cold sintered ZnO samples with strengths exceeding 100 MPa.

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Ceramic multilayer substrate technology: Integration of functional materials

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Low-temperature co-firing ceramic technology (LTCC) allows the fabrication of robust multilayer substrates. By sintering glass-ceramic green tapes with screen printed low-resistance conductors, interposers, RF-modules, and three-dimensional IC packages can be manufactured. Besides a high integration density through the applied multilayer wiring, the use of ceramic material results in substrates with excellent heat and humidity resistance for the use in high reliability and harsh environment applications. The integration of passive elements like resistors, capacitors, and inductors can further reduce the size of the electronic module. Embedded resistors for example are commercially available and already find widespread application. However, the integration of other functional ceramics such as piezoelectric-, NTC-, or varistor-materials can expand the area of application. To allow the LTCC-integration, the functional material itself needs to be sinterable at temperatures of 900°C or below. In addition, a detailed understanding of the sintering behavior together with possible interactions between the functional ceramic and the LTCC-material is needed. This contribution addresses the potential and the technological challenges for a successful integration of functional materials into ceramic multilayer substrates. Based on the example of a tailored, screen printable ZnO-based varistor composition, relevant steps of the material- and process-development will be described in detail.

This project is part of the German BMBF funded »SAPHIR – High performance ceramics made in Tridelta Campus Hermsdorf« consortium (03RU1U162D).

Rare earth co-stabilizing of zirconia – an engineering toolbox for creating structural ceramics with tailored mechanical properties

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Transformation toughening, a stress induced martensitic phase transformation associated with volume expansion and shear is the main source of toughness and strength in zirconia structural ceramics. The commercially available portfolio of TZP (tetragonal zirconia polycrystal) materials is however very narrow and dominated by yttria and ceria stabilized zirconia materials which either lack toughness and low temperature degradation resistance or strength.

Shifting from co-precipitated starting powders to “stabilizer-coated powders” which are either made by wet chemical methods or by intensive co-milling of monoclinic zirconia and the stabilizer oxides open a new perspective to manufacture TZP materials with very favorable combinations of strength toughness and low temperature degradation resistance. Stabilizers may either be single rare earth oxides or combinations of oxides with larger and smaller trivalent cations.

By proper selection of starting powders, powder processing and sintering off-equilibrium TZP materials featuring grains with a core-shell structure can be created. The over-stabilized shell ensures good LTD resistance, the under-stabilized core ensures high transformability, transformation efficiency and toughness. Such materials are highly attractive for the biomedical field but also as a matrix material in composite ceramics.

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Radomes made of Ceramic Matrix Composites using wet nonwoven technology and their investigation in wind tunnel testing.

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Ceramic Matrix Composites (CMC) are a promising group of materials for the production of radome structures. They are characterized by their high-temperature stability, fracture toughness and constant strengths over a wide temperature range, what makes them favorable for this application. However, the complex shapes of the tip, is a challenge for the manufacturing of the component. In order to address the complex component shape, the German Aerospace Center (DLR) developed a preforming method that achieves three-dimensional near net-shaped short fiber preforms via wet nonwoven technology. The preforms were converted into fiber-reinforced $Al_2O_3/SiOC$ and $C/C-SiC$ ceramics using PIP-process and LSI-process respectively. The resulting radomes had their performance evaluated in DLR's *arc-heated wind tunnel 2* (L2K) and the *vertical test section Cologne* (VMK) wind tunnels. The tests in the L2K were carried out successfully and thus the resistance to realistic heat flows could be confirmed. In the VMK, the flow conditions were mapped according to a suitable trajectory. The experiments could also be carried out successful and were tested over multiple trials and withstood loads of up to Mach 3, and a thermal load of up to 700 Kelvin at different angles of attack. Regarding the successful tests, they reveal the potential of wet nonwovens for complex shaped fiber-reinforced materials, and present a promising application for radome designs.

Particle synthesis of CuO-Fe₂O₃ composites by Pulsation Reactor Technology and study the dispersing character as slurry for Metal Air Batteries

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IBU-tec Advanced materials AG

Copper (II) oxide-Iron (III) oxide composites are widely used materials for many different applications, such as pigment for paints, coating on magnetic tapes, catalysts or main component in metal air batteries. The precise control of the thermal treatment steps during particle synthesis is a beneficial method of tailoring the material characteristics to the required application. Due to that fine particles of CuO-Fe₂O₃ with specific properties have been thermally synthesized by the pulsation reactor. The pulsation reactor (PR) consists of a hot gas generator that produces a pulsating stream. That means: fast and unsteady-state periodic combustion of natural gas in a chamber, with a resonance tube and a separator. The particles produced using this technology are thus subjected to rapid thermal treatment as it advances in the hot gas stream. Therefore, the process is suitable for the production of powders with modified characteristics. This work describes the characteristic of the composite particles with the addition of Carbon to improve the conductivity. During the synthesis in the PR, the effect of process parameters such as temperature, residence time and configuration on properties of the produced particles were studied applying different analytical methods. According to this work, it was concluded that the PR technology could reach a wide range of composite particles with tailored properties in a continuous process. The produced particles were successively dispersed using wet milling process. Also, the effect of wet milling parameters on the dispersed particles were studied and optimized to select the best conditions for the production of stable non agglomerated suspension for the application as metal air battery slurry.

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Structure-property relationships: A-site cations redistribution in complex polar perovskite oxides

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Tailoring the electromechanical properties of a material without altering the original composition is an emerging phenomenon for the optimization of functional properties. Post-sintering annealing with varying maximum temperatures, cooling rates, and atmospheres can influence the crystallographic phases, domain structures, conductivity, mechanical properties, and the temperature stability of the electromechanical properties. However, the influence of post-sintering heat-treatment on crystal structure, domain structure, and functional properties is not well known. In this contribution, influence of annealing conditions on the functional properties, structure, and defect chemistry of A-site complex polar perovskite oxides $(A,A')B_2O_6$ will be highlighted.

A powder technological study on the solid state processing of Al-doped LLZO powder and microjet reactor coating of single crystalline Ni82 CAM powder

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Solid-state batteries are the most promising next-generation energy storage devices with high energy densities up to 500 Wh/kg, that is decisively determined by the specific capacity of the cathode materials and the use of a solid-state electrolytes. A large amount of research work in recent years has focused primarily on the development of new cost-efficient cathode active materials (CAM) based on a higher content of nickel in CAM materials such as $\text{LiNi}_0.8\text{Co}_0.1\text{Mn}_0.1\text{O}_2$ (NCM811). To increase the high energy density of more than 500 Wh/kg, the focus of research is currently on coatings of such cathode materials to increase the interfacial stability between the cathode and the electrolyte for better electrochemical performance, increasing cycling and storage life of a cell. In contrast to the state of the art processes, simple, fast, cost-effective, scalable and environmentally friendly process developments for the further development of LLZO solid electrolytes and stable NCM cathode composites will be presented and compared in the lecture. Special emphasis will be placed on the powder-technological grinding and preparation parameters as well as the coating parameters for setting special interfacial properties of the functional powders.

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Promising Applications for Oxygen Generators based on ceramic Membranes

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Fraunhofer IKTS is developing mixed conducting oxygen membranes (MIEC - Mixed Ionic Electronic Conductor), membrane separators and membrane reactors. Presently, the establishment of a spin-off company is in the focus of a public funded project.

The new company focuses on local production of pure oxygen for industrial customers. The process is based on a membrane separator driven by vacuum. The separation process uses a mixed conducting ceramic material abbreviated as BSCF ($\text{Ba}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.8}\text{Fe}_{0.2}\text{O}_{3.7d}$). Thin-walled, long ceramic capillaries are used to separate pure oxygen from air at 850 °C.

The plant costs were already decreased by improvement of membrane manufacturing and by a modular concept of the membrane plant. Typical running costs are usually lower compared to mature technologies because of a lower electricity demand. Nevertheless, the oxygen price is growing steeply with decreasing demand rate and scatters widely depending on transportation effort and infrastructure. For that reason, customers with low but continuous O_2 demand are preferred for on-site O_2 production. At the other hand, high and growing energy costs increase the running costs and decreases customers benefit.

The use of O_2 for medical purposes, fish breeding or aquafarming, welding and flame cutting is considered as well as production of process heat by fuel combustion for metallurgy, glass and ceramic production. Combustion efficiency is increased by O_2 enrichment or by oxy-fuel entailed by saving of primary fuel or by a higher production capacity. Besides, oxy-fuel enables an easy and very efficient route for CO_2 capture and utilization. Nevertheless, the largest benefit is observed for ozonization of waste water to eliminate micropollutants.

Phase transformation induced by electric field in barium titanate at room temperature

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Piezoelectric materials are part of our daily routine: whether as microelectronics, sensors or actuators. Barium titanate is the classic model for ferroelectric systems and it is indispensable for dielectric applications. In addition, its properties are highly temperature and grain size dependent. Barium titanate has excellent properties with grain sizes of approximately 1 μm , but undergoes marked weakening as the grain size decreases. However, the fundamental mechanisms behind the electromechanical behaviour are still not fully understood. A wide range of unimodal grain size distributions between 0.4 μm and 15 μm was successfully sintered via spark plasma sintering (SPS), quenched sintering and two-step sintering, avoiding abnormal grain growth.¹ Samples with intermediate grain size, showed excellent electromechanical and dielectric properties. They possess a balance between microstructural strain, existence and mobility of domain walls, which in turn allows the field induced crystal phase transformation.

In order to elucidate the structural response to an applied electric field, the samples were measured with *in situ* high energy X-rays. The structural data obtained were processed by means of STRAP² and the field induced phase transformations were quantified. This induced phase transformation is stronger in samples whose grain size distribution curve is located around 1 μm . These results contribute to the understanding of fundamental questions about the ferroelectric effect in barium titanate and consequently other similar systems.

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Actinide Alkoxides Designed for Actinide Oxide Materials

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Metal alkoxides are well-established precursors for the syntheses of phase pure metal oxides of the s-, p-, and d-block metals, as well as the lanthanides. Rare examples of actinide oxides derived from actinide alkoxides were demonstrated for the formation of UO_2 thin films or nanocrystals.^{1,2}

Although ternary actinide oxides, such as MUO_3 or M_2UO_4 (M = alkali metals), MUO_4 with (M = earth alkaline metals, transition or p-block metals) or MThO_3 (M = earth alkaline metals) have been successfully synthesized using solid state, melt or multiple source precursor approaches, they face synthetical difficulties or phase inhomogeneity.

Herein, we present straightforward syntheses to obtain ternary actinide oxide materials starting from tailored molecular precursors. Therefore, various heterobimetallic actinide alkoxides of thorium(IV), uranium(IV) and uranium(V) with several mono-, bi- and trivalent metals have been successfully synthesized. They were applied in certain processes to demonstrate a clean conversation to the desired ternary actinide oxides AnM_nO_x (An = Th, U).

Influence of Sintering Additives on Modified (Ba,Sr)(Sn,Ti)O₃ for Electrocaloric Application

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Over the past decade much effort has been paid for solid-state cooling using caloric effects such as the electrocaloric (EC) effect as an alternative to vapor compression-based technologies. Promising EC materials for prospective applications should possess large entropy change, low dielectric loss and high dielectric strength to achieve large EC temperature change in the intended working range. Therefore, we developed lead-free ferroelectric materials based on modified (Ba,Sr)(Sn,Ti)O₃ to achieve large and broad EC temperature change around room temperature. The fabrication of (Ba,Sr)(Sn,Ti)O₃ multilayer ceramic (MLC) components for high EC effects remains challenging because of the high sintering temperature of the material and abnormal grain growth. In the present work, we investigate the influence of sintering additives on the sintering behavior and the resulting microstructure of (Ba,Sr)(Sn,Ti)O₃ bulk ceramics prepared through solid-state synthesis. Adding CuO significantly decreases the sintering temperature whereas the addition of MgO leads to a significant decrease of grain size. The dielectric, ferroelectric, and electrocaloric properties of (Ba,Sr)(Sn,Ti)O₃ bulk ceramics with sintering additives will be presented and discussed in detail. The optimization of the sintering process of MLC components based on (Ba,Sr)(Sn,Ti)O₃ with MgO addition to achieve higher breakdown fields and thus larger EC effects is subject of our ongoing research.

This work is supported by the Fraunhofer lighthouse project "ElKaWe – Electrocaloric heat pumps".

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Strength Testing of AM Ceramics

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The strength of ceramic AM components may depend on the orientation in which tensile stresses act with respect to the build direction. This is due to various aspects of the manufacturing method, i.e. insufficient layer bonding, delaminations or AM-method characteristic surface structures. For digital light processing (DLP)-based photolithography, such structures may stem from overpolymerization but also from the voxel effect. In order to provide reliable material data for 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. Thus, an innovative test body is presented which allows to manufacture a specimen set of sufficient size for statistical evaluation to be printed as one part by vat photopolymerization (VPP) using lithography-based ceramic manufacturing (LCM). Moreover, this design also allows the evaluation of the influence of surface structures. Conditions for a successful test execution and stress evaluation are discussed. In order to assess the practical applicability of the method and its meaningfulness, a round-robin test has been carried out. Several participants provided test bodies manufactured in different orientations from the same material. Tests were conducted in one lab and compared to tests performed on an alternative test infrastructure. The results on strength, strength-build direction relationship and but also practical aspects of the method will be presented.

Stress-induced tailoring of energy storage properties in lead-free Ba_{0.85}Ca_{0.15}Zr_{0.1}Ti_{0.9}O₃ ferroelectric bulk ceramics

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Electrical energy storage plays a crucial role in modern electronics as well as in more efficient energy use of renewable energy sources. In recent years, efforts have been put into developing energy storage devices with high energy and high power densities. Among others, dielectric ceramic capacitors have such high power densities and also fast charge-discharge times. Therefore, they are promising candidates for devices, such as pulse power and power electronics applications. To enhance the energy storage properties stress can be used as hysteretic processes are influenced by mechanical fields. In this study, the stress-modulated energy storage properties of lead-free polycrystalline Ba_{0.85}Ca_{0.15}Zr_{0.1}Ti_{0.9}O₃ was investigated as a function of temperature from 25 °C to 55 °C. The externally applied uniaxial compressive stress of -160 MPa increased the recoverable energy storage density by 226 % to a maximum value of 274 mJ/cm³, in addition to enhancing the energy storage efficiency by approximately 10 % to a value of 88.2 %. The macroscopic mechanical constitutive behavior is presented as well as the stress-dependent dielectric properties and the Rayleigh behavior in order to elucidate the effect of stress on the energy storage properties. Importantly, the stress-induced tailoring of energy storage performance can be utilized for other dielectric ceramics to tune their extrinsic polarization mechanisms to enhance the recoverable energy density and reduce the hysteretic losses.

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3D-Printing Filament System for Manufacturing of Tailor-Made Refractory Products

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The development of filament system for manufacturing of refractory ceramics and ceramic reinforced Metal Matrix Composites has been carried out. Elaborated thermally debindable filaments were manufactured with 52 vol% of solid phase and 48 vol% of polyethylene based binder via winding aided filament extrusion setup.

The binder development was conducted with the focus on the thermal stability of the product over the whole temperature range of the thermal treatment and was based on in-depth thermal analyses. Selected filaments based on the AR78 spinel were manufactured and subsequently printed into form of casting nozzles using Xerion Fusion Factory Printer. Elaborated thermal treatment for binder removal and sintering ensured very good quality of refractory products.

Sintered nozzle prototypes were subjected to thermal shock at 850 °C for 15 min with evaluation of the impact of thermal shock on their mechanical and structural properties using strength tests, SEM analyses and computed tomography. The final appliance test was carried out in the steel casting simulator by pouring approx. 100 kg of steel at 1620 °C through the preheated to 600 °C nozzle prototype.

Elaborated FFF-technique with internal layer-to-layer structure allows the production of thin-walled refractory products which removal of the binder causes purposeful incorporation of functional porosities resulting in excellent thermal shock resistance with capability of withstanding refractory temperatures and giving the part an exceptional lightweight potential. Developed filament enables successful utilization of broad selection of fine grained ceramic and metal powders for production of refractory parts intended to work in various metallurgical processes.

Towards hydroforming of thermoplastic ceramic tapes

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In the promising markets of high-performance electronics and mobility electrification, high-temperature-resistant ceramic components are needed as circuit carriers and electronic housings. Multilayer technologies that stack and laminate single tapes have a low material utilization rate and only allow simple component geometries for high volumes. Hydroforming is a technology that uses a fluid or gas to form complex 3D structures from simple shapes. It does not only enable new geometric design freedom but is also economical due to automatization and the possible production volumes. We developed a thermoplastic alumina tape based on a polyamide binder system that can be formed at 40-60°C. Compared to 3D-forming of conventional ceramic tapes at room temperature, the use of thermoplastic binders enables improved material flow that reduces defects such as cracks in the stretched areas. Due to the higher wall thickness and the solidification of the binder after demolding, the 3D-formed green parts are stable and show little relaxation. Subsequent sintering resulted in defect-free parts that demonstrate future use as housings.

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Ceramic and Glass Particles for Precision Drug Delivery

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Chemical processing of functional ceramics and glasses has played a key role in converging disciplines, which is especially true for biomedical applications. For example, the development of biocompatible drug-carriers that can hold back the payloads and release the drugs or antibiotics at the specific diseased area is a materials processing challenge. The selective transport and retention of drugs in sufficiently high concentrations at the target site is inhibited by various physiological barriers, which reduces or even blocks the therapeutic efficiency of molecular drugs. Therefore, advanced drug-delivery systems designed to overcome biological barriers are needed to meet the specific traits of physiological and disease-related barriers. In this context, chemically functionalized SiO₂ nanoparticles act as efficient drug-carriers to transport higher amounts of therapeutic payloads to diseased sites that also reduces the undesired off-site effects. Moreover, hollow nanocarriers can incorporate more than one drug enabling theranostic and theraregenerative approaches. Finally, silica nanoparticles can be modified with surface-bound target ligands to exploit the overexpression of receptors and promote cell specific attachment of the carriers for a localized high concentration of drug around disease sites. This talk will discuss the potential benefits of silica nanoparticles towards precision drug delivery.

Exploring Future Recycling Possibilities for Different Solid Oxide Cells

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Solid Oxide Cells are able to operate in two modes: either as electrolyzer, thereby converting electrical energy into valuable chemicals like hydrogen or a synthesis-gas ($H_2 + CO$), or by converting hydrogen or C_xH_y -gases into electricity. Additionally, a bifunctional mode, known as reversible solid oxide cell, is possible. As the German and European future energy system will be largely based on renewables like wind and solar power systems, which are available only intermittent, electrolyzers will play a crucial role in "storing" the surplus energy at times of low consumption as direct fuel. Besides energy storage the produced gas can also be used as basis for e.g. e-fuels or carbon-based chemicals. Four types of electrolyzers exist: alkaline, polymer electrolyte membrane, alkaline proton-exchange membrane and solid oxide. The first three operate at low temperatures the latter at 600-900°C. The materials used in an SOC are mostly high Cr-containing steels as interconnects, nickel as fuel-side contact, glass-ceramics as sealant and ceramics in the cell and air-side contacting.

Irrespective of the fact that all electrolyzers will play their individual role in the near or mid future, early thinking about the End-of-Life (EoL) or End-of-Use (EoU) stacks is relevant and necessary. Within the technology platform (H_2 Giga), funded by the BMBF recycling capabilities for all electrolyzer types are characterized. In this presentation, we will focus on recycling strategies for three sub-types of planar cells, namely electrolyte-, metal- and fuel electrode-supported cells [1]. Additionally, first results concerning the ceramic cell materials, aiming for either closed- or open-loop recycling, are presented.

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Destructive and non-destructive 3D-characterization of inner metal structures in ceramic packages

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Ceramic multilayer packages provide successful solutions for manifold applications in telecommunication, microsystem, and sensor technology. In such packages, three-dimensional circuitry is generated by combination of structured and metallized ceramic layers by means of tape casting and multilayer technology. During development and for quality assurance in manufacturing, characterization of integrity, deformation, and positioning of the inner metal features is necessary. Visualization with high resolution and material contrast is needed. Robot-assisted 3D-metallography is a useful technique to characterize such multimaterial structures. In that, many sections of the specimen are polished and imaged automatically. A three-dimensional representation of the structure is created by digital combination of the image stack. A quasi non-destructive approach is to perform X-ray computer tomography (CT) with different beam energies. The energies are chosen to achieve a good imaging of either the metal features, or the ceramic matrix of the structure. The combination of the respective tomograms results in a high contrast representation of the entire structure. Both methods were tested to characterize Ag and Ag/Pd conductors in a ceramic multilayer package. The results were compared in terms of information content, effort, and applicability of the methods.

Viscosity Measurement of Silicate Melts by Hot Stage Microscopy

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The viscosity of silicate melts represents a key parameter to control the ceramic and glass manufacturing processes and to understand nature phenomena (e.g., volcanic eruptions). The techniques generally used are time wasting, requiring equilibrium conditions and limited to small viscosity ranges. The reduction of testing time represents a challenge for both academic and industrial aims. For this purpose, hot stage microscope (HSM) technique was selected as alternative for a rapid viscosity determination and an experimental methodology was set up. Glasses with a measured viscosity-temperature dependence (Vogel-Fulcher-Tammann, VFT) were selected. Specimens (pressed powders) were heated at 10°C/min till melting and characteristic temperatures (CT) (Start sintering, End sintering, Softening, Sphere, Hemisphere and Melting) were found. Each CT viscosity was calculated based on the experimental VFT parameters. The viscosity was calibrated introducing correction factors based on glass chemistry to introduce the effect of surface tension on CT definition. By this way, two independent data sets can be obtained – CT (by HSM) and the corresponding characteristic viscosity (from the glass composition) – to be used to calculate the VFT parameters. The comparison between the calculated and the experimental viscosity shows a good correspondence, significantly improved with respect to previous attempts in the literature disclosing a promising prospect of this noncontact technique.

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Influence of composition and process control on the thermistor properties of doped barium titanate

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Barium titanate-based ceramics with Positive Temperature Coefficient of Resistance (PTCR) properties become increasingly important due to their numerous possible applications e.g., as cabin heaters in electrical vehicles. The change of operating conditions like on board voltage requires further adjustment of current PTCR materials. For this purpose, the whole manufacturing process must be considered and the influence of the selected process parameters and chemical additives understood.

In our contribution, the process parameters of grinding, such as grinding medium and duration, and sintering, such as temperature and sintering time, were investigated in detail. In addition, influence of donor dopant concentration of two different donor materials and acceptor dopant concentration were investigated. To evaluate the different impacts, structural studies were carried out using scanning electron microscopy and functional properties determined by temperature-dependent measurement of the electrical resistance. The results will be presented in detail. The improvement of the dielectric strength of the PTCR ceramics through the defined addition of silicon dioxide and its influence on the structure and electrical properties are subject of ongoing investigations.

Novel C-fiber/PEEK hybrid yarn based non-oxide ceramic matrix composites

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Carbon fiber reinforced silicon carbide (C/C-SiC) is a non-oxide ceramic matrix composite, which is, e.g., applied in engine nozzles for space applications or automotive brake disks. It is produced via the liquid silicon infiltration process (LSI), in which a carbon fiber reinforced polymer (CFRP) is first manufactured, then pyrolyzed under inert conditions at temperatures above 1000 °C into a carbon fiber reinforced carbon, which is eventually siliconized under vacuum above 1420 °C.

In order to obtain a C/C-SiC with favorable mechanical properties, it is crucial to create a CFRP with a good fiber bundle infiltration (FBI). Due to their low viscosity, thermosets are usually used as matrix materials. However, thermosets are not recyclable. Thus, the use of thermoplastics such as polyether ether ketone (PEEK) is of interest. Nevertheless, the high melt viscosity of thermoplastics also leads to problems regarding the FBI.

To overcome this problem, in this study C/C-SiC based on a novel developed short fiber hybrid yarn is investigated. It is shown for the first time, that C/C-SiC can be fabricated from hybrid yarn. The fiber bundles of the yarn consist of C- and PEEK fibers. The hybrid yarn is first warm pressed at about 400 °C to CFRP, pyrolyzed and infiltrated with silicon. Microstructural analyses show a desired FBI with PEEK in the CFRP, a low amount of silicon and a homogenous SiC-network in the C/C-SiC state. Phase analyses by XRD confirmed the low Si and high SiC content. Three point bending tests revealed a pseudo-ductile behavior with a Young's modulus of 28 ± 5 GPa, a strain to failure of 0.4 ± 0.1 % and a flexural strength of 97 ± 13 MPa.

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Powder aerosol deposition (PAD): a promising coating tool (not only) for functional ceramics

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Lithium ion conducting electrolytes and cathode active materials for all-solid-state batteries, sodium ion conducting membranes for future batteries, solar cells of organo-halide perovskites, materials for flexible thermoelectric materials, or even dense layers of Moon dust – such exciting applications can be manufactured with the Powder Aerosol Deposition (PAD), besides of course simple passivation films of alumina or zirconia. PAD is a novel technique to manufacture dense ceramic or ceramic-like coatings from a wide range of materials. The deposition occurs fully at room temperature, directly from the dry powder. Film thicknesses range from below 1 μm to over 100 μm . A high temperature step is not required, sometimes mild annealing improves the electrical properties. First, this contribution introduces the audience into the field of PAD and overviews recent applications. It gives also hints for future industrial commercialization.

In the second part, some scientific problems are discussed, like the influence of aerosol generation, the influence of particle and crystallite sizes as well as the question of plastic deformation of the nanosized crystallites that form during film formation. Suggestions and opportunities for future research approaches conclude the presentation.

Investigation of recycle-containing magnesia-carbon refractories in a steel casting simulator

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With the aim of conserving natural raw material resources, saving energy, reducing CO₂ emissions and because of further advantages, the recycling of refractories with subsequent reuse for refractory production is becoming increasingly important. This contribution presents studies on magnesia-carbon refractories produced using recycled MgO-C provided by Horn & Co. Minerals Recovery GmbH & Co. KG. The focus is on the possible influence of the recycled material in the refractory on the inclusion population in the steel. It is well known that non-metallic inclusions in steel can seriously affect its properties.

Sample bars that differed in the grade and the proportion of the contained recycle were tested in a steel casting simulator. The specimen to be examined was fixed in a special crucible, which also contained the steel (16MnCr5). The steel was melted under argon atmosphere at 1650 °C. In addition, 30 min after reaching this temperature, a basic slag was added to the surface of the melt. Another 30 min later, the heating was switched off. The chemical composition and the size of the non-metallic inclusions in steel samples taken from the melt and in samples from the steel slowly solidified after switching off the heating were examined by an automatic feature analysis using a PSEM ASPEX. The steel contained mainly MnO-MnS inclusions, followed by MnS-Al₂O₃. No negative influence of the recycled material on the steel cleanliness in terms of critical non-metallic inclusions was detectable.

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Combining mechanically stable ZrO₂ with electrically conductive TiO_x via Vat Photopolymerization

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Due to the combination of the advantageous properties of both ceramic materials, the material combination ZrO₂/TiO_x (titanium suboxides) offers interesting fields of application, e.g., for electrodes for the generation of cold plasmas, for cleaning or for the electrolytic splitting of water. ZrO₂ contributes its mechanical strength and electrical insulation properties to the material composite, while titanium suboxides are known for their electrical conductivity but also for low mechanical strength. Processing both materials via Vat Photopolymerization into complex shaped components is very challenging, not least because of the opacity of the dark titanium suboxide to visible and UV light. In this presentation, results of a German-Korean research project between the partners Fraunhofer IKTS and KIMS are presented. Besides the presentation of the lithography-based MultiCAMP process, the development of components from the mentioned material combination via multi-material additive manufacturing and subsequent thermal co-processing will be discussed.

Preparation of SrTiO₃ nanocubes by CO₂ laser vaporization (LAVA) and hydrothermal maturation

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SrTiO₃ is of particular interest for numerous applications such as photocatalytic water splitting, as an electrode material for thermoelectrics or as piezoceramics for sensors. We report on the processing of single-phase SrTiO₃ faceted nanocubes with high crystallinity and an average particle size of 35 nm that were prepared by a CO₂ laser vaporization (LAVA) of a coarse strontium titanate powder followed by a hydrothermal maturation and acid etching. Starting from a coarse raw ceramic powder, the SrTiO₃ nanoparticles formed by laser induced gas-phase condensation are spherically shaped with a narrow size distribution and are merely softly agglomerated by weak van der Waals forces. The composition of the nanoparticles corresponds to that of the starting powder, as confirmed by XRD and FT-IR measurements. Further hydrothermal treatment at 275°C for 4 hours leads to the formation of faceted SrTiO₃ nanocubes with increasing crystallite size and crystallinity, as demonstrated by TEM, HR-TEM and XRD measurements. However, as a result of this treatment SrCO₃ impurities are formed. During a final washing step in 0.1 M HCl, SrCO₃ impurities were dissolved and thus single-phase SrTiO₃ nanocubes were successfully obtained. The presented process facilitates the production of single-phase, highly crystalline SrTiO₃ nanopowders in sufficient quantities for subsequent use in a variety of applications, in particular for hydrogen production by photocatalytic water splitting.

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Synthesis and characterization of molybdenum-containing mesoporous bioactive glass nanoparticles for biomedical applications

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Abstract

Mesoporous bioactive glass nanoparticles (MBGNs) based in the system: $\text{SiO}_2\text{-CaO-MoO}_3$ were synthesized using a micro-emulsion-assisted sol-gel process. The synthesized MBGNs were characterized in terms of morphology, composition, in vitro bioactivity, and in vitro cytocompatibility. Scanning electron microscopy (SEM) confirmed that the particles had spherical morphology with a disordered mesoporous structure. Energy dispersive X-ray spectroscopy (EDX) confirmed the presence of Ca, Si, and Mo in the synthesized MBGNs. Preliminary results indicated that molybdenum-doped MBGNs form apatite crystals upon immersion in simulated body fluid (SBF). The effect of the release of molybdenum ions on cellular osteogenic differentiation of MC3T3-E1 cells (pre-osteoblast-like cells) will also be measured using an enzyme alkaline phosphatase marker.

Fracture Behaviour and Sub-Critical Crack Growth of Flame-Sprayed Ceramics

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The potential of flame-sprayed ceramic parts spans a wide field of applications, including high-temperature insulating linings (thermal barrier coatings) or protective coatings against wear and tear. For mechanical design purposes, fracture characteristics like crack resistance, behaviour under sub-critical loading, and the scattering of the flexural strength are crucial parameters. Therefore, the presented study aimed for an insight into the fracture processes in such flame-sprayed materials: Pure Al_2O_3 and an alumina-rich composition within the Al_2O_3 - ZrO_2 - TiO_2 system were employed for the preparation of self-supporting parts via rod flame-spraying. Both materials were investigated by means of cyclic and biaxial flexure tests in order to derive the R-curve-potential, the sub-critical crack growth parameters, and the fracture statistics. It was revealed, that the addition of ZrO_2 and TiO_2 was beneficial in terms of the crack resistance and flexural strength and also allowed for better buffering of thermal shock.

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Medical Instruments made of ATZ bioceramics - CNC versus LCM

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Alumina and YTZP ceramics have been established in joint arthroplasty since the 1970s and 1980s. Zirconia-rich dispersion ceramics of alumina and YTZP have also been known for decades for high strength, high fracture toughness and low wear rates. ATZ (alumina toughened zirconia) is used as joint implant by Mathys since 2007. New efforts were made to use ATZ for production of a surgical cutting tool.

This paper summarizes the initial investigations on standard ceramic manufacturing (CNC) compared to LCM (lithography-based ceramic manufacturing) of a ceramic cutting tool. The product development began with the formulation of clinical requirements and the design suitable for ceramic part and manufacturing process. After defining the requirements for the ATZ-ceramic based on standards (ISO 13356, ISO 6474-2), suited raw materials were defined and the slurry development for the LCM process was started. A debinding process and thermal treatment - sintering and HIP - were elaborated. High purity and elimination of any porosity are mandatory for clinical use. The whole process was accompanied by the determination of properties such as green density, microstructure, density after the thermal processes and geometry.

From appropriate raw materials meeting chemical requirements a suitable ATZ slurry was prepared. It was used to produce green parts. By adjusting debinding and thermal processes sinter densities of about 99,00 % theoretical density were reached. The microstructure could be adjusted that grain sizes for Al₂O₃ in the range between (0.39 - 0.41) µm and for YTZP in the range between (0.38 - 0.46) µm were produced.

First cutting tools made of ATZ ceramic were successfully realized with conventional CNC production and LCM.

Investment Casting and the evaluation of zircon in ceramic shells

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This presentation will provide an insight into the investment casting of nickel-based superalloys and the use of ceramic shells as expandable molds.

In this context, zircon is presented and evaluated with regard to its thermal, chemical and thermo-mechanical properties.

At the end, an outlook of the material-side development will be given and discussed.

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LTCC – Auslaufmodell oder Enabling Technology für neue Anwendungen?

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LTCC technology (LTCC – Low Temperature Cofired Ceramics) has been established for many years for the production of highly integrated and reliable ceramic PCBs, components (filters, LNA), antenna modules and sensors. The intrinsic material properties (CTE adaptation to Si, high thermal conductivity and operating temperatures, excellent dielectric properties) in combination with technical features (multilayer structure, integration of passive functions, non-electrical functions) make LTCC a perfect choice for the design and manufacturing of microsystems for harsh application conditions or high operating frequencies.

The energy transition and electrification of transport are changing the market requirements with regard to the electronic systems required. In the future, engine and transmission controls for combustion engines will lose importance, but systems for autonomous driving or real-time communication will gain in importance. New fields of application with high potential such as supercomputers and quantum sensors are on their way from research to industrial application. For this, the environment for electronics is changing significantly (high vacuum, ultra-low temperatures) and there are material and technological challenges that need to be solved.

The presentation gives an overview of the current performance of LTCC technology as well as some research topics that are being addressed to fulfill the requirements in new applications.

Precursor Design for Metal Chalcogenide Nanofilms as Catalysts for HER in Electrochemical Water Splitting.

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In recent years, hydrogen gained interest as energy storage for sustainable harvested energy. A common method for hydrogen production is the electrochemical water splitting. Promising electrode materials for this method are metal sulfides and selenides, whereby thin film coatings of these materials can be used as cost effective alternatives. Since the availability of precursors for metal chalcogenides is limited, the demand for novel molecular structures is high.

In this work, nanoscale thin films of metal sulfides were produced by chemical vapor deposition (CVD), for which the design of precursors was tailored in a targeted manner. In this work, different metal chalcogenides were obtained by the variation of ligand classes and replacement of the heteroatom. The synthesis of precursors containing sulfur and selenium was successful. Complexes with the transition metals of cobalt and nickel were synthesized and provide ideal precursors for CVD.

The conversion could be demonstrated by various analytical measurement methods. Thin films of Ni_3S_2 were successfully generated. With the application in the photoelectrochemical cell (PEC), its suitability as a novel material with good electrical properties was demonstrated. Thus, thin films of metal chalcogenides represent promising materials as catalyst in the water splitting reaction

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Improving Electrode Materials by Nanoscale Coatings using Plasma Enhanced CVD with Application in Sustainable Energy Storage

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The several applications for batteries demand ongoing innovation and new energy technologies as attractive alternatives for energy storage. Batteries are now an essential component of modern life, used in everything from energy reservoirs for renewable energy sources to smart devices like electric automobiles and smartphones. Solid state batteries are now attracting more and more interest due to their potential to replace traditional batteries as a new generation device promising both, more safety and higher performances. The reduced flammability is attributed to solid state batteries as their electrolyte mostly composes of ceramic, polymeric or composite (ceramic-polymeric) materials. In this work, metal oxide nanostructures will be used as coatings on electrodes with application in solid state batteries. The presentation of different metal oxide coatings will be achieved by using volatile precursor gas phase deposition methods such as the plasma enhanced chemical vapour deposition (PECVD). In this context, the interfacial materials and composite electrolyte structures are investigated for their changes and influences on the performance of the batteries.

Intrinsic compressive stress relaxation in ceramic films manufactured by powder aerosol deposition (PAD)

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The powder aerosol deposition (PAD) is a novel method to manufacture dense ceramic coatings at room temperature. Films can be deposited on various kinds of surfaces like metals, ceramics, or plastics. This allows coating of low melting or thermally unstable materials that would not withstand the conventional coating processes.

Coatings manufactured by PAD are nanocrystalline, dense and bear an intrinsic compression stress. The intrinsic compression stress can be relaxed by a heat treatment at comparatively low temperatures. The films are usually harder than the bulk material and a thickness range between 0.5 μm and 200 μm can be realized.

Pure TiO_2 and mixtures of TiO_2 and MgO were deposited and the intrinsic stress in the films was determined in the as-deposited state and after heat treatment at different temperatures. The intrinsic stress in pure TiO_2 already starts to decrease at 100 $^\circ\text{C}$ and is largely finished at 300 $^\circ\text{C}$. The addition of MgO shifts this relaxation behavior by 50 $^\circ\text{C}$ to higher temperatures. Explanations based on the nanocrystalline structure for this behavior are discussed.

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Blacklight sintering - a disruptive innovation in ceramic processing?

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Rapid sintering has attracted spotlight attention with multiple forms of energy transfer already demonstrated. Blacklight sintering[1], using intense illumination with UV-light to transfer power directly into the ceramics, now allows to process ceramics on-the-spot without contact or container. Extraordinary practicability with minimal sample handling and process times in the seconds range, allow hundreds of iterations per day. Moreover, fabrication in quick succession immediately enables mass productions with tons-per-year capacity.

The minimal process time and the absence of any container minimize the energy needed so drastically, that efficiency competes with or even outperforms current industrial standards. Simultaneously, the efficiency gains of up to 80 % are independent of batch size. This talk will foreshadow the prospect of blacklight sintering by contrasting energy efficiency to industry standards and reflecting on scalability and associated changes in the value chain.

Removal of pharmaceuticals from aqueous solutions using hybrid ceramic membranes

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The supply of clean pollutant-free drinking water is becoming increasingly difficult due to anthropogenic influences such as contamination with pharmaceutical residues. The problem is exacerbated by the large consumption of medicines in the increasingly aging societies of the industrialized countries. In order to meet the high demands on water quality, new solutions for water treatment should be developed.

The aim of this study is to investigate the applicability of a novel hybrid membranes for the separation of micropollutants from water. The membrane was developed to combine the technological advantages of ceramic filter elements and polyelectrolyte films for the removal of pharmaceuticals from aqueous solutions.

The coating of the ultrafiltration alumina ceramic membranes with polyelectrolyte films was performed using layer-by-layer technology. By using this technology thin chains of polyelectrolytes are applied onto the ceramic surface, forming a network of nanopores. The separation by this network results in improved retention of small molecules such as pharmaceuticals and microplastics which otherwise cannot be removed using standard ultrafiltration methods. This research compares properties such as permeability, pore size distribution and zeta potential for the original ultrafiltration ceramic membrane and the newly developed hybrid membrane. The retention of widely used drugs such as diclofenac, ibuprofen and sulfamethoxazole was reported in a series of filtration tests.

The presented study offers a new perspective on the removal of nanoscale molecules from aqueous solutions and shows the potential of applying new techniques for a successful removal of pharmaceutical contaminants from drinking water.

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Cold Sintering of Functional Materials: A Path to a Possible Sustainable Future

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Cold Sintering involves a transient phase that permits the densification of particulate materials at low temperatures 300 °C and below. Sintering at such low temperature offers so many new opportunities. It permits the integration of metastable materials that would typically decompose at high temperatures. So cold sinter enables a platform for better unification of material science. Now ceramics, metal and polymers can be processed under a common platform in one step processes. With controlling the forming process new nanocomposites can be fabricated. Polymers, gels and nanoparticles can be dispersed, interconnected and sintered in the grain boundaries of a ceramic matrix phase. With the ability to sinter metal phases, multilayer devices can be co-sintered with electrodes made from metals such as Al, Ag, Fe and Cu. With appropriate binder selection, polypropylene carbonate and its de-binding at 130 °C we can remove organic binders and leave metals and other more stable polymers within the layers that then can be co-sintered under the cold sintering process and form unique combinations of materials in multilayers. This talk will cover some of the fundamentals of cold sintering, as well as some new examples of this technology across different material systems, ranging from ferroelectrics, semiconductors, and battery materials.

Mixed Ionic Electronic Conductors (MIEC) for Self-Pressurizing Combustion

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The transition to a carbon-free renewable energy economy demands innovative solutions for efficient and affordable energy conversion. Self-pressuring combustion is considered as a completely new and promising process for a direct energy conversion from fuel to power. The combustion process is conducted in a pressure vessel with constant volume. MIEC tubular membranes or Oxygen Storage Materials (OTMs) formed to pellets are the core components of the new process as they deliver the required oxygen in sufficient amount. Using of “solid oxygen” from the MIEC material takes the compression step out of the process and enables the fuel combustion to highly compressed gas. The power can than be collected by a gas expander. Comparable to the fuel cell, thermodynamic efficiency of the process is not restricted. In contrast to internal combustion engines, steam power plants or other processes based on cycling of a working fluid, a much higher efficiency is expected. Liquide ammonia and hydrogen are evaluated as alternative fossil-free fuels in different test reactors. For this purpose, a membrane reactor based on one-site closed $\text{Ba}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.8}\text{Fe}_{0.2}\text{O}_3$ for ammonia combustion and an OTM-based high-pressure reactor for hydrogen combustion are in a development stage.

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Development of Tubular One-Side Closed Porous Ni-Cermet Anode Support for Thin Film Based Membrane Electrode Assemblies

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One of the big challenges of transition to emission-free economy is the development of a carbon-free processes for ammonia production. At present up to 3% of world fossil energy fuels are demanded for the Haber-Bosch process. A decentralized ammonia production could be an essential part of the solution for this problem. One of the main objectives of the CAMPFIRE alliance is a decentralized ammonia production using renewable energies as an innovative energy source for emission-free maritime mobility. The subproject CAMPFIRE 04 deals with the development of a tubular membrane reactor for Solid-State Ammonia Synthesis (SSAS). The combination of water electrolysis and ammonia synthesis in a single device intensifies the ammonia production process and avoids the individual steps for hydrogen generation and purification. Besides, it eliminates the CO₂ emissions of the conventional Haber-Bosch process utilizing natural gas.

The SSAS is based on proton conducting ceramic thin-film electrolyte consisting of a doped barium zirconate. It is deposited by magnetron sputtering (INP) or dip-coating on a Ni-Cermet tubular anode (IKTS). Ammonia formation takes place on a highly porous Ba_{0.5}Sr_{0.5}Ti_{1-x}Ru_xO_{3-??} outer layer of the tube (LIKAT). The performance of the assembly is tested within a special designed SSAS test rig (ZBT).

100 Years Commercial Broadcasting in Germany - Challenges to Technical Ceramics

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In October 1923 in Berlin the first German commercial radio programme was broadcasted via 400m-band. Only 6 month later its first programme hour "Berliner Funk Stunde" had 100,000 registered listeners. The prehistory of this important social event was characterised by some technical milestones, that were in parts connected to technical developments in ceramics. Isolation parts made from steatite showed excellent isolation properties even at high frequencies. Fired thick film metallization were the basis for reliable electrical connectors but also for vacuum save joining of metals and ceramics. Substantial experiences with glazing of ceramics were a good starting point for successful joining of ceramics and glasses. Ceramic materials became valuable and economic alternatives to natural dielectric materials like mica. Developments during the following decades lead to technical highlights like condensators with well-defined temperature coefficient and/or high dielectric permeability, high power condensators or fully ceramic amplifier tubes. Without exaggeration we can state that challenges from HF-technics were the origin for the development of a wide diversity of functional ceramic materials. In the beginning beside STEMAG and Rosenthal the Hermsdorf Porcelain factory (later HESCHO GmbH) within Kahla AG was one of the important players in this field. In the thirties HESCHO became the technology leader for high frequency components in civil and military applications. By means of original documents from our archive examples for the vehement technical developments of that time are illustrated. With current developments made by companies at and around Hermsdorf the bridging from the early 20th century to present age is given.

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Nanoporous ceramic membranes in H₂-economy

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Hydrogen is becoming a backbone of energy supply and storage in Germany. Various cleaning and separation processes are necessary in the entire chain from H₂ generation, its storage and transport to the final use. When using classic separation processes such as washing processes, cryogenic processes, distillation, adsorption, etc. a lot of energy is consumed which significantly reduces the overall efficiency of the H₂ economy. Membranes allow separation processes with up to 85% less energy consumption^[1]. Ceramic membranes allow separations under harsh application conditions such as very high pressure or high temperatures.

Various ceramic membranes made of carbon and zeolites with pores in the Ångstrom range were synthesized on coarse pored, ceramic supports in tubular geometry. After their characterization in the laboratory, application tests were carried out on drying of H₂ after its generation and storage, on H₂ separation from natural gas after transport in the natural gas grid and on H₂ purification in the maritime sector. In any case, very excellent separation results could be achieved. An initial scaling of the membrane geometry and production took place, whereby the reproducible synthesis could also be demonstrated on an enlarged scale.

Feasibility of Fabrication of Multilayer Capacitors from NBT-based Ceramics

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During high power spikes, extreme electric fields are applied to capacitors which can raise the temperature of this component and cause failure. Thus, a new group of electroceramics is required to replace them in near-engine, or pulse power applications. Therefore, a new class of lead-free dielectric materials such as $\text{Na}_{0.5}\text{Bi}_{0.5}\text{TiO}_3$ (NBT) has emerged. However, so far, NBT suffers from low power density, non-plateau permittivity, and high loss factor at elevated temperatures. To improve such shortcomings, combining NBT with BaTiO_3 (BT), CaZrO_3 (CZ), and BiAlO_3 (BA) can tailor it for the desired application. Fabricating MLCCs with similar properties as the bulk ceramic is of advanced technology and requires controlling many parameters. More challenging than this is selecting electrode material for making MLCCs. Currently, precious metal elements such as Pt, Pd, Ag, and their alloys are being used in industry to make MLCCs. However, due to their high cost, an even more difficult challenge is to replace them with nonprecious elements such as Cu. To put Cu into the application, certain criteria have to be considered. First, the sintering temperature of the NBT-based solid solution has to be lower than the melting point of Cu. Second, since Cu is sensitive to oxidation and cannot be fired in air, a reducing atmosphere with low oxygen partial pressure is required for sintering it. However, NBT contains bismuth oxide which is from a group of reducible oxides. Reduction of Bi_2O_3 to Bi can cause serious problems such as increasing the conductivity of the ceramic which can then deteriorate the temperature dependant properties. This challenge can be overcome to some extent by modifying the defect chemistry of NBT by donor dopants.

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Tailoring 3D printed ceramics via two-photon-polymerization and lithography-based ceramic manufacturing

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Vat-Polymerization techniques promise the highest accuracy of all additive manufacturing technologies. This also applies for ceramic-AM, where the most detailed ceramic structures can be achieved at different length scales. In this work, the possibilities of using Two-Photon-Polymerization (2PP) and Lithography-based Ceramic Manufacturing (LCM) techniques are explored. Both techniques work on the same chemical principle but require different optical properties. The material requirements for these two different technologies are discussed, with special focus on the resin selection. In the case of 2PP, Yttria stabilized Zirconia (YSZ) nanoparticles were successfully utilized in high weight fractions of up to 85wt% at excellent transmittance of ca. 90%. It enables the production of technical ceramics with an accuracy of 500nm and with remarkable structural properties forming a ceramic meta material. In order to fabricate parts in larger specimen size additional technologies faster than 2PP such as LCM are required, which combine good accuracy with small printing time and reasonable object size. The versatility of this technique in fabricating ceramics with tailored microstructure and properties is demonstrated in two study cases: (i) Multilayer design of alumina and alumina-zirconia materials manufactured using the 2K-(LCM) technology, reaching a biaxial strength of 1GPa, associated with the residual stresses upon cooling down from sintering, in comparison to 650 MPa on 3D-printed bulk alumina. (ii) Textured alumina microstructure combining alignment of high aspect ratio templates with Templated Grain Growth method, to reach biaxial strength of 670 MPa, compared to 570 MPa measured on equiaxed alumina sintered. The possibility of combining both LCM and 2PP techniques for 3D printed parts is discussed.

Multi-material Extrusion Process for Composite Structures

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In many functional ceramic components, specific properties of several materials are combined. However, several materials need multiple process steps, usually. A wet paste co-extrusion technology, named SIMPEX® - Simultaneous Piston Extrusion Technology, has been developed for continuous shaping of multi material ceramic structures. Paste formulations of Al_2O_3 - Al_2O_3 : Cr^{3+} and TZP-WC/TZP combinations have been examined. Pastes were prepared without abrasion using an acoustic mixer and were rheologically characterized with a torque rheometer. Their flow behaviour was considered in the construction design of new extruder components. The compatibility of paste components was evaluated by determination of shrinkage behaviour and thermal expansion during sintering. Extermination of shrinkage joining in co-sintering with final hot isostatic pressing experiments were carried out. Microstructure and phase interface of the composite ceramics were characterized.

Finally, a demonstrator for a fully automated scalable process technology has been developed, which is suitable to produce e.g., tubular, prismatic or complex strand composite structures in continuous process of highest purity ceramics.

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Development of low-reactive setter plates based on high-purity MgO and MgO-rich materials

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Firing processes are among the most cost-intensive, but also property-determining process steps in ceramic production. The causes are complex firing units, cost-intensive, highly stable firing aids and the high energy requirement. Setter plates play a decisive role in the properties of the ceramic materials to be sintered. When sintering functional ceramic components, they must be as inert as possible in order not to initiate any reactions with the sintered material. Due to their special chemical, electrical and thermal properties, MgO ceramics offer good prerequisites for low-reactive setter plates in high-temperature technology. In the main topic "Cost-reduced firing processes" of the development project "Saphir-Processing", setter plates based on high-purity MgO and MgO-rich ceramics for the thermal treatment of functional ceramics are being developed. The presentation deals with the first results for the selection and characterization of the raw materials and the development of contamination-free processes for preparing the powder for the tape casting process. The materials were subjected to various milling processes and the grain size distributions were compared. Different dispersing agents were examined regarding effectiveness and sedimentation stability for dispersing the powders in solvents. With the addition of binders and plasticizers, the first green tapes were cast and characterized.

Hybridization of Materials and Technologies for the Manufacturing of Highly Functionalized and Reliable Ceramic Components

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Disruptive innovations require novel material and component concepts. Within this presentation, three different manufacturing strategies for ceramic components are presented and discussed, in which different materials (sequential or simultaneous manufacturing of multi-material components) or shaping methods are hybridized. On the one hand, the resulting component properties and functionalities can be further increased. Possible combinations of properties that can thus be realized in a component are electrically or thermally conductive and insulating, magnetic and non-magnetic, dense and porous or different optical properties (e.g. colors or transparency). On the other hand, the manufacturing costs can also be reduced, by combining the benefits of different shaping technologies.

Using different examples from the field of space applications (ceramic reactor for thermal decomposition of H_2O_2 , ceramic igniter, ceramic aerospike nozzle), processing (high-performance thermal cyclers, ceramic components with integrated heaters or sensors), and luxury (multi-colored ceramic components), not only the potentials and existing challenges of the different strategies are discussed, but also the potential of ceramic materials in general is shown.

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Blacklight sintering of barium titanate

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Blacklight sintering^{1,2}, a new sintering technique controlled by absorption of high energy UV light, is used to rapidly densify barium titanate. Compared with other methods, no electrodes or complex dies are required, resulting in an effective technique for energy-efficient sintering. However, careful selection of parameters is critical to the process. Phase purity is confirmed by X-ray diffraction, and with scanning electron microscopy, homogeneous microstructure throughout the sample thickness could be realized. Directly after sintering, higher electrical conductivities and respective leaky ferroelectric behavior are observed due to the higher defect concentration. This is attributed to a quenching effect from the rapid cooling. Migration and pinning of oxygen vacancies affect the polarization loop and lead to aging behavior. An additional short annealing step in oxygen-rich atmosphere results in di/ferroelectric parameters which are similar to the reference sample. This illustrates that it is possible to produce ferroelectric ceramics by blacklight sintering.

Designing 3D-printed alumina-based multi-materials with tailored mechanical response

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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 layers act as an effective barrier to crack propagation, providing a minimum strength for the 3D architecture. It could be shown that the remaining 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. These two case studies demonstrate the potential of the LCM technology in the design of multi-material complex architectures with tailored mechanical response under distinct loading scenarios.

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Processing and Investigation of phosphors in ceramic light converter tapes

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A new system of near-UV emitting LEDs and radiation-converting ceramic tapes has been developed for durable and energy-efficient illuminated signage systems.

Previous LED-based signage systems are usually very limited in terms of their colour, since either only single-colour LEDs or white-light LEDs in combination with colour filters consisting of foils are used. In addition, these organic colour filters absorb a large part of the light and age as a result of ambient UV radiation, so that their endurance is limited to less than seven years. Although the entire colour space can be displayed with RGB LEDs, but a very high level of technical effort is required to implement large format illuminated advertising in a visually attractive manner.

In a collaborative project with partners from industry and research, a new approach was therefore being pursued in which UV or blue radiation (365 - 465 nm) excites specially developed and synthesized phosphors in a ceramic converter film and red, green or blue light is emitted. For this purpose, phosphors in pure form and phosphor blends are used, which are integrated into a ceramic tape. These tapes are manufactured by tape casting and formed into a variable shape by punching out in the green state.

The thermal process is followed by debinding and sintering, which is individually adapted to the phosphors used. The ceramics thus produced are used for radiation conversion, light scattering and to protect the LEDs from external influences. In this way, energy-efficient, durable and large format displays should be designed, which can map the RGB colour space and its colour mixtures (CIE1931 colour space). An appropriate demonstrator was prepared within the project and its properties were characterised using various analysis methods.

Rapid posttreatment of powder aerosol deposited garnet-type lithium ion conductor films using LED radiation.

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The Powder Aerosol Deposition (PAD) is a novel coating technique for producing dense films from a wide range of ceramic materials. Since the deposition takes part at room temperature, the coating of heat sensitive substrates such as polymers is possible. During the deposition process, ceramic particles impact onto the substrate surface and break into nanocrystalline fragments. However, this mechanism of film formation may cause a decrease in the functional properties of the ceramics, such as the electrical conductivity.

Thermal annealing enables to restore the functional properties. This posttreatment is usually carried out by time and energy intensive oven processes. A novel approach utilizes high power light emitting diodes (LED) for the annealing process. The garnet-type lithium ion conductor $\text{Al}_{0.2}\text{Li}_{6.025}\text{La}_3\text{Zr}_{1.625}\text{Ta}_{0.375}\text{O}_{12}$ (ALLZTO) was synthesized and deposited using PAD. The electrical conductivity of the PAD layers was increased by more than a decade in less than two minutes under LED irradiation. Screen-printed temperature sensors were employed to determine the temperature evolution during the posttreatment process.

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Powder-Aerosol deposited (PAD) calcium cobaltite as textured p-type thermoelectric material

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Oxide thermoelectric semiconducting materials like p-type calcium cobaltite $\text{Ca}_3\text{Co}_4\text{O}_9$ are investigated as oxidation- and temperature-resistant thermoelectric materials for thermoelectric generators (TEGs). To realize TEGs in planar film technology, the powder aerosol deposition (PAD) method is emerging recently. PAD is a method to obtain dense ceramic films directly from the synthesized starting powders without a subsequent high-temperature step.

In the present work, $\text{Ca}_3\text{Co}_4\text{O}_9$ (CCO) powders are processed by PAD to ceramic films at room temperature. The thermoelectric properties of the films (film thickness 10 – 20 μm) are characterized from room temperature to 900°C. Additionally, the layer morphology and texture of the films will be investigated.

As result, the Seebeck coefficient of the CCO-PAD film is very close to pressed and sintered CCO-bulk materials during the 1st heating cycle to 900°C. The morphology of the films after the thermal treatment shows strongly aligned crystallites resulting in a strong texture of the films. The electrical conductivity increases strongly during the 1st heating cycle to 900°C and remains almost constant afterwards. Compared to CCO-bulk materials, the films provide higher electrical conductivity which could be explained by the oriented crystal growth in-plane direction of the film.

The relationship between thermoelectric properties and layer morphology as a function of thermal annealing parameters will be further investigated.

Thermal and mechanical characterization of an interpenetrating metal-ceramic composite

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Interpenetrating phase composites with co-continuous phases can be an expanding alternative to common composite materials, manufactured with reinforcing particles, fibers or other 2D-reinforcements. With an increase in mechanical properties and possible cost reduction, the combination of light-weight metals with ceramics is in focus of the research. To keep the light-weight potential, increase the stiffness, strength, as well as wear- and creep-resistance in comparison to the metallic phase is aimed by the combination of the materials. Furthermore, the goal is to make the coefficient of thermal expansion adjustable and prevent brittle and spontaneous fracture.

In this study an AlSi10Mg light-weight aluminum alloy with an open porous alumina foam is brought together in a gas-pressure infiltration process to manufacture the composite. The ceramic preform has an open porosity of approximately 73% with a narrow pore size distribution, manufactured by mechanical stirring of a stabilized slurry in a patented process by Morgan Advanced Materials Haldenwanger GmbH. Previous studies focused on the isotropic elastic behavior, damage behavior under uniaxial tensile and compression load and different in-situ investigation methods. The results of the characterization with a focus on the thermal and mechanical properties, is presented in this contribution, to show the potential of the introduced interpenetrating metal-ceramic composite.

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Preparation of a slurry based on LAVA 2Y TZP nanoparticles

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The laser vaporization (LAVA) of ceramic raw powder mixtures is a versatile gas phase condensation process for the production of ceramic nanoparticles. In this way, tetragonal ZrO₂ nanopowders were prepared with homogeneous yttria (2 mol %) and particle size distribution ($d_{50} = 13$ nm). Spark plasma sintering of these powders resulted in dense ceramics with high fracture toughness (13 MPam^{1/2}), strength (1380 MPa), and transformability during crack propagation (77 %). Additionally, the ceramics showed high resistance to hydrothermal degradation. However, the fabrication of complex components based on 2Y TZP nanoparticles requires the development of a suitable slurry system. Screening of dispersing agents resulted in the choice of an ammonium polyacrylate based dispersing agent. Dispersing by rotational mixing followed by ultrasonic treatment showed the best results. The maximum solid content suitable for slip casting was determined to be 50 wt %. The IEP was found to be in the range of pH 4-5. Accordingly, the zeta potential at pH 9 was -30 mV. The viscosity at pH 9 was in the range of 20 mPas for a solid content of 50 wt %, which guaranteed the processability for slip casting. The influence of sinter temperature on the microstructure and the resistance to low temperature degradation was evaluated. However, the tendency to crack formation when slurries containing nanoscale powders are used remains a problem in the production of components using the slip casting process.

Complex nitride ceramic components for various applications made by modern shaping

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Non-oxide ceramics such as nitrides still have an unrivalled combination of excellent properties, such as hardness, high mechanical strength, wear and corrosion resistance even at high temperatures. Especially silicon nitride is for high temperature applications up to 1450°C, but also for medical applications. Aluminium nitride shows a unique combination of high thermal conductivity and electrical insulation with potential for disruptive innovations in the field of high-power electronics. However, high-performance ceramic products of these materials are mainly processed with high effort by using conventional shaping method, sufficient for many various applications (e.g. cutting tools, bearings, prostheses or as substrates in electronic applications). Nevertheless, the degrees of freedom in components design are limited by the used shaping methods, especially under the aspect of constantly increasing requirements for complexity and function. With the help of additive manufacturing (AM) as a modern shaping technology, there is the possibility of significantly expanding these limits for completely new fields of applications. However, the AM processes for high-quality nitride components as well as the material portfolio are still limited and under progress.

The authors would therefore like to provide an insight into the development status of silicon nitride and aluminium nitride components at Fraunhofer IKTS using the so-called CerAM VPP process (Lithoz LCM technology), based on three interesting applications - a finger implant and an aerospike nozzle, both based on different silicon nitrides (density > 99 %) as well as a heating-cooling element based on aluminium nitride (thermal conductivity >170 W/mK).

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Vat photopolymerization of aluminum nitride components with high strength and thermal conductivity

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While lithographic additive manufacturing (AM) of oxide ceramics is starting to become well established, the use of non-oxide ceramics for this particular printing approach is still less matured. One main reason for that is the higher light absorption that non-oxide ceramic powders often have due to their coloration.

Within this contribution, aluminum nitride was developed for the use in vat photopolymerization (VPP)-based AM via a digital light processing and subsequent sintering. The mechanical, thermal, and microstructural properties of the AM parts were compared to reference samples shaped with conventional forming techniques. Thermal conductivity of AM samples exceeded $180 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$, while flexural strength of AM samples were higher than 320 MPa. These material properties in combination with the high precision of the VPP process allow the production of highly complex components that have not been feasible before and that are fully functional. The feasibility of this method to generate complex-shaped and defect-free parts was successfully shown by fabrication of a variety of demonstrator parts, including heat sinks and heat exchangers, highlighting the great potential of AM aluminum nitride parts. Based on these findings, VPP-based AM is a highly promising alternative to the conventional manufacturing methods for aluminum nitride ceramics, especially in the context of advanced thermal management operations.

Particle analysis: method transfer from X-ray sedimentation granulometry to laser diffraction, repeatable and reproducible!

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In some industries, particle sizes are traditionally determined by X-ray granulometry, which, in addition to long measurement times, also involves considerable effort in terms of radiation protection. By optimizing calculation parameters, it is possible to transfer X-ray granulometry results to the PSA series in a reproducible and repeatable manner, with a deviation of less than 5%!

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Influence of ceramic Kelvin Cell's strut shape on mechanical properties

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The replica technique is one of the most established manufacturing methods of highly porous scaffolds for bone tissue engineering. Despite a multitude of possible templates, this method is limited in terms of reproducibility and periodicity. We overcome this issue by 3D-printing of designed periodic polymer templates which we further process with the replica technique to obtain cellular ceramics with adjustable micro- and macrostructure. The periodic structure (Kelvin cells) was created using a computer-aided design (CAD) software changing the strut shape and thickness. The structures were 3D-printed with rigid polymer and afterwards processed with the classical replica technique. The combination of polymer 3D-printing and the replica technique brings us one-step closer to the excellent replication of cellular materials.

Material & technology solutions on-hand - The bridge to reduce CO2 footprints

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Our challenges to reduce CO2 emissions and lift energy efficiencies are urgent, we need fast, pragmatic solutions. The good news is that a lot of solutions are on-hand that can be quickly applied.

Examples LIB & SSB

Today's Carbon-based anodes in LIBs are the limiting factor in energy densities.

The good news: New anode materials based on 100% Silicon are on-hand to lift energy density by 30% using a PECVD process - a glass coating process applied for decades. Others apply a novel plasma "printing" process to produce fuse-bonded metallic coatings on electrodes to lift the energy density by 50%. This process originates from a steel coating process since more than 15a.

These existing and adapted processes have the potential to boost LIBs and SSB in the next years, - they are safe, scalable and have proven durability.

Examples Heat Recovery

Heat recovery systems have been existing for decades as well, - here e. g. heat pumps and heat storage systems. Hi-temp. processes in ceramic firing apply heat recovery to a good extent in e. g. drying. However, the total thermal balance still shows significant heat losses being released to our environment.

The good news: A new generation of heat pumps allows to take waste heat from e. g. drying up to 150°C, which is ideal to re-enter to the drying process. On the other side std.-heat recovering systems can only be applied when the heat is used in another process simultaneously. If this is not given, any waste heat is released to the environment e. g. during weekend or in batch firing.

The good news: New heat storage systems can be applied to store waste heat with temp. above 1000°C, - this with flexible modules, to be re-used when needed or being electrified.

We need to find and bridge existing technologies, see their potential, adapt and scale them up – solutions are on hand.

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The effects of calcination parameters of garnet-type ALLZTO solid electrolyte powders on the deposition of thick films via powder aerosol deposition (PAD) method

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To realize the All-Solid-State Battery (ASSB) future solid electrolytes (SE) play a crucial role on removing the safety concerns due to their chemical stability and exhibiting high total conductivity at room temperature (RT). The crystal phase is one of the main aspects that determines the total conductivity of a SE at RT. The Al and Ta dopants in garnet-type LLZO stabilize the type cubic phase, which provides higher conductivity in LLZO. Therefore, ALLZTO stands out as a promising SE in prospective industrial applications of ASSBs regarding to its properties mentioned above.

Ceramic powders in the desired composition can be directly deposited on a wide variety of substrate materials at room temperature via the powder aerosol deposition (PAD) method and dense films in the μm -range are obtained in several minutes. As the deposition depend on process parameters (carrier gas, flow rate, scan speed etc.), starting powder properties (e.g. particle size distribution, and crystallite size) also have to be suitable for a successful deposition.

In this work, we synthesized ALLZTO SE powders by the mixed oxide technique. The steps in the calcination process have been modified to obtain highly pure cubic phase in the structure. XRD/Rietveld and particle size distribution analyses were carried out on the calcined powders and the effects of starting powder properties on the PAD process were demonstrated. Electrochemical properties of SE thick films were determined and correlated with the purity of starting powders. The current work shows a successful deposition of cubic ALLZTO powders on various substrates. This is promising for both the industrialization of the PAD method and the utilization of ALLZTO powders in ASSB technology.

Improving the properties of SiC/SiC CMC manufactured by LSI process and development of three-dimensional components

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Increasing performance and efficiency of aero engines is mandatory for more environmentally friendly air transport. Rising temperatures, especially for combustion section and turbine structures, as well as lower weight of the components are key requirements for new materials. Together with adapted environmental coating systems and cooling features ceramic composites are good candidates for aircraft applications. They are capable of withstanding high temperatures in an aggressive environment while the density is 2/3 lower than conventional nickel-based alloys.

Liquid silicon infiltration process (LSI) for the manufacture of silicon carbide fiber reinforced silicon carbide composites (SiC/SiC CMC) has recently been optimized. By using a novel phenolic resin, the remaining unconverted carbon in the matrix could be eliminated and the silicon proportion was reduced by ~60 %. This resulted in a higher Young's modulus and improved proportional limit stress. Furthermore, our Institute of Structure and Design is investigating turbine vanes made from SiC/SiC material together with several other DLR institutes in DLR's project "3DCeraTurb". One goal of 3DCeraTurb is to design and manufacture ceramic stator guide vanes for a high-pressure turbine, to experimentally investigate the blade performance in a wind tunnel and to evaluate performance, damage and lifetime for the later application in an aircraft engine. The focus at our institute is the enhancement of the manufacturing process from plate dimensions to more complex three-dimensional components.

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Development of Ceria-stabilised Zirconia for 3d-Printing with Stereolithography Technology

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There is currently a strong need for high-performance ceramics that can be combined with new shaping technologies on the market. 3D-printing is currently one of the emerging shaping technologies that is opening up new perspectives.

While ceramic materials are brittle and subject to catastrophic failure that is difficult to predict, ceria-stabilized zirconia-based composites can provide new ceramic materials with a plastic deformation domain before rupture, excellent resistance to processing flaws and a Weibull modulus approaching that of a steel.

In current study, we explored the influence of SLA stereolithography shaping on this new ceria-stabilized zirconia-based material with unique mechanical behavior.

In order to do so, firstly, a slurry compatible with the CERAMAKER process was developed. Ceramaker refers to the patented SLA technology from 3DCeram enabling shaping of technical ceramics. This was followed by studying the influence of the shaping parameters (layer thickness, lasing power, etc.) on the green part quality. Finally, the effect of the sintering temperature on the microstructure and mechanical properties of optimized SLA-printed material was also studied.

Through this study, 3DCERAM has been able to prove that this new material could be shaped by SLA technology and some of the results of the study shall be presented – including - different sintering parameters and their influence on optimal densification and material properties, SEM observations showing original layer structures, and microstructure development attributable to the shaping process.

Refractory Masonries: Achievement of High Temperature Data and Modelling of Thermo-Mechanical Behavior

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Refractory linings are, in addition to loads due to corrosion and creep processes, particularly affected by thermomechanical stresses caused by the restricted thermal expansion of the lining. These stresses can occur within individual components as well as in bricks and can lead to plastic deformation, cracks, and material failure. Thus, comprehensive knowledge about thermomechanical behavior is mandatory for an accurate prediction of occurring stresses to design load-optimized linings. This paper presents a method for utilizing refractoriness under load (RUL) tests to determine a temperature dependent static Young's modulus for refractories. In a first step, RUL tests with a negligible load are carried out in order to determine the materials temperature dependent thermal expansion coefficient. Afterwards, several RUL tests with higher loads are carried out. The measured data of change in temperature and length are then corrected by the thermal expansion and used to construct elastic lines for several temperatures, where the elastic slope is determined using the change in length and the respective load. Thus, Young's modulus for several temperatures can be determined. The obtained values for the Young's modulus are then compared to resonant frequency damping analysis measurements and validated using a finite element (FE) model of the RUL test.

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Carbon molecular sieving membranes for H₂ separation

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Many extraordinary properties have been reported for graphene, a 2D carbon nanostructure. It has also been demonstrated that thin films made of graphene, and its oxygenated derivative graphene oxide, show unique permeation properties for the smallest molecules (He, H₂, H₂O), which is attributed to the formation of a graphite-like structure. However, before such membranes can be practically used, there are several subjects that remain to be addressed, such as making large areas of membranes on regular porous ceramic supports, the stability of the material, and also the adhesion on the support.

The novel membranes reported here are fabricated on a specially designed 8YSZ mesoporous support, which has been recently commercialized. The latter consists of two 8YSZ membrane layers with a pore size of ~5 and ~3 nm on top of a porous α -Al₂O₃ support disc or tube. This support is stable in all the harsh conditions tested so far (e.g. hydrothermal conditions, strong acids and bases). In addition, the 8YSZ support was modified with a strongly bonded carbon layer, to provide the required adhesion. The developed 8YSZ/carbon membranes are highly stable and show the same transport performance for the smallest gases as the initial graphene-based membrane.

For example, with the use of a reductive thermal treatment at 750°C, high permselectivities (> 30) for the gas pairs He/N₂, H₂/CO₂, H₂/N₂ and H₂/CH₄ is obtained.

Manufacture of diaphragms for alkaline electrolysis by thermal spray techniques

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Alkaline electrolysis is a well-established technology and does not require noble metal catalysts. However, the current density is currently too low in comparison to alternative technologies like polymer electrolyte membrane electrolysis. An important factor for improvement is the ionic conductivity of the diaphragm and the performance of the electrodes.

Higher operating temperature above 100°C can improve both, ion conductivity and electrode performance, at the same time. On the other hand, degradation mechanisms as corrosion and dissolution reactions are accelerated by increased temperature.

As standard Zirfon diaphragms are not suitable in this temperature range, zirconia based stable diaphragms have been developed by thermal spray methods. The atmospheric thermal spray (APS) technology allows the manufacture of mechanically stable layers with a certain porosity level. For the necessary mechanical stability of the diaphragms a low porosity level and a rather high thickness is needed, however, a good performance in the alkaline electrolysis requires good ionic conductivity, i.e. the opposite (high porosity and low thickness). The paper will present results on the optimization of the microstructure using different process conditions and also special pore forming additives. The performance of such diaphragms in alkaline electrolysis will be shown and compared to standard Zirfon diaphragms.

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Microstructural analysis of carbon-bonded SiC refractory castables corroded by CaO-SiO₂ slag

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SiC as a refractory material is often used in the iron and steel making industry or in waste incineration plants. Carbon-bonded SiC castables with improved slag corrosion and thermal shock resistance were designed. Crucibles and impeller with different carbon content were prepared, tested with CaO-SiO₂ slag under inert conditions to investigate the slag corrosion. This study addresses the microstructural evolution of carbon-bonded SiC refractory material degraded by the CaO-SiO₂ slag infiltration and corrosion. The infiltrated area has been analyzed by digital light microscope, SEM and EBSD with a focus on infiltration depth and the structure influencing factors, such as carbon content, the open porosity and pore size distribution. It has been found that the slag infiltration can be influenced by the carbon content and open porosity as expected. However, the infiltration is not primarily controlled by the open porosity and the carbon content, but distinctly by the pore size distribution. It has been reported that the oxidation resistance of SiC grains is face oriented. Therefore, the primitive cell structure of SiC coarse grains embedded in slag after corrosion was investigated by EBSD in order to identify the SiC grain face orientation. Despite rounded edges, no clear face-orientation controlled SiC dissolution by a CaO-SiO₂ slag could be detected.

Powder bed fusion of crack healing ceramics

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Laser-based powder bed fusion (PBF-LB) of ceramic materials have not been developed sufficiently. The process- induced crack formation is the key challenge of the associated additive manufacturing method, which prevents its successful industrial implementation. Therefore, we have adopted a strategy to accept crack formation as an inherent characteristic of the manufacturing process while enhancing the mechanical properties of the parts by repairing the cracks using a thermal post-processing. Crack healing was successfully demonstrated in the PBF-LB-manufactured alumina titanium carbide (TiC) composite parts after thermal treatment in the air at 900 °C. Crack healing is achieved through the phase trans- formation from TiC to TiO₂ in the form of rutile. The built parts exhibited a mechanical strength up to two times higher than values demonstrated in the previous studies on alumina PBF-LB without preheating, achieving an average flexural strength values above 200 MPa.

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Characterization of zirconia toughened alumina foam filters and bulk materials

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Ceramic foam filters are state of the art for the purification of metal melts like aluminum, steel, iron and copper. During the application of ceramic foam filters, they have to withstand sudden thermal shock without any failure or dimensional change. Obviously, the higher the processing temperature of the metal, the higher the thermal shock and thus the higher the demands on the filter material. In this study the suitability of zirconia toughened alumina as a material for ceramic foam filters for copper melt filtration is to be investigated.

The physical and thermomechanical properties of the different zirconia toughened alumina compositions are characterized both as bulk material and as ceramic foam. The examined properties are the mechanical strength, Youngs Modulus, pore size distribution and thermal shock resistance.

Influence of matrix densification on the properties of weak matrix oxide fiber composites (OFC)

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Like other ceramic matrix composites (CMC), oxide fibre composites (OFC) are offering a quasi-ductile fracture behaviour due to embedded ceramic fibres in a ceramic matrix. OFC offer high strength for application temperatures up to 1100 °C depending on the used fibres. If the fracture behaviour of the OFC is derived by the weak matrix concept, a certain amount of porosity is needed to enable the damage-tolerant behaviour. The influence of a decreasing porosity on the mechanical properties of a Nextel™ 610/Al₂O₃-ZrO₂ composite was investigated. The standard material was reinfiltrated with zirconium-n-butoxide up to seven times thereby decreasing the open bulk porosity. Matrix porosities in the range of 34 - 47% were investigated. The microstructure of the samples was investigated in regard to possible porosity gradients due to reinfiltration and the effects of matrix densification. The three-point flexural strength and interlaminar shear strength (ILSS) were used to determine the influence of the porosity on the fibre- and matrix-dominated mechanical properties, respectively. With decreasing porosity, the bending strength decreased. The damage-tolerant behaviour was lost at a matrix-porosity of 34 % and lower. The interlaminar shear strength increased first but reached its maximum at a matrix porosity of 35 %. At higher densities, fibres were acting as flaws and cause a renewed decrease in interlaminar shear strength.

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Robocasting of reaction-bonded carbides

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Reaction-bonded carbide structures were fabricated by a combination of robocasting and liquid silicon infiltration (LSI). Porous preforms with various complex shapes were printed with aqueous pastes consisting of SiC, B₄C and carbon powder. These samples were then infiltrated with liquid silicon to obtain reaction-bonded silicon carbide (RBSC), reaction-bonded boron carbide (RBBC), and a combination of both, reaction-bonded silicon boron carbide (RBSBC). The fabrication of carbide-based materials using LSI is of great technological interest since the high temperatures and pressures required in conventional processing can be avoided. The microstructure and the mechanical properties of the fabricated materials were investigated.

Additionally, samples consisting of several pastes were printed by using a multi-material printer. Multi-material robocasting offers the possibility of combining materials and thus their properties, as demonstrated in this work with different configurations. Layered structures with different layer heights as well as core-shell structures were fabricated.

Residual stresses that occur during processing, which in some of the materials can lead to crack formation, were theoretically calculated and experimentally determined using Raman spectroscopy.

3D printed ceramic lightweight bone substitutes with periodic minimal surface microarchitectures for osteoconduction and bone augmentation

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Introduction

Autologous bone is still the gold standard bone substitute in the clinic. Therefore, the microarchitecture of newly developed synthetic bone substitutes, which reflects the distribution of material in the scaffold often, aims to recapitulate the microarchitecture of natural bone. The microarchitecture of natural bone, however, is optimized towards a mechanically stable lightweight structure. In context of synthetic bone substitutes, the application of triply periodic minimal surface (TPMS) algorithm could yield in stable lightweight microarchitectures, which, despite their demanding architectural complexity, can be produced by additive manufacturing. In this study, we produce scaffolds from hydroxyapatite (HA) by a lithography-based layer-by-layer methodology and characterized them for compression strength, osteoconductivity and bone augmentation.

Results and Discussion

The in vivo results, based on a rabbit calvarial defect and a bone augmentation model showed that bony ingrowth into periodic minimal surface microarchitectures, as a measure of osteoconduction and bone augmentation was as good as in pore based or lattice derived microarchitectures. The compression strength and the microarchitectural integrity in vivo, however, were significantly higher for TPMS microarchitectures.

Conclusions

Since TPMS and other periodic minimal surface microarchitectures are true lightweight structures, optimized for high mechanical stability with a minimal amount of material, such microarchitectures appear most suitable for bone substitutes used in the clinic to treat bone defects in weight-bearing and non-weight-bearing sites and for bone augmentation in context of dental implant placement.

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Three-dimensional extrusion on demand for ceramic-metal-composite parts

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Plastic paste extrusion is state-of-the-art in the manufacturing of ceramic materials. The process was also successfully applied on metal and metal-ceramic composite materials using a combination of powder metallurgical methods and ceramic processing. Here, the powder raw materials are mixed with an aqueous organic binder system in order to form a plastic paste at ambient temperature. However, the geometry of the extrudate is limited since the pastes are pressed through a rigid die with constant cross section. So, each component to be manufactured demands a separate die. The production of dies for specific structures (e.g. honeycombs) is complex and expensive and the extrusion process cannot shape 3D-structures with varying cavities or undercuts without additional processing effort.

The new approach of three-dimensional extrusion on demand addresses these disadvantages. The plastic material is supplied from a separate extruder while the shape being manufactured is generated by a 3-axis machine with computerized numerical control. Providing the pastes continuously or non-continuously from the extruder enables the manufacturing of complex or hollow structures. The 3D-manufacturing comprises the computer-aided design, the formulation of the plastic paste from the ceramic and metal powders and an aqueous binder system, the 3D-shaping and the thermal treatment. Drying proceeds environmentally friendly due to the use of water as solvent and the organic binder can be removed by thermal debinding. The final consolidation during sintering in inert-gas atmosphere transfers the materials into their final state. The invented method enables future applications for a wide range of materials, such as metal-ceramic composites.

Additive Manufacturing of nanoparticle-based ceramics

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Over the past decade, additive manufacturing, in particular the fused deposition modeling (FDM) process for the fabrication of small-scale 3D polymers, has progressed from modern scientific research to industrial mass production. The present study aimed to transfer this approach of free-form fabrication of polymer-based filaments to other classes of materials such as glass and ceramics, while enabling the use of current FDM 3D printers for industry-standard filaments. To this end, processing parameters have been developed for each individual manufacturing step, from polymer-ceramic synthesis through filament extrusion and 3D printing to the final debinding and sintering stages. For this purpose, a polymer blend of polyvinyl butyral (PVB) and polyethylene glycol (PEG) was infiltrated with crystalline SiO_2 and ZrO_2 nanoparticles, respectively. The nanoparticles prepared by laser vaporization (LAVA) were homogeneous, monodisperse, and softly agglomerated and had an average particle diameter of 18 and 17 nm at a production rate of 33 and 27 g/h, respectively. A filling level of up to 25 vol% of nanoparticles in the polymer-ceramic mixture allowed the production of filaments by extrusion, which could be printed using a commercial 3D printer. After subsequent two-stage debinding of the PVB and PEG components and final sintering, porous ceramic bodies with high dimensional stability, strength, and damage tolerance were obtained.

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Luminescent nanoparticles synthesized by laser vaporization (LAVA)

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Luminescent nanoparticles are of outstanding importance for various modern scientific and industrial fields. These range from fluorescent markers for biomedical applications to advanced display technologies as well as illumination devices using long-lasting phosphors. In recent studies, we have investigated the synthesis of luminescent nanoparticles by laser vaporization. This versatile gas-phase condensation process enables the synthesis of doped nanoparticles with an average size below 50 nm using microscale starting powders such as ZrO_2 , $SrTiO_3$, $MgAl_2O_4$, or $SrAl_2O_4$ mixed with luminescent rare-earth- or transition metals containing oxides such as Eu_2O_3 , EuO , Er_2O_3 , Dy_2O_3 , or Ti_2O_3 . By exploiting the advantageous properties of NP, we were able to achieve increased emission intensities for wavelengths over the entire visible electromagnetic spectrum. Varying the oxygen partial pressure during synthesis or during subsequent thermal treatment allows to tailor the crystalline phase and composition of the ceramic matrix, as well as the oxidation state of the phosphor, to meet individual requirements for specific applications. In addition to instant fluorescence, we have provided nanoparticle-loaded contact lenses for light therapy with long-lasting phosphorescent properties that enable improved quantum yield and uniform, steady energy transfer over an extended period of time.

Development of filter starter tubes for industrial continuous casting of steel

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Filtration is a promising approach to increase the cleanliness of cast steel melts. However, the large quantity of processed steel and the harsh casting conditions during state-of-the-art continuous casting make the application of conventional filter components particularly challenging. One approach is to immerse the ceramic filter in the tundish in order to allow for flexible removal and exchange of the component in the case of premature clogging or failure. Depending on the tundish design, this technology is not always feasible. In the present study, extruded refractory materials were investigated regarding their suitability for the manufacturing of large-scale cellular filter starter tubes for fixed implementation at the bottom of the tundish. The applied material had to allow for defect-free extrusion of the cellular component and high resistance against oxidation, corrosion, erosion, thermal shock and mechanical stresses at high temperatures during the continuous casting process. Therefore, bar-shaped samples of alumina-based compositions were extruded using a cellulose-based plasticizer. The density, porosity, shrinkage behavior, mechanical properties and thermal shock resistance of the samples were investigated depending on the addition of coarse alumina fractions, of ZrO_2 and/or of carbonaceous binders and fillers. Al_2O_3 - ZrO_2 materials with coarse alumina fractions, either sintered or carbon-bonded, provided the most promising property profile and were used to manufacture prototypes of filter starter tubes.

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SiOC membranes for water cleaning and emulsification

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Polymer-derived ceramics (PDC) can be flexibly processed from polysiloxanes using a variety of different shaping processes, whereby their surface properties can be influenced. In this paper, some examples of ceramic flat membranes with symmetrical or asymmetrical membrane structure produced by tape casting or phase inversion tape casting are shown, where the latter process has been adapted to a preceramic polymer for the first time. The production of only partially converted hybrid ceramic ($T < 600^{\circ}\text{C}$) or ceramic SiOC membranes ($T < 1000^{\circ}\text{C}$) is controlled by the pyrolysis temperature. While membranes pyrolyzed at lower temperatures exhibit distinct hydrophobic surface properties, high pyrolysis temperatures result in more hydrophilic surfaces. The use of SiC and TiO_2 particles as filler materials should further modify the porosity/pore sizes, the surface properties and introduce photocatalytic properties into the symmetric membranes. The membranes prepared show narrow pore size distribution (0.1-3 μm) and medium porosities (25-75%), which qualify them for use as microfiltration membranes and were investigated with respect to their water permeability, oil-in-water emulsion generation, and/or separation of oil-containing emulsions.

Development of perovskite-based alternative fuel electrodes for solid oxide electrolysis cells (SOECs)

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Solid oxide electrolysis cells (SOECs) have become a key technology in the field of climate-neutral energy supply. This is due to their ability to produce various chemicals and gases (e.g. hydrogen, syngas) using electricity from renewable sources. One big issue regarding the commercialization of SOECs is the degradation of the currently used electrode materials, especially the Ni-based fuel electrode. Therefore the development of alternative materials which combine sufficient performance with the lowest possible degradation rate is needed. Perovskite-based materials have been investigated in the last years as all-ceramic possible substitutes. In this work, four alternative materials (i.e. strontium-iron-niobate double perovskite (SFN), a strontium-iron-titanate material (STF), a lanthanum-strontium-titanate (LST) and a lanthanum-strontium-iron-manganese (LSFM)) were synthesized and their chemical compatibility with a commonly used 8YSZ-electrolyte was analyzed. At this point, SFN double perovskite shows the lowest interaction between the electrode and electrolyte after thermal treatment. After the evaluation of these results, button cells containing alternative fuel electrodes made of SFN and STF were electrochemically measured. These electrochemical tests of full cells, running for 7 days under typical SOEC operating conditions (e.g. temperature, humidity and current density), give a first impression of the performance and the degradation of the alternative fuel electrode materials.

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Thermally and mechanically stable superhydrophobic glass coatings containing nanoparticles

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Superhydrophobic coatings continue to be of great interest in science and industry due to their wide range of applications. Functional surface coatings that protect a surface from environmental factors such as heat, moisture and rain result in longer surface durability. As an alternative to oxides such as SiO_2 and TiO_2 , hexagonal Boron nitride (hBN) particles can also be used for coatings due to its high temperature stability, inert properties and antimicrobial effect.

In a first step hBN particles were functionalized with 1H,1H,2H,2H-perfluorooctyl-triethoxysilane (PES). Glass coatings containing functionalized nanoparticles were prepared on soda lime substrates leading to a thermally stable (400 °C) superhydrophobic surface (? 180°). In addition, the commercially available polymer based resins Silikofal® HTT and Silikophen® P80/X was used as an adhesive system. After several scrubbing cycles of the coating on a sponge, the contact angle never decreased below 130°, showing high mechanical stability. The particles were investigated by x-ray diffraction (XRD), fourier transform infrared (FTIR) spectroscopy and the coatings were examined by scanning electron microscopy (SEM), atomic force microscopy (AFM), scratch test and contact angle measurements.

Raw materials for 3D-printing of refractories

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Many industries have started to implement 3D printing technologies in their manufacturing processes. The ceramics industry is already printing high added value products, focussing on very complex shapes and high precision surface definition coupled with the need for dense ceramic bodies. The construction industry focuses on large scale projects like printing of whole buildings or bridges as they need to further scale up the productivity in this sector. While the ceramics industry first of all needs very fine powders as feedstock for the additive manufacturing, the construction industry also needs to integrate coarser materials to achieve the required large scale structural integrity. Furthermore 3D printing of building materials typically requires a cold setting cementitious binder system with precisely adjusted rheology stiffening and setting times, that is able to develop the required strength without external heat treatment. This paper will discuss potential raw material options for the 3D printing of refractories. Both high temperature resistant oxide powders and aggregates, but also potential binder concepts will be highlighted. Since 3D printing of refractories is rather at its early stage, it allows to build on experience in other industries. But it can also build on existing refractory knowledge for example low cement castable technology, and castable self flowing/gelling technology as required for shotcreting.

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Effect of coated and uncoated cBN-particles on microstructure formation in Al₂O₃-cBN-composites sintered with FAST/SPS

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Cost-effective, wear-resistant materials are of great interest for a wide range of applications in a variety of industrial fields. Extremely wear resistant materials are mostly based on materials containing diamond or cubic boron nitride (cBN). Whereas materials produced by high-pressure and high-temperature synthesis are limited in size and shape, materials produced by conventional ceramic technologies additionally have the advantage of lower costs. One very promising ceramic matrix material is cBN reinforced Al₂O₃. However, the behaviour is strongly dependent on the properties of the interface between the metastable cBN phase and the alumina matrix. With the use of field assisted sintering technique (FAST) with its high heating rates and the Al₂O₃-matrix material with low sintering temperature the transformation of the metastable hard cubic into its soft hexagonal phase should be prevented. In the current study, beside uncoated cBN particles also cBN particles with different Ti-based coatings were used and the effects on the stability of the cBN particles were investigated and discussed based on sophisticated microstructural investigations.

Electrophoretic deposition of alumina protective layer for innovative interface concepts between metallic interconnect and glass sealing in solid oxide electrolysis cell

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Improving the stability of the interface between the metallic interconnect and the glass-based sealing is one of the key factors to enable the development of highly performing Solid Oxide Cell stacks. The application of an alumina-based protective coating is a viable solution for the modification of interconnect surface, to enhance the mechanical performance of the interconnect-sealing interface (i. e. for pressurised operation) and limit possible detrimental reactions during the long-term operation of the stack. An innovative aluminization process achieved by electrophoretic deposition (EPD) of aluminium precursors on Crofer22APU stainless steel is here presented and discussed. The obtained alumina layer results in increased surface roughness of the Crofer22APU steel substrate, allowing a proper tuning of the interfacial properties. Alumina-coated Crofer22APU tested in dual atmosphere Solid Oxide Electrolysis Cell operating conditions at 850 °C demonstrates enhanced performance compared to the unmodified steel substrate. The evidence proves the effectiveness of the obtained alumina layer in limiting the formation of chromates and avoiding detrimental interfacial reactions at the interface with the Crofer22APU.

An approximate 25% increase in torsional shear strength is found for alumina-coated steel/glass sealant system compared to the unmodified steel substrate: the deposition of alumina-based coating strengthens the steel/sealant interface leading to a cohesive failure of the joint.

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Combustion engine In-cylinder catalytic coatings applied by aerosol deposition

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Aerosol deposition (AD) which is also called room temperature impact consolidation is a promising technology for depositing ceramic coatings at room temperature. Sub-micro ceramic particles are accelerated by compressed carrier gas reaching high velocities in a vacuum chamber. Via kinetic impaction, ceramic particles can be packed to form coatings at room temperature. In this work, perovskite $\text{La}_{0.8}\text{Sr}_{0.2}\text{CoO}_3$ (LSCO) coatings were deposited by aerosol deposition. Due to the poor thermal stability of LSCO, the decomposition of LSCO can hardly be avoided in coating techniques as thermal spray, leading to a reduction in the catalytic performance. As aerosol deposition is a room temperature coating technology, the decomposition of the LSCO was fully avoided. The obtained LSCO coatings showed excellent catalytic performance in the conversion of hydrocarbons and carbon monoxide. Aerosol deposition showed advantages for depositing LSCO coatings than the thermal spraying technologies.

Mechanical properties of BaCe_{0.65}Zr_{0.2}Y_{0.15}O₃ – Ce_{0.85}Gd_{0.15}O₂ dual-phase proton-conducting material with emphasis on micro-pillar splitting

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BaCe_{0.65}Zr_{0.2}Y_{0.15}O₃ – Ce_{0.85}Gd_{0.15}O₂ (BCZ20Y15-GDC15) dual-phase material revealed potential for H₂ production technologies due to its exceptional H₂ permeation and chemical resistance. In this work, mechanical properties of BCZ20Y15-GDC15 dual-phase material were investigated to evaluate the mechanical behavior and develop strategies to warrant structural stability. Elastic modulus, hardness and fracture toughness values were studied using different indentation-based methods. The fracture experiments at different length-scales both revealed that the introduction of GDC15 makes the material tougher, facilitating the further design of robust and reliable components. The interpretation of the complex pillar splitting load-depth curves was aided by slice-by-slice post-test FIB milling together with in detail microstructural analysis. Thus, the road towards widespread application of micropillar splitting for dual-phase materials is paved.

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POSTER

New Hydride Modified Aluminum Complexes as Reactive Precursors for Chemical Vapor Deposition

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Abstract

Hydride modified aluminum complexes are attractive precursors due to their volatility and high reactivity of aluminum-hydride bonds beneficial for an efficient chemical vapor deposition process. Here, we present synthesis and structural characterization of mono- $[\text{HAl}(\text{TFB-TBA})_2]$ (**3**) and di-hydridoaluminum enaminate $[\text{H}_2\text{Al}(\text{TFB-TBA})_2]$ (**4**) (TFB-TBA = N-(4,4,4-trifluorobut-1-en-3-on)-tert-butylamine) as monomeric and dimeric complexes, respectively. The structural motif of compound (**3**) shows a penta-coordinated Al center formed by one terminal hydrido (H) ligand and bidentate coordination of two enaminate ligand, whereas the single crystal X-ray diffraction analysis of compound (**4**) revealed a dimerization through a bridging enaminate unit and two terminal hydrido ligands on each Al center. The solution ²⁷Al NMR showed a single peak in the region characteristic for five-coordinated Al center, which suggested that the solid-state structures are maintained in the solution. Both complexes has been investigated by both TGA and IR analysis. Application of both **3** and **4** in the CVD process produced homogeneous deposits with uniform morphologies, which transformed into crystalline phases of Al₂O₃ upon annealing and elemental composition confirmed by XPS analysis.

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In vitro evaluation of the angiogenic potential of dissolution products from bioactive glasses

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The study of new bioactive glass (BG) compositions is of great interest, therefore different approaches have been considered, for example, the incorporation of therapeutic ions, such as Sr, Zn, Mg, B and even less common ions, is a focus of investigation to enhance the intrinsic properties of BGs. Angiogenesis is an essential process in which new blood vessels are formed from existing ones and it is important for the regeneration of tissues. When appropriate stimuli trigger endothelial cells, they sprout from a root vessel, migrate, proliferate and finally align to form tubes. The objective of this study was to evaluate the tubular formation of endothelial cells after stimulation with dissolution products of silicate-based BGs. Glasses based on the 45S5 composition were immersed in cell culture media (CCM) at different concentrations. The supernatants were used to evaluate the biological effect on Huvecs, NHDF and MG-63 cells. The tubular formation of Huvecs was assessed by culturing cells in a gel-based extracellular matrix and quantified using image analysis. Additionally, the VEGF in CCM was evaluated, as well as the expression of genes involved in angiogenesis, such as Angiopoietin 1, hypoxia-inducible factor 1 and VEGF-A. The results showed that there is a dose-dependent effect of BG ionic products in the tubular formation, being the number of tubes higher for lower concentrations, however, the amount of VEGF produced from cells was significantly superior for higher BG concentrations. Additionally, the incorporation of boron in BGs resulted in the formation of more nodes and longer tubes compared to cells without conditioned media. These results provide information to understand the effect of BG in angiogenesis and their potential as biomaterials.

Improvement of air side contacting in SOCs

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Solid oxide cell technology has been developed for last decades massively as it might be one of the possible solutions in energy transition of the world in terms of electricity generation by using fuel, i.e. green hydrogen to power, or green hydrogen production by electrolysis of water [1, 2]. However, this ceramic based cell technology can be improved to increase the working efficiency with lifetime and decrease the cost of the end-product which hopefully supports market entry.

According to literature, one possible and highly profitable way of improving SOCs is to focus on the air side contacting. Relative contribution of air side contacting to the stack resistance is high which points out also that even a small improvement in this region can have a big impact on overall stack performance [3, 4]. Conventional design for this region has milled or stamped gas channels on interconnector for gas distribution but contact is typically not optimal between IC and cathode where electron transport should be proper. A solution might be to rebuild cathode contact layer by stencil printing of rib-channel-form onto the cathode. This prevents at the same time use of milling process which is cost and time consuming.

This work focuses on manufacturing and characterizing of rib-channel-form as a layer supported by electrochemical measurements to find out the best material-microstructure-design variation.

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Magnetic Field-assisted Chemical Vapor Deposition of MgFe₂O₄ Films for Photoelectrochemical Water Splitting

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Single-phase magnesium ferrite films (MgFe₂O₄) were grown by magnetic field-assisted chemical vapor deposition (CVD) of mixed-metal precursor [MgFe₂(OtBu)₆] as a function of the applied field strength (B = 0.0, 0.5 and 1.0 T). The formation of monophasic MgFe₂O₄ deposits was confirmed by X-ray diffraction and photoelectron spectroscopy. The cross-sectional analysis (FIB-SEM) of the film revealed an increased densification and crystal growth, upon application of the magnetic field when compared to zero-field deposition. The MgFe₂O₄ films deposited under zero-field and field-assisted conditions were used as electrodes in a photoelectrochemical (PEC) water-splitting reaction. All the three samples showed a stable performance and photocurrent values, however, the photocurrent was found to gradually decrease with increasing applied magnetic field (0 T: 5.74 mA/cm², 0.5 T: 2.33 mA/cm², and 1 T: 1.33 mA/cm² at 1.23 V (vs. RHE)), which is possibly due to change in absorption properties and crystal orientation, decreasing photo absorption intensity provided by the UV-vis results and the latter being evident in the disappearance of (220) peak in MgFe₂O₄ films grown under the influence of the external magnetic field.

Glass-based sealant solutions for α - α -alumina joints in Na-Zn cells operating at 300°C

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In the framework of the SOLSTICE project (European Union's Horizon 2020, grant agreement No 963599, www.solstice-battery.eu), a solid electrolyte Na-Zn cell, based on the ZEBRA® battery concept, is being optimised. Two different glass-based systems have been considered as potential candidates for obtaining a proper joining between the α -alumina electrolyte and the α -alumina cap, to ensure an appropriate separation between the cathode and anode compartments. The key issues to be addressed for this application are the corrosive environment (Na liquid, vapor, and NaAlZn) at 300°C and the long-term durability.

A systematic characterization of both a silica-based glass and a silica-free glass, considering different characteristics and joining temperatures (assessed by thermal analyses), is reviewed and discussed.

Water-based ink formulations containing glass particles are investigated in terms of rheological properties and printability by using robocasting, thus avoiding the use of organic solvents and minimizing the number of waste in the whole process.

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Lamination of magnesium aluminate spinel ceramic by organical adhesive

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During the last years, the use of polycrystalline transparent ceramics in optical components has constantly increased. By integrating the transparent ceramics division of the CeramTec-EETEC GmbH (PERLUCOR®) Fraunhofer IKTS significantly expands its existing competencies in the development of transparent ceramics.

A research and development center for transparent ceramics at IKTS is built up (funded by the Free State of Thuringia). The research work is among others focused on the lamination of transparent ceramic with other material. Transparent spinel ceramics represent a wear-resistant alternative for protective components instead of commercially available materials such as polycarbonate, tempered or surface hardened glass. Various applications such as optical scanners, mobile displays or optical sensors require a lamination of the spinel ceramic with optical quality. To produce the laminates, investigations were carried out in terms of surface pretreatment using microwave plasma, the application of organic adhesives and their curing. The mechanical properties of the composites produced with glass and plastic substrates were investigated. Based on the produced laminates the optical qualities of the composites are determined and their suitability for specific applications is assessed.

Secondary Phase Formation during Sintering of STF

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Mixed ionic-electronic conducting perovskites play an important role in the development of membranes for different application, such as oxygen separation, fuel cells or membrane reactors. Among those perovskites iron-doped strontium titanate with the formula $Sr_x(Ti_{1-y}Fe_y)O_3$ (STF) is a very promising candidate, due its viable performance and supreme stability. It was found that under certain conditions during sintering of STF with $y = 0.25$ a second phase, made up of strongly textured hexagonal crystals forms. First investigations hinted that those crystals consist of strontium-hexaferrite. However, the exact conditions of the phase formation as well as the impact on performance remain to be investigated. During this work, the secondary phase was characterized via XRD, RAMAN and TEM/EDX. The conditions of formation were specified via sintering experiments with different parameters in regards of stoichiometry, temperature, and atmosphere. We confirmed that the second phase consists of strontium hexaferrite, and not only grows on the surface but is also present in the bulk. Additionally, we observed that strontium-non-stoichiometry improves the sintering behaviour of STF but also favours the formation of the second phase. We found that temperatures above 1300°C led to formation of strontium-hexaferrite in STF with an A-site-occupation of 97%, while a fully occupied A-site prevented formation for all measured samples. Furthermore, we could show, that presence of strontium-hexaferrite does not impact performance of STF in terms of conductivity. This research helps to understand the sintering behaviour of iron-doped strontium titanate and deduces an improved sintering protocol.

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R-curve envelopes for lithium disilicates with micro- to nanometric microstructures

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Objectives: Recent investigations on the mechanics of dental lithium disilicates have gathered evidences of a probable R-curve behavior, which is difficult to ascertain directly. Here we use an indirect approach to obtain the contours of the R-curve using the relationship between strength and initial crack size obtained from quasi-static fracture toughness tests on specimens having a range of initial crack sizes (a, j).

Methods: Using 6 dental LS2 glass-ceramics with varying crystal sizes, Surface-Crack-in-Flexure specimens (3 mm x 4 mm x 25 mm) were indented with a Knoop indenter under forces varying from 19.8 – 98 N, grinded to remove the lateral cracks, and tested in four-point bending or in biaxial flexure. Fracture toughness was calculated using the Y-factor of Strobl et al. The relationship between strength and initial crack size was established in a double-logarithmic plot. The tangent condition formed by the multiple $K_{I,appl}(a, j)$ curves gave a smoothed polygonal envelope for the R-curves.

Results: The presence of an R-curve effect was confirmed all the evaluated lithium disilicates, being more pronounced for materials containing micrometric size crystals, and less pronounced for those exhibiting nanometric crystal sizes. Testing in biaxial flexure reduced the magnitude of the R-curve for one material showing micrometric lithium disilicate crystals. The nanometric material showing 41vol% β -Spodumene plus 29vol% $\text{Li}_2\text{Si}_2\text{O}_5$ presented the lowest R-curve, compared to other two nanometric materials containing only 29-32 vol% $\text{Li}_2\text{Si}_2\text{O}_5$.

Conclusions: Dental LS2 glass-ceramics show R-curve behavior, which is significantly affected by the size of the crystalline microstructure, as well as type of crystal.

Chemical resistance of commercial LTCC against thin film etching media

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Low-temperature co-fired ceramics (LTCC) are used to fabricate robust multilayer circuits. Typically, thick-film technology is applied for metallization. For specific sensor applications, thin films are deposited directly on the as-fired LTCC-surface. These deposited thin films are structured either by lift-off or by etching. The latter is less error-prone and thus preferred in industry provided the selected materials allow it.

200 nm Ni-thin films were deposited on three different commercial constrained-sintered LTCC (CT708, CT800 and DP951) by electron beam physical vapour deposition. The thin-films were structured by covering corresponding sections with a UV-curable photo resin and subsequent etching of the uncovered surface, leaving behind the desired structure. The etched Ni-thin film showed high difference in failure rate and sheet resistance regarding the used LTCC-material. DP951 had the lowest sheet resistance and no failure, whereas the CT800 had a failure rate of 40 %. The LTCC with high failure rate showed a strong chemical attack by the used etching medium. To address this phenomenon, the chemical resistance of the three different commercial LTCC (CT708, CT800 and DP951) against four different commonly used etching media (sulphuric acid, phosphoric acid, aqua regia, and hydrofluoric acid) is investigated. The dissolved ions are analyzed by ICP-OES to correlate the LTCC-composition and its chemical resistance.

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Preparation of octacalcium phosphate by co-precipitation method

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Octacalcium phosphate ($\text{Ca}_8\text{H}_2(\text{PO}_4)_65\text{H}_2\text{O}$) has been indicated as a precursor of biological apatite crystals in the bone as well as tooth dentin and enamel. Octacalcium phosphate possesses superior osteoconductivity than hydroxyapatite and tricalcium phosphate. The synthesis of Octacalcium phosphate is found to be very challenging and time-consuming. The synthesis of octacalcium phosphate is highly influenced by molarity, pH, temperature, and order of reagent addition and stirring rate. A minor variation in these conditions adversely affects the precipitation of Octacalcium phosphate. Thus current report aims to prepare octacalcium phosphate and optimize the necessary reaction conditions required for its synthesis. The co-precipitation method was used for the synthesis of octacalcium phosphate. The calcium:phosphate ratio used in this experiment was 1.33. The reaction was performed at pH 6 under a constant stirring rate. The reaction between calcium and phosphate salts resulted in the formation of octacalcium phosphate within four hours. The phase confirmation of octacalcium phosphate was analyzed by X-ray diffraction. The surface morphology of the sample was characterized by scanning electron microscopy. The formation of low crystalline octacalcium phosphate was noticed and the characteristic peaks matched the standard pattern. Fourier transform infrared spectroscopy revealed the presence of necessary functional groups associated with octacalcium phosphate and scanning electron microscopy revealed broken plate-like surface morphology. It was found that pH and temperature played a key role during octacalcium phosphate precipitation. Thus, the synthesis time for octacalcium phosphate was reduced from days to hours.

Towards the development of textured $\text{La}_2\text{NiO}_{4+\delta}$ membranes and their oxygen-transporting properties

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Due to its high chemical and thermal stability in CO_2 -containing atmosphere and its anisotropic crystal structure, the Ruddlesden-Popper phase $\text{La}_2\text{NiO}_{4+\delta}$ has attracted considerable attention in the field of oxygen-transporting membranes. To fabricate textured $\text{La}_2\text{NiO}_{4+\delta}$ membranes, the fine-grained equiaxial $\text{La}_2\text{NiO}_{4+\delta}$ matrix particles are mixed with the large plate-like $\text{La}_2\text{NiO}_{4+\delta}$ template particles, obtained by molten-flux synthesis [1], in different mass ratios. The mixtures are uniaxially pressed at 150 MPa for 15 min and then sintered at 1673 K for 15 h in ambient air to form membranes. The template particles are aligned in the membranes by the pressing force. During sintering, the growth of the plate-like particles is favored by the consumption of the small matrix particles. X-ray diffraction measurements demonstrate that with an increasing proportion of the plate-like particles in the membranes a preferred orientation of $\text{La}_2\text{NiO}_{4+\delta}$ is achieved. Scanning electron microscopy micrographs of the $\text{La}_2\text{NiO}_{4+\delta}$ ceramic membranes exhibit individual plate-like crystals embedded in the matrix, while energy-dispersive X-ray spectroscopy reveals a homogeneous distribution of lanthanum and nickel. The effect of the content of the template particles in the textured $\text{La}_2\text{NiO}_{4+\delta}$ ceramic membranes on the oxygen permeation performance is discussed in detail.

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Humidity-Induced Degradation of Lithium-Stabilized Sodium-Beta Alumina Solid Electrolytes

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Sodium-beta alumina is a solid-state electrolyte with outstanding chemical, electrochemical, and mechanical properties. Sodium polyaluminate is successfully employed in established Na-S and Na-NiCl₂ cell systems. It is also a promising candidate for all-solid-state sodium batteries. However, humidity affects the performance of this solid electrolyte. In the presentation, the effect of humidity on disk-shaped samples of Li-stabilized sodium-beta alumina stored in three different environments is shown. It is demonstrated how impedance analysis and additional characterizations were used to investigate the consequences of the occurring degradation, namely ion exchange and subsequent buildup of surface layers. Our measurements have shown that sodium-beta alumina's ionic conductivity gradually deteriorates up to two orders of magnitude. This is due to layers developed superficially during storage, while its fracture strength of 240 MPa remains unaffected. Changes in microstructure, composition, and cycle life of Na|BASE|Na cells highlight the importance of proper storage conditions: In just one week of improper storage, the critical current density collapsed from the maximum of 9.1 mA cm⁻², one of the highest values reported for sodium-beta alumina, to only 1.7 mA cm⁻² at 25 °C. The results validate former observations regarding sodium-beta alumina's moisture sensitivity and suggest how to handle sodium-beta alumina used in electrochemical cell systems. We also plan to present our newest results for the utilization of this promising ceramic in a novel electrochemical cell system.

Use of natural pore-forming agents in ceramic materials

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Porous ceramic materials can be synthesized by a variety of processes. In addition to the mechanical properties, the formation of the pores and the pore network is of crucial importance for a large number of applications. In particular, the use of natural cellulose-based pore-forming agents allows the porosity to be specifically adjusted from open-cell to closed-cell in the ceramic materials according to the applications, depending on the geometry (fiber, sphere, gel). The advantage of these pore-forming materials lies on the one hand in the natural surface structuring, and on the other hand they have a much better CO₂ balance. The potential of natural pore-forming agents and their influence on mechanical and thermal properties is demonstrated on the basis of different ceramic porous materials. The analysis of the microstructure and the pore network is carried out by means of μ CT measurements and simulations.

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PTCR behavior of lead-free BNBT-based thermistors

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Semiconducting ceramics with a positive temperature coefficient of resistance (PTCR) behavior are used as passive components for applications as heaters of inrush current limiters. Lead free BNBT ceramics of composition $(\text{Bi}_{0.5+k}\text{Na}_{0.5})_{0.03}\text{Ba}_{0.97}\text{Ti}_{n-y-z}\text{Nb}_y\text{Mn}_z\text{O}_3$ were prepared using the mixed oxide route. The Curie temperature of BNBT97 is at $T_c = 145$ °C.

In order to obtain semiconducting ceramics at room temperature, niobium ($y = 0.0005 - 0.004$) was used as a donor dopant. For $y = 0.002$, a relatively low resistivity at room temperature (~ 100 $\Omega\cdot\text{cm}$) was observed. Larger donor concentrations led to high-resistivity samples without any PTC behavior.

Additional acceptor doping with manganese ($z = 0 - 0.0006$) led to a significant increase in the resistivity jump (up to five orders for $z = 0.0006$ Mn) near T_c .

The variation of the B-site excess also showed a slight improvement in the PTC properties, especially for $n = 1.005$.

In addition, the effect of Bi non-stoichiometry ($k = -0.05 - 0.1$) on the PTCR behavior was examined. The room temperature resistivity is controlled in a small range of Bi deficiency or excess with an almost stable resistivity jump behavior, while larger variations of the Bi-content result in insulating behavior.

The obtained results allow tailoring the PTCR response of this BNBT composition through proper selection of dopant concentrations, stoichiometry and sintering conditions.

Advanced method for determination of ceramic lifetime and method-based fault diagnosis for sustainable filtration applications (presented by Maham Mannan)

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Usually, the key drivers in the selection for a particular filtration membrane solution are the preciseness for separation and economic factors. This typically comprises the number of required units, the associated cumulated energy costs for utilization and chemical costs for any required chemical dosing and cleaning. Mainly, the attention for selection is paid to the membrane's surface chemistry and roughness for cleanability purposes, the feed side fluid dynamics for a sustainable flux and the structure and morphology of the separating layer for permeance and operating pressure. As a result, introductions of new membrane systems often feature deep dives into these well-travelled aspects of membrane science, usually lacking a discussion of the systems lifetime. An advanced method was developed to quantify the end-of-life point taking the identification of specific criteria into consideration. Not only the determination of potential stresses, execution of accelerated tests as well as interactions amongst miscellaneous environmental conditions have been studied intensely but also accelerated conditions with conditions to be experienced in common operations which eventually could result in failure. The method comprises a valid determination for warranty reserves as warranty claims for failure of a product is an identifiable business cost. Also, as set and actual targets sometimes barely align, the above-mentioned method in combination with statistical approaches can be used to reflect the circumstances for malfunction independent of the origin of the stresses such as temperature, pressure, vibration, and chemical exposure.

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Dense and porous LATP electrolyte system for solid state batteries: Preparation and conductivity measurement

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$\text{Li}_{1.3}\text{Al}_{0.3}\text{Ti}_{1.7}(\text{PO}_4)_3$ (LATP) is a high conductive solid-state electrolyte for use in potential solid-state batteries. Concerning higher demands on those batteries, especially in the field of electromobility and portable devices, the solid-state concept offers an opportunity to achieve higher energy densities. In addition, with the elimination of liquid components, there is also no risk of leakage, which offers clear advantages in terms of flammability or contact with the environment.

LATP powders were prepared by the sol-gel method. The powders were analyzed by differential thermal analysis (DTA) and X-ray diffraction (XRD). Samples were sintered at different temperatures and they were examined by density measurements, impedance spectroscopy, and by scanning electron microscopy (SEM). Further, porous LATP structures are discussed, which represent a new class of materials that potentially provide more effective contact between the active material and the solid electrolyte in all-solid-state batteries. The proposed solid electrolyte foam was fabricated using the polymer replica technique. The used dip coating process demonstrates its advantages when structures with complicated geometries are to be coated in a reproducible manner. The influence of the aqueous suspension on the homogeneity and the reproducibility was examined. Parameters such as slurry solids content and pH used were optimized to produce stable LATP slurries, adjustments that could result in an efficient coating process. The structures obtained after burning out and sintering were analyzed using μCT and their electrical properties were characterized.

poXos® - pure oxygen on-site Commercialization of membrane plants for local oxygen production

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Fraunhofer IKTS is intended to commercialize a new technology for oxygen production. The patented process is based on mixed conducting ceramic membranes capable to separate oxygen from air at elevated temperatures if a gradient in oxygen partial pressure is applied across the membrane. These membranes are put into modules which represent the basic units for devices for local oxygen production from air. The spin-off company will be established in spring 2023 and will offer commercial oxygen generators under the trademark poXos® standing for pure oxygen on site.

In general, the built up oxygen generators combine the best characteristics of current commercial technologies for oxygen production such as low energy demand and high purity of the oxygen produced on-site. Especially customers with low or medium oxygen demand entailed with high oxygen costs should be addressed by poXos®.

The initial market for the new startup will be the ozonization for waste water treatment. Here poXos® generators will solve currently existing issues concerning e.g. logistics, security of supply, energy consumption and operational costs, economic footprint and process complexity.

The project poXGen (FKZ: 03EFSTH033) is part of the EXIST program and funded by the Federal Ministry of Economic Affairs and Climate Action and the European Social Fund.

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Synthesis and characterization of inorganic phosphate-based raspberry-like microparticles as coating components for bone implants

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Biomedical implants have shown great promise as bone tissue replacement in recent decades. In addition to mechanical properties, biocompatibility and the prevention of infections are crucial for a long lifetime of an implant. To enhance implant bio-integration and healing process, bioactive ceramic coatings have been developed. Especially calcium and magnesium phosphates (CaP and MgP) have received great attention due to their similarity to the mineral phase of natural bone. High cell adhesion as well as biodegradation are quite important features and depend on the porosity of the surface. However, the influence of porosity of the coating is not fully understood yet. An increase of porosity can be achieved by using nanoparticles (NPs). In particular, CaP and MgP NPs offer a number of advantages with regard to biomedical applications such as high biocompatibility and osteoconductivity. Here, we present our recent activities in the research field of the synthesis and characterization of CaP and MgP raspberry-like microparticles for the coating of bone implants. CaP and MgP NPs were prepared by a sol-gel process. In the second step, NPs were processed to micro-raspberry particles by the spray drying method. To obtain an antibacterial effect, Cu ions were also introduced in the process. The resulting microparticles could be annealed at high temperature to receive a crystalline phase. The characterization of the resulting particles was done by dynamic light scattering, scanning and transmission electron microscopy, inductively coupled plasma mass spectrometry and x-ray diffraction analysis. First coating experiments with phosphate-based particles were performed on Ti substrates by high-velocity suspension flame spraying.

Digital light processing (DLP) 3D printing of magnesium phosphate minerals and cements

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Bioceramic materials have gained increasing attention in recent decades. However, the production of complex 3D structures with high resolution is still challenging. Therefore, an additive manufacturing technique called digital light processing (DLP) is used to fabricate different 3D structures. This process is commonly based on a photopolymerizable resin containing a photo initiator and an UV or photo absorber. The resin and thus the final properties of the printed structures can be adjusted by adding fillers such as bioceramic powders relevant for bone regeneration applications. Here, a water-based and biocompatible poly(ethylene glycol diacrylate) (PEGDA) resin containing the photo initiator Lithium-Phenyl-2,4,6-trimethylbenzoylphosphinat (LAP) enables the production of 3D structures via DLP. The addition of calcium magnesium phosphate cement (CMPZ) powder, acting as photo absorber, has led to higher accuracy of the final structures. After curing the printed construct in a di-ammonium-hydrogen phosphate (DAHP) bath for hardening, the resulting mechanical properties are adjusted without post-process sintering. Solid loading of up to 40 wt.% CMPZ powder is possible, and the resins are investigated regarding their rheological behavior and printability. The resulting constructs are analyzed in respect to their surface morphology using SEM, their porosity, phase composition using XRD, and mechanical properties influenced by the hardening process for different durations.

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Efficiency of different tin oxide antimony oxide mixtures as dopants to induce selective laser induced autocatalytic metallization of alumina ceramics

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Selective laser induced metallization is a state-of-the-art technology to manufacture mechatronic integrated devices (MID) based on polymer substrates and has recently been adapted for alumina substrates enabling applications at higher temperature and in harsh environments. In this study alumina ceramics were doped with antimony doped tin oxide (ATO) in different amounts and antimony tin oxides and pressurelessly sintered in air at 1500°C. Ceramics were laser activated with green ps-lasers and state of the art IR-lasers to elucidate the respective optimum doping compositions for a reliable metallization process. It was found that the suitable compositional field using the ps-laser was extremely wide with sufficient metallization results in all cases. For the IR-laser a very specific ATO amount and composition was necessary. Besides metallization the ATO dopant strongly influence the formation of alumina microstructure and its mechanical properties. Antimony rich compositions lead to columnar grain shapes inhibit densification, reduce strength and hardness but improve fracture resistance.

Electrospun $\text{Ca}_3\text{Co}_4\text{O}_{9+x}$ nanofibers and nanoribbons: Microstructure and thermoelectric properties

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Oxide-based ceramics offer promising thermoelectric (TE) materials for recycling high-temperature waste heat, generated from industrial sources. To further improve the functional performance of TE materials, their power factor should be increased. Here, we focused on the nanostructuring of the oxide-based ceramics, resulting in multiple intermediate phases and nanopores that increase the Seebeck coefficient. This goal was achieved by compacting electrospun nanofibers of calcium cobaltite $\text{Ca}_3\text{Co}_4\text{O}_{9+x}$, known to be an auspicious p-type TE material owning good functional properties and thermal stability in air, enabling its utilization in high-temperature applications. Polycrystalline $\text{Ca}_3\text{Co}_4\text{O}_{9+x}$ nanofibers and nanoribbons were fabricated by sol-gel electrospinning and calcination at intermediate temperatures to maintain small primary particle sizes. Bulk ceramics were formed by sintering pressed compacts of calcined nanofibers during TE measurements. The bulk nanofiber sample pre-calcined at 973 K exhibited an improved Seebeck coefficient of $176.5 \text{ S}\cdot\text{cm}^{-1}$ and a power factor of $2.47 \text{ W}\cdot\text{cm}^{-1}\text{K}^2$ similar to an electrospun nanofiber-derived ceramic compacted by spark plasma sintering.

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Laser-based Powder Bed Melting of Oxide Ceramic Powders by Ultra-short Pulsed Laser Radiation

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The application of additive technologies for the production of ceramic components is currently being investigated and developed internationally. Laser-based powder bed melting is partially established industrially for ceramic materials. Nevertheless, there are disadvantages with regard to the density of the components and the necessary post-processing steps.

By means of ultra-short pulsed laser radiation, a new process technology is being developed. The aim is to realise near-net-shape ceramic components with a density of > 90 %. The ultra-short pulsed laser radiation enables a significantly smaller interaction zone with the powder material. This results in a well-defined melting zone and, in turn, an increase in the structural resolution of the component. Furthermore, the development of a ceramic powder with a bimodal powder particle size distribution increases the powder bed density and thus, the resulting component density. In order to develop this process technology, specific modifications of the experimental setup become necessary. For this purpose, a preheating mechanism is integrated in order to increase the temperature range during the manufacturing process. Additionally, the development of thermally induced stresses in the component is prevented. Furthermore, the generated bimodal ceramic powders are quantified with regard to the material-specific flow, transport properties as well as the process suitability. Based on these results, laser beam-material interactions are investigated on single layers in order to determine the correlations between these interactions, achievable material properties as well as the resulting component quality.

Chemical Vapor Deposition of Phase-Pure Uranium Dioxide Thin Films from Uranium(IV) Amidate Precursors

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Homoleptic uranium(IV) amidate complexes have been synthesized and applied as single source molecular precursors for the chemical vapor deposition of UO_2 thin films. These precursors decompose by alkene elimination to give highly crystalline phase-pure UO_2 films with an unusual branched heterostructure.

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Improving the performance of Transverse Multilayer Thermoelectric Generators using the concept of Babin plots

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Transverse MultiLayer ThermoElectric Generators (TMLTEG) are ceramic-metal composite devices that convert heat directly into electrical energy with potential applications in the field of waste heat recovery and energy harvesting for low power applications. TMLTEGs are fabricated from low-cost, non-toxic and environmentally friendly oxide ceramic layers and screen-printed metal stripes. Manufacturing TMLTEGs using the standard ceramic multilayer technology can enable competitive device pricing with an expanded range of operation compared to commercially available thermoelectric generators.

Using analytical calculations of the anisotropic thermoelectric properties, we present the concept of Babin-plots that describe the power output and thermoelectric energy conversion efficiency of TMLTEGs as a function of device geometry and material properties. Optimal parameters were defined for different thermoelectric oxide ceramics, taking into account device fabrication restrictions and working conditions in possible application scenarios.

The Ball-on-Three-Balls-test: Improving accuracy while simplifying stress evaluation

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The Ball-on-Three-Balls-test has proven to be an accurate and easy-to-use option for strength testing. However, the stress distribution must conveniently be calculated based on Finite-Element-Analysis (FEA). For the maximum stress, a fitted function was provided by Börger et al. in 2002 based on these results. This function is based on results which were generated under the assumption of punctiform load introductions, a condition that is valid for many specimens in practice. Deviations from these ideal conditions occur through large specimen deformations, plastic deformation of the balls, friction, or an increase in contact-area between the balls and the specimen.

These non-linear effects are investigated by FEA for a wide range of specimen geometries and materials. It is shown that the maximum stress of both exceptionally thin and thick (relative to the support-radius) specimens is sensitive to the area of contact between the loading ball and the specimen. Furthermore, thin specimens are subject to large deformations, which significantly decrease the maximum stress.

Following a new stress analysis, a shorter fitted function is presented. For specimens with exceptional geometries, non-linear effects are considered with adjustment factors added to the new fit function.

Therefore, an easy-to-use stress determination is provided for most practical specimens while also allowing stress determination for exceptionally thin or thick specimens with increased accuracy.

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Development of Zn₂TiO₄ coatings by suspension spraying of binary ZnO-TiO₂ suspensions

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Due to their outstanding properties regarding photocatalytic activity, thermally sprayed Zn₂TiO₄ coatings are of interest for various industrial applications. The expensive price of the Zn₂TiO₄ feedstock material prevents the coating from bringing it into the market. Thermal spraying of tailor-made binary suspensions is an appropriate way to get Zn₂TiO₄ coatings with both, excellent properties, and reasonable prices. Instead of using a costly blended oxide powder, two stable water-based suspensions containing ZnO and TiO₂ particles with low sedimentation rates were prepared. A binary suspension is mixed and used in a SHVOF-spraying process, in which dense coatings were obtained. The coatings composition is analyzed via XRD and XRF measurements. UV-Vis measurements and comparable tests of Rhodamin B degradation via irradiation with ultraviolet and visible light are performed. The electrical and microstructural properties of the coatings are investigated. The high potential to use appropriate binary suspension feedstock from ZnO and TiO₂ to obtain dense Zn₂TiO₄-coatings with desired features is presented.

Tribological characterization of UHTCMCs for brake applications

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The main requirements for brake materials are a stable coefficient of friction, a low wear rate, a low life cycle cost and a low weight. Depending on the application, brakes can be made of steel, C/C or C/C-SiC. Steel possesses a relatively high and stable coefficient of friction (COF) and a low wear rate. However, powerful vehicles require more power for braking. For these applications, ceramic matrix composites, such as C/C and C/C-SiC composites, are preferred for their higher damage tolerance and high temperature resistance. C/C composites possess low oxidation resistance, high wear rate and low COF at temperatures below 200°C. C/C-SiC composites have high COF and thermal shock resistance, but high wear rate, decreased fatigue resistance and unstable COF. Thanks to their properties, such as high oxidation resistance and good high temperature strength, fibre reinforced ultra-high temperature ceramic matrix composites (UHTCMCs) are a promising class of material for braking application. The aim of this work is to investigate the tribological behaviour of these novel materials. In this study, UHTCMC materials constituting the braking pads were tested against C/C, C/C-SiC and Steel discs. Two different brake pressures (1-3 MPa) and two kinds of reinforcement were tested: a long pitch fibre fabric and a chopped pitch fibre reinforcement respectively. Also, recordings of COF and temperature trends were shown. SEM analysis was carried out in order to investigate the friction layer formation. Preliminary results showed a quite stable COF for the tests with the Steel and C/C-SiC disc with a value of 0.5-0.4 and a low COF for the C/C disc with a value of 0.3.

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Physico-chemical characterization and cytotoxicity of PDA nanoparticles doped tricalcium silicate cements

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Tricalcium silicate (TCS) is a widely used calcium silicate cement for many different endodontic treatment procedures such as root end filling, root canal filling, repair of root perforations and vital pulp therapy because of its appropriate physicochemical and favourable biological characteristics. The long setting time and insufficient strength of tricalcium silicate (TCS)-based cements are a main disadvantage for their clinical use. Therefore, the aim of this study was to develop an advanced TCS cement by incorporating polydopamine (PDA) nanoparticles and to evaluate their setting time and compressive strength. The PDA nanospheres ($d_{50} = 135$ nm) were synthesized by the self-oxidative polymerization of dopamine (DA) in an alkaline solution at room temperature. The results show that the initial and final setting time of the cements were reduced by 63.9% and 46.7%, respectively, by adding 4 wt% PDA nanoparticles. The compressive strength of the cements reached 39.6 MPa by the addition of 4 wt% PDA and was thus 37.5% higher than that of pure TCS. Furthermore, the good biocompatibility of cements containing 4 wt% PDA was readily confirmed by using a fibroblast cell line. Our results showed that a TCS cement reinforced with PDA nanoparticles could be a promising dental restorative material.

Oxide ceramic substrates for laser-induced direct metallization

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Laser-induced direct metallization (LDM) is a selective chemical metallization process capable to apply 10-15 µm thick metallic structures on 3D surfaces. It can be used to apply conducting paths, antennas or sensoric structures on complex shaped structural parts in order to integrate electronic functionalities. In contrast to other plating technologies for ceramic substrates both the effectivity of the LDM process as well as the properties of the metallic structures such as adhesion strength and roughness strongly depend on the complex interaction between a pulsed laser beam and the substrate material during the selective laser activation step prior to chemical metallization. A variety of oxide ceramic substrate materials based on alumina in combination with two different laser systems were investigated. This poster gives an insight into the LDM metallization process and the influencing factors on the complex laser-material interactions during activation. Furthermore, an overview of combinations of material compositions and lasers investigated to date and their influence on metallization properties is given. A demonstrator part with 3D conducting paths and surface mounted LED is presented showing the prospects for the manufacturing of functionalized ceramic parts.

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Two component ceramic injection molding (2C-CIM) of oxide ceramics

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While 2C-injection molding has become a standard procedure in processing of plastics, the process is not very well documented in processing of ceramics. In ceramics the possibility to join different ceramic materials opens new perspectives to combine different ceramic materials in one component and exploit specific advantages (structural and functional) of the individual materials. The main difficulty in 2C-CIM is to join two dissimilar materials during the molding process and prevent distortion and separation of the joined part during sintering. 2C-CIM components combining alumina, ZTA and ZTA-TiN were carried out on a standard injection moulding machine with one injection unit employing the insertion technology. The first subcomponent was molded and inserted into a second mold in which the second subcomponent was joined with the first during the second molding process. The dwell time of the inserted part in the mold and thereby the equilibration of the temperature are crucial to avoid distortion of the 2C-samples during the molding process. Dilatometry results show that the individual ceramic materials were successfully modified with sintering additives in order to harmonize the shrinkage during sintering. SEM and microscopy of the interface prove the bonding of the two components. Non-destructive testing by computer tomography was employed to monitor the behavior of the 2C-CIM components along the process chain.

Novel mineral-organic bone adhesive compositions and adhesive strength evaluation through hydroxyapatite cement based biomimetic in-vitro test system

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Despite decades of research, no satisfying bone adhesive material has been developed yet. Recent papers have shown that certain magnesium phosphate-based cement compositions can serve as bone adhesives. Extensive research on the matter requires a reliable test setup to evaluate the performance of potential material compositions. Commonly, samples from animal bones are used for adhesive tests. Bone samples vary depending on their origin and must be mechanically processed into test specimens at great expense, which severely limits the number of tests. A further limitation is the inability to perform long-term performance tests, as specimens begin to decompose during storage. So far, there are no standards regarding processing and testing. Here, a completely synthetic approach is used to perform a high-throughput pre-study and the possibility to evaluate long-term stability, before finalising results by testing on natural bone as well. Calcium phosphate cement is utilized to produce samples composed of hydroxyapatite with constant properties. Both approaches complement each other and allow effective bone adhesive research. Adhesive strengths of different mineral adhesives could be evaluated using a test system based on synthetic hydroxyapatite. Although no direct prediction of specific adhesive strength on natural bone can be made from this, trends can be predicted and suited candidates can be further evaluated on bone. This way, new adhesive compositions including magnesium oxide of various reactivity, amorphous trimagnesium phosphate produced by heat-treatment of magnesium phosphate hydrate as well as phosphoserine were shown to provide adhesive strengths up to 10 MPa on bone.

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Fabrication and characterization of rubidium-doped mesoporous bioactive glass/gelatin coating on bioactive glass scaffold for bone tissue engineering

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Porous three-dimensional bioactive glass (BG) scaffolds were prepared using foam replication. The BG scaffolds with higher porosity and interconnected pore structure have been used extensively in bone tissue engineering (BTE). The unique interconnected structure of scaffolds is beneficial for bone cell growth and nutrient transport. However, highly porous scaffolds lack mechanical properties. The challenge of BTE scaffold is a balance between biocompatibility and mechanical properties. In the case of porous BG scaffold, incorporation of mesoporous bioactive glasses (MBGNs) and gelatin-coating enhances compressive strength, improves cell-scaffold interaction, and promotes apatite-forming ability in the SBF of scaffolds. Moreover, incorporation of boron (B), zinc (Zn), and rubidium (Rb) ions in BG composition leads to increasing angiogenesis, osteogenesis, and antibacterial activity of BG. Hence, this work focuses on coating BG scaffold by gelatin incorporated with Rb-doped MBGNs (Rb-MBGNs). The coating aims to improve compressive strength and enhance bioactivities. Comparatively to uncoated BG scaffold, Rb-MBGN/gelatin coating on the BG scaffold increased compressive strength. Effects of incorporating Rb-MBGN in gelatin coating were found to improve hydroxyapatite formation in SBF and alkaline phosphatase activity (ALP). In addition, vascular endothelial growth factor (VEGF) release was promoted by adding Rb-MBGN to gelatin coating. Rb-MBGN/gelatin coated BG scaffolds exhibited appropriate compressive strength, HA formation, osteogenic and angiogenic potential and are, therefore, potential for bone tissue engineering.

Effects of hydrothermal aging in Y-TZP multilayers zirconia used for dental applications

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The use of yttria-stabilized zirconia-based ceramics (Y-TZP) is widespread in the field of dentistry, due to its features of biocompatibility, high aesthetic combined with excellent mechanical properties. Recently, it was launched in the market a new propose of zirconia CAD/CAM blocks that aims to combine the high fracture toughness of 3Y-TZP with the elevated translucency of 5Y-TZP. In the multilayer configuration, each layer contains a different content of Y_2O_3 stabilizer causing the mechanical and optical properties to change gradually due to the different amount of cubic and tetragonal phase. As it is a new configuration of material, it is necessary to carry out studies of the interface in order to determine the mechanical behavior and the failure mechanism. Thus, this work performed biaxial flexural test after low temperature degradation in 134 °C for 20 h at 2.2 bar. The bilayer zirconia samples were produced with commercial powders of 3Y-TZP and 5Y-TZP. In this way, we demonstrate if the microstructural degradation negatively affected the mechanical properties and the translucency of material. The 12 mm diameter samples were obtained by uniaxial compaction followed by sintering at 1450 °C. Then, biaxial test is performed using the piston on three balls technique and microscopy investigation are accomplished to investigate the crack propagation mode and the microstructure of the material.

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Microstructure effects on the magnetic permeability spectra in (non-) stoichiometric Ni-Cu-Zn ferrites

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Ni-Cu-Zn ferrites are used as magnetic materials for multilayer inductors. These passive devices are fabricated using the multilayer technology based on ferrite tape casting, printing of coil structures using Ag paste, stacking and cofiring at 900°C. In order to better understand the fundamental effects and to tailor the properties of these soft ferrite materials, intense investigations of correlations between chemical composition, processing, microstructure and magnetic parameters are necessary.

In this study we focus on Ni-Cu-Zn ferrites with or without a small iron-deficiency. The microstructure of the ferrites, i.e. density, grain size, phase composition, element distribution, is investigated as function of sintering temperature and dwell time. Permeability spectra of the samples were measured between 1 MHz and 1 GHz. Correlations between microstructure and permeability were studied based on the nonmagnetic grain boundary model (NMGB). The influence of grain size and non-magnetic secondary phases, e.g. porosity, or segregated grain boundary phases, generated by variations of the chemical composition and sintering process on the magnetic permeability is revealed and discussed in terms of the NMGB model.

A Long Time Heat Storage System with Zeolite Granulates

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Hydrophilic zeolites are known for high water adsorption capacity combined with a high enthalpy of adsorption. Therefore, zeolites are commercially used for short term heat storage and transferring of heat. However, long term heat storage for using solar energy or waste energy of industrial processes is still not available on the market.

Binderfree zeolite NaY beads were analyzed by drying in a heating chamber at different temperatures up to 300 °C in a closed heat storage system. By adsorption of water up to saturated water content (adsorption capacity up to 30%) temperatures up to 80 °C were detected over a longer period of time.

Based on these results a prototype zeolite heat storage system with a thermal capacity of 150 kWh was constructed and built. The narrow place between the heat exchanger plates of the storage was filled with 900L of NaYBF zeolite beads. Inside the zeolite structure the thermo-chemically bonded energy can be saved over a long period of time optimally at 200°C under 50 mbar. The energy can be generated in summer at thermal solar collectors or by electric heating with solar cells or waste energy of industrial processes. The goal of the project was also to find the optimal conditions for controlled energy rate at constant temperature by varying the wetting systems.

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Membrane Reactor - A Promising Candidate for Green Chemistry

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The so-called membrane reactors can increase the yield of chemical reactions by enhancement of the selectivity due to selective dosing of reactants or by increasing the conversion by shifting the chemical equilibrium due to selective removal of reaction products.

The reactor design is dependent on the reaction conditions, the applied membrane type, its role and location in the reactor, the catalyst, and the reaction itself. Also, a combination of membranes in one unit can be considered to enhance the effectivity of the application even further.

The key role is the interaction of the catalyst with the membrane. They must be matched to get optimal operating conditions and maximum yields. Key is that none of both should outperform the other but must be optimized with high activity and stability. Fraunhofer IKTS focusses on applied research in this field and together with its partners IKTS combines the expertise of catalysis activities with the expertise of membrane activities to develop highly efficient processes with fully optimized reactor designs and membrane/catalyst systems. The aim is the realization of sustainable chemistry and the industrial commercialization of membrane reactor applications. For this instance, the sustainable synthesis of methane and methanol, the reforming of methanol, and partial oxidation of hydrocarbons to syngas have been intensively studied so far. Other important reactions Fraunhofer IKTS is planning to deal with are for example the methanol-to-olefins reaction, the synthesis and cracking of ammonia, de-/hydrogenation reactions, the Fischer-Tropsch-Synthesis, hydroformylations and esterifications.

Reactive-field-assisted-sintering of non-stoichiometric yttrium-aluminum-garnets and their behavior in etching processes

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Yttrium-aluminum garnets (YAG) are promising structural materials for application in plasma etching chambers. When processing this material via reactive sintering starting from Al_2O_3 and Y_2O_3 , the formation of secondary phases is difficult to avoid. The formation of secondary phases and their influence on etching performance are still barely investigated.

In the present work, Al_2O_3 and Y_2O_3 starting powders were mixed in different molar ratios and used for the preparation of yttria-alumina ceramics via FAST/SPS at a temperature of 1600 °C. In addition to the stoichiometric composition of YAG ($\text{Y}_3\text{Al}_5\text{O}_{12}$), samples with a higher amount of either Y_2O_3 or Al_2O_3 were produced. To study the plasma interaction of the material, the samples were exposed to a reactive etching plasma. Besides investigating mechanical properties, the surface morphology, phase-volume content, chemical composition before and after plasma etching, and the influence of possible secondary phases on the erosion mechanism were analyzed.

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Piezo-phototronic Ultraviolet Photodetectors based on ZnO bicrystal interfaces

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Ultraviolet (UV) photodetectors have captured a great deal of attention in recent years due to their applications in various engineering fields. In all of these practical applications, UV detectors need to possess a high signal-to-noise ratio, fast response and recovery time, high selectivity and stability, and acceptable responsivity (gain of a detector). As a direct wide-bandgap semiconductor, ZnO ($E_g = 3.3$ eV) has demonstrated promising properties for UV detection with a relatively high UV-to-visible rejection ratio. Nevertheless, a major challenge that prevents producing high-quality ZnO-based photodetectors is their high dark current under externally applied bias. In the present research, we designed and examined a novel photo-device system consisting of two ZnO single crystals and a thin ZnO varistor ceramic layer. ZnO bicrystals, with various crystallographic configurations i.e. O1O, O1Zn, and Zn1Zn, were prepared using a diffusion bonding technique followed by a high-temperature epitaxial solid-state transformation step, resulting in a single boundary between two crystals. Conductivity evaluations illustrated all the prepared bicrystal systems featured varistor-like behavior, proving the formation of a double Schottky barrier at the grain boundary with a height of 0.9 eV and approximate breakdown voltage of 3.7 V. Moreover, it was shown that the I-V characteristics of the samples are highly stress-dependent, which had a great influence on the dark current amplitude and subsequently on the UV photodetection performance of the modeled systems. The origin of the photo responsivity change with mechanical load and crystal orientation is explained according to the modification of the formed double Schottky barrier.

Titanium Oxide: combination of electrical conductor and insulator

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Titanium oxide is characterized by its crystal structure in a special variability in the connection with oxygen. Even a small oxygen deficit or minimal doping is enough to turn a good insulator ceramic into a conductive ceramic. Depending on the reduction conditions, electrical resistances can be set in the range between 10^{-3} ?cm and 10^{+3} ?cm .

The elements of the 5th subgroup are of particular interest as doping elements since they have ionic radii like titanium. To adjust the lowest possible specific electrical resistance via doping, doping with niobium or niobium oxide has proven to be advantageous. A partial substitution of Ti^{4+} by Nb^{5+} takes place in the titanium oxide crystal lattice, which increases the number of charge carriers and thus causes electrical conductivity. Depending on the sintering atmosphere and amount of doping, the specific electrical resistance of these doped titanium oxides is in the range between $10^{-2} \text{ ?cm} \dots 10^{+2} \text{ ?cm}$.

Material composites with a conductive and an insulating area can be produced by co-sintering pure titanium oxide with doped titanium oxide. These composites are interesting for a wide variety of technical applications, for example as electrode material.

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High temperature dielectrics for temperature-stable multilayer capacitors

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Application of electronic systems in harsh environments including operation temperatures well above 200°C is challenging due to lack of commercially available passive components. To address this problem, the development of new dielectric materials for capacitors has gained considerable attention recently. This work focuses on the synthesis and optimization of complex perovskite $0.45\text{Ba}_{0.8}\text{Ca}_{0.2}\text{TiO}_3$ - $(0.55-x)\text{Bi}(\text{Mg}_{0.5}\text{Ti}_{0.5})\text{O}_3$ - $x\text{NaNbO}_3$ and $(1-x)(0.94\text{Bi}_{0.5}\text{Na}_{0.5}\text{TiO}_3$ - $0.06\text{BaTiO}_3)$ - $x\text{CaZrO}_3$ solid solutions. These materials exhibit beneficial functional properties, e.g. temperature-stable permittivity paired with low dielectric losses (22 %) over a wide temperature range. Additionally, enhanced high-temperature performance was achieved using dopants. Promising compositions were selected to manufacture multilayer ceramic capacitors (MLCCs) with various precious metal electrodes, which display a weak temperature dependence of capacity (10 %) between -80°C and 300°C. Addition of sintering aids is effective to reduce the sintering temperature from around 1150°C to as low as 900°C enabling the use of silver paste for electrodes during cofiring. It is demonstrated, that low-temperature cofiring allows fabrication of multilayer capacitors with temperature-stable dielectric response and low losses.

Influence of surface roughness and electrode design on dielectric breakdown strength of alumina

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The development of ceramic insulators for electronic applications depends significantly on an understanding of the influences of the measurement process and the microstructure. The surface, in particular, has a large number of potential failure points due to defects. While pores and the measurement process have been discussed in some detail, surface defects have not been sufficiently considered. It has been suggested that surface defects may act as initiators of dielectric breakdown (DBS) in a similar way to mechanical failure, thereby significantly reducing the risk of failure in alumina ceramics.

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Thermoelectric performance and high-field behavior of Pb-free PTCRs

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Positive Temperature Coefficient Resistors (PTCRs) are used as passive elements in many applications such as heaters, inrush current limiters and motorstarters. To meet the requirements for this variety of different applications, compositional modifications are necessary to yield the desired electric properties, like resistivity, Curie temperature or high-temperature behavior. Furthermore, reliable PTCR ceramics must withstand high voltages and maintain their properties even after high-field and temperature cycling. Common PTCR ceramics consists of BaTiO₃. Various A-site substitutes (replacing Ba) are added to achieve the desired properties. Pb was demonstrated to fulfill the mentioned electric conditions best. However, according to the RoHS directive enacted by the European Union, Pb is considered as a harmful substance which makes the development and investigation of Pb-free alternatives necessary.

This study focuses on investigations of the BaTiO₃ system and the tuneability of the electrical properties via acceptor and donor doping (e.g., Ca, Sr). The microstructure is investigated in detail using electron microscopy and X-ray diffraction and correlated to electrical parameters. The gained structure-property relationship serves as crucial input for the development of novel Pb-free protection devices.

Deviation from stoichiometry and point defects in substituted Mn-Zn ferrites from thermogravimetry measurements

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Mn-Zn ferrite based soft magnetic materials are used for high frequency applications, i.e. power ferrites with low losses, or high-permeability ferrites. Their basic composition $(\text{Zn}_x\text{Mn}_{1-x})_{1-y}\text{Fe}_{2+y}\text{O}_4$ is optimized for zero magneto-crystalline anisotropy and magnetostriction. These ferrites usually contain more than 50 mol% Fe_2O_3 , with the excess iron creating a significant concentration of ferrous ions Fe^{2+} . Due to the complex phase diagram the fabrication of Mn-Zn ferrites requires an oxygen partial pressure controlled sintering procedure to avoid decomposition of the ferrite during cooling. Substitutions of cations (Co and Ti) modify the magnetic properties, e.g. the temperature-dependent power loss density, but also affect the ageing stability at elevated temperatures.

To further investigate the impact on point defects of the substitutions, the deviation from stoichiometry, δ (as in $\text{Me}_{3-\delta}\text{O}_4$), was measured thermogravimetrically at 1100 °C as a function of the oxygen partial pressure, p_{O_2} for Co-, and Co-Ti-substituted Mn-Zn ferrites. Point defect thermodynamic modeling of the oxygen activity dependence of δ suggests that cation vacancies are the majority ionic defects at high oxygen partial pressure and that cation interstitials dominate at more reducing conditions.

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Controlling the LLZO ceramic composition for high-quality thin solid-state batteries separators obtained by tape casting

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Ceramic solid-state batteries are considered future energy storage systems enabling higher energy density and high safety. $\text{Li}_7\text{La}_3\text{Zr}_2\text{O}_{12}$ (LLZO) garnet is a potential candidate for such batteries due to its stability to lithium metal anodes and high ionic conductivity. Developing industrially scalable and economically feasible manufacturing techniques is crucial to making LLZO-based solid-state batteries a viable alternative to liquid electrolyte lithium-ion batteries. Among the various options, tape casting is a promising method for manufacturing ceramic cell components with adjustable thickness and size. Successful implementation of this technique for LLZO-based batteries requires understanding the chemical transformations along the entire manufacturing chain. In particular, the change in the chemical composition of the LLZO surface due to its protonation in air and protic solvents, as well as the Li evaporation during sintering, critically affect the final component properties such as relative density, flatness, mechanical stability, and ionic conductivity. Hence, this should be understood to achieve full control of the process reproducibility. We present a route to fabricate high-quality thin LLZO ceramic separators using tape casting. We systematically investigate the effects of material composition and process conditions during the various steps of tape casting on the properties of the resulting separators. X-ray diffraction, Raman spectroscopy, scanning electron microscopy, and 3D white light topography confirmed the successful fabrication of flat, dense, single-phase LLZO ceramic separators successfully used in full-cell tests.

Microscale Modelling of CVI SiC/SiC Composites

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Ceramic Matrix Composites (CMCs) are advanced materials with exceptional properties such as high temperature resistance, low thermal expansion, high strength and stiffness, and excellent wear resistance. However, their processing and manufacturing methods is challenging and complex, thus making it difficult to achieve consistent and reproducible properties. To ease this process, modelling and simulation is utilised.

Modelling and simulation provide a powerful tool for predicting the behaviour of CMCs under various loading conditions and for optimizing their design. By using computational models, the response of CMCs to various environmental conditions, such as temperature, pressure, and humidity can be determined, and it becomes possible to predict the effect of different processing parameters on their microstructure and to investigate their properties. This allows for an optimization of the design and processing of CMCs to achieve desired properties and improve their overall performance. Thus, time and cost for research and development are reduced.

The present analysis addresses this issue for the in modelling of the microscale geometry of this special CVI SiC/SiC using micrographic digital imaging and Matlab.

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Approaches for Modelling and Simulation of Ceramic Matrix Composites

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The machining of brittle materials ranging from ceramics and glasses to composites such as ceramic matrix composites is already an established part of the process chain in branches of industry such as aerospace, automotive and medical engineering. Compared to ductile materials such as metals, the key challenge in machining brittle materials is the rapid and sometimes seemingly random propagation and expansion of cracks in the material as early as the initial contact between tool and material. This behaviour extends throughout the entire machining process. The associated damage and machining mechanisms are not well understood, especially for complex brittle composites such as CMCs.

Numerical simulations of the machining process offer the potential to study and improve the understanding of the underlying mechanisms. For CMC, the complexity of the anisotropic material structure with brittle-hard materials represents a crucial difficulty. In the context of this work, approaches for modelling and simulating ceramic matrix composites for the machining process will be introduced and the state of the art from different areas of machining such as ceramics and carbon fibre reinforced plastics will be consulted.

Carbon-bonded alumina filters for aluminum melt filtration

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In the present study carbon-bonded alumina filters, have been investigated as a potential filter material for filtration of aluminum. The first time, short- and long-term pilot scale filtration trials were conducted, and the filter behavior during filtration of aluminum alloy was determined by the use of PoDFA (Porous Disk Filtration Apparatus) for the short-term trials and LiMCA (Liquid Metal Cleanliness Analyzer) for the long-term trials with wrought alloy 6xxx aluminum. All applied filters were also investigated post-mortem by SEM (Scanning Electron Microscopy) analysis. Furthermore, sessile drop experiments with capillary purification were performed to evaluate the wetting behavior as well as any reactions occurring between the filter material and the aluminum alloy being filtered.

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Three-dimensional microstructural analysis to characterize failure mechanisms of a novel SOEC

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A novel Ni-GDC cermet supported intermediate temperature electrolysis cell containing a three-layer GDC-YSZ-GDC electrolyte has been developed as promising candidate for hydrogen production. However, initial studies revealed cell failure and crack formation in the electrolyte layer at low operating temperatures and high electrolysis currents.

To study the origins of failure different methods were used for microstructural investigations.

Focused ion beam tomography was performed to generate series of images which allowed for the reconstruction of three-dimensional microstructures of the cathode functional layer and electrolyte. For comparison, X-ray computed tomography was conducted for the non-destructive reconstruction of larger volumes. In combination with the direct assessment of cracks and other defects using laser confocal microscopy, an overview of the different three-dimensional characterization methods and findings will be given.

Ammonia synthesis based on piezo-assisted materials

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Although new innovations for sustainable chemical synthesis have made great development recently, finding technically workable answers to surpass fossil fuels remains difficult. A viable route to a green fuel in this context is electrochemical synthesis of ammonia (NH₃) because of its high energy content (18.8 MJ/kg), cheap storage costs, and zero carbon footprint. Through the Haber-Bosch process, nitrogen fixation has been carried out for more than a century. This process requires high pressure (20 – 40 MPa) and temperatures (400 – 600°C). The industrial NH₃ synthesis requires 3-5% of the global natural gas production and consumes 1% of the global energy, which underlines the need of alternative NH₃ synthesis routes.

Through mechanical vibrations piezoelectric materials effect the electrochemical processes accelerates the transfer of electrons from catalyst to molecular absorbates thereby improving the sluggish nitrogen reduction reaction (NRR) kinetics. The created transient dipoles in surface-absorbed non-polar N₂ molecule increases the N₂ coverage on the electrocatalyst surface. The feasibility of (Na_{0.5}K_{0.5}NbO₃) KNN deposited on stainless steel and on a titanium electrodes via the sol-gel route using alkoxide precursor will be discussed with respect to piezo-enhanced nitrogen fixation. By applying piezo-actuation the ammonia production could be enhanced by more than four times in comparison without any piezo actuation.

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HWH COMPETITION

Additive manufacturing of carbon fiber reinforced green bodies for C/C-SiC ceramic matrix composites

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Liquid silicon infiltration is a cost-effective manufacturing route for C/C-SiC, which is a ceramic matrix composite with a high specific stiffness associated with a quasi-ductile material behavior. Within this process, a carbon fiber reinforced polymer (CFRP) is fabricated using conventional polymer processing methods, pyrolyzed above 1000 °C in inert conditions and infiltrated with liquid silicon at temperatures of at least 1420 °C in vacuum. To reduce the cost-intensive machining while improving the geometric freedom of design, a new additive manufacturing route of C/C-SiC was investigated. The fused filament fabrication (FFF) technology was used to manufacture CFRPs made of thermoplastic polyetheretherketone (PEEK) as the matrix with short carbon fibers as the reinforcement. Additionally, a thermo-oxidative crosslinking step of the CFRP prior to pyrolysis was introduced to prevent their re-melting. According to literature, thermo-oxidative crosslinking of PEEK is dependent on the specific surface area (SSA). Therefore, the SSA of the green bodies was investigated via computer-aided image analysis as a function of the layer height, the infill density, and the printing direction using statistical design of experiments to enhance the stability of the CFRPs during pyrolysis. It was shown that it is possible to adjust the SSA selectively between 8.0 and 11.3 mm²/mg by modifying the mesostructure of the CFRP and thus also the phase composition of the C/C-SiC using the FFF technology.

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Crack length determination in all-oxide Ceramic Matrix Composites

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Ceramics, with their high thermal and chemical stability, are used in high temperature applications. The introduction of ceramic fibers eliminates brittleness and leads to a quasi-ductile behavior of the resulting ceramic matrix composite (CMC), as microscopic failure mechanisms such as microcracking, crack branching, and fiber pull-out result in a damage tolerant material. Compared to non-oxide CMC, an all-oxide CMC has the advantage of not being susceptible to oxidation, making it suitable for high temperatures and aggressive oxidative atmospheres. Since crack propagation in CMC differs from that in metals or monolithic ceramics, existing methods for fracture mechanical characterization based on the estimated crack length need to be adapted. The work investigates the applicability of the standard fracture toughness test ASTM E1820 and focuses on the determination of a reliable crack length, which is required for the fracture toughness calculations. Here, the use of a visual measurement system is explored. Notched bend specimens were cyclically loaded according to the ASTM standard. The crack length was determined from the material compliance as well as visually by strain measurements. The results show the suitability of the visual measurements and an influence of the notch geometry on the crack path. The crack length calculated from the compliance indicates an underestimation of the effective crack length. Therefore, the crack length determination and the considered standards need to be adapted. The achieved results allow a comparison of different approaches to estimate the effective crack length of CMC.

Feedstock Development for FDM/FFF of Al₂O₃ based Ceramic Matrix Composites

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Oxide ceramic matrix composites (O-CMCs) are gaining attention as a serious substitute for metals in several applications. They offer lower density, comparable strength and high oxidation resistance, enabling efficiency improvements originally in the aerospace sector as well as more recently in automotive or commercial aviation. However, the production process is time-consuming and limited to simple geometries. Fused Deposition Modeling (FDM) as a category of Additive Manufacturing (AM) is a promising rapid prototyping technique offering simplified shaping, production of complex shapes, and little waste.

In this project, a printable feedstock was developed starting from commercially available raw materials. In addition to composition, four point bending test of the FDM filaments after sintering (1200 °C), printing parameters, thermal treatment and the strength of printed structures were investigated. After sintering a relative density of 55% could be achieved. By sintering the fiber composite for 5h the mechanical strength could be doubled, in comparison to the pure matrix material. Optimized printing parameters including infill orientation, were evaluated and investigated by optical microscopy. Samples with different infill orientation were sintered and tested using four point bending to evaluate the mechanical properties.

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Closed-Loop Recycling of Solid Oxide Cells

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Fuel Cells and Electrolyzers will play a significant role in the future energy system and industry. Due to their high flexibility and efficiencies, especially using power-heat-coupling, Solid Oxide Cells (SOCs) are showing off to be a promising technology on an industrial scale.

As every material-based technology SOC face the increasingly relevant question of resource efficiency and critical raw material use. This results in the necessity of developing recovery strategies along the whole life cycle – from the raw material procurement to the End-of-Life (EoL) or End-of-Use (EoU) state. Large potential lays also within the recycling of waste material during the cell manufacturing process.

Recovery methods for all major types of SOC are still not developed to scalable strategies however there already exist first concepts for the several types as shown in a recent overview publication by Sarner et al. [1].

Using the example of the fuel-electrode supported SOC, first recycling steps with respect to the green support layer composed of Ni(O) and stabilized zirconia (YSZ), the sintered support layer with the adjacent fuel electrode (Ni(O) / YSZ fine grained) and finally as complete half-cell including the YSZ electrolyte have been characterized on the basis of a master thesis performed in 2022. While direct recycling of untreated substrate green tapes shows off to be possible by re-suspending it with solvent, the recovery of sintered material – substrate and half-cells – comes along with a slight influence on sintering behavior and microstructure, nevertheless seems to be a feasible way, especially using tailored recycle fractions.

Dislocation-based plastic deformation of polycrystalline SrTiO₃ by room-temperature cyclic indentation

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Dislocations in ceramics are drawing increasing attention owing to the great potential in tuning versatile mechanical and functional properties. Contrasting single crystals, polycrystalline ceramics are relatively cheap and easy to fabricate, making them promising candidates for engineering applications. However, besides pores and microcracks, a major challenge for plastic deformation in polycrystalline ceramics is caused by grain boundaries (GBs) which results in dislocation pile-up and cracking at the GBs due to lack of sufficient slip systems in ceramics, particularly for room temperature bulk compression tests. In this study, we adopt a different approach using cyclic indentation to investigate the dislocation behavior of polycrystalline SrTiO₃ at room temperature. A Brinell indenter was used to first plastically deform single crystal SrTiO₃ with surface orientations (001), (011), and (111) to analyze the surface slip trace pattern in these representative surface orientations for benchmarking. Then we applied the method to polycrystalline SrTiO₃ for up to 10 cycles. Dislocation etch-pit examination of the polycrystalline samples suggests that plastic deformation can be initiated at the GB and extends into the grains during cyclic indenting by dislocation multiplication mechanisms. The deformability of individual grains is found to be dependent on the number of indentation cycles. The results show ~60% of all grains within the plastic zone are deformable after a single cycle but cyclic deformation increased the deformability of individual grains to ~90%. These findings will pave the road for further investigations of dislocation-based mechanics and functional studies on polycrystalline SrTiO₃.

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Co – Extrusion of Alumina Core – Shell Structures Fabricated by Robocasting

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Porous ceramics have a wide range of applications due to their exceptional properties, such as adjustable permeability, high surface area, high – temperature stability, thermal shock resistance, and good mechanical stability. In order to enhance the compressive strength of porous alumina, core – shell structures with a dense core and porous shell were fabricated by combining co – extrusion and robocasting. To obtain porous alumina, different amounts of spherical cellulose particles were added to the paste, which were then burned out of the printed green bodies during heat treatment. This leads to a porosity ranging from 18% to 55% in the porous samples, whereas the dense alumina shows a porosity of 1.7%. To investigate the influence of the dense core on the properties of the porous alumina samples two different core – shell ratios were realized. The core – shell samples were characterized in terms of their porosity using the rule of mixture. The compressive strength of the fabricated structures was investigated and compared to the theoretical strength of porous samples without a dense core. The theoretical strength of porous reference samples was calculated using the exponential Spriggs approach. A positive influence of the dense core reinforcement on the mechanical properties was demonstrated. For a porosity of 20%, the core – shell structure show a compressive strength of 850 ± 100 MPa, whereas the porous samples without a dense core show a strength of 706 ± 12 MPa.

Fabrication of thermally and mechanically stable superhydrophobic surfaces via modified hexagonal Boron Nitride Nanomaterials

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Superhydrophobic coatings continue to be of great interest in science and industry due to their wide range of applications. Functional surface coatings that protect a surface from environmental factors such as heat, moisture and rain result in longer surface durability. As an alternative to oxides, hexagonal Boron nitride (hBN) particles can also be used for coating. In a first step hexagonal Boron nitride (hBN) particles were hydroxylated with further functionalization with 1H,1H,2H,2H-perfluorooctyl-triethoxysilane (PES). Glass coatings containing functionalized hexagonal boron nitride (hBN) particles were prepared on soda lime substrates leading to a thermally stable (400 °C) superhydrophobic surface (? 180°). In addition, the commercially available polymer based resins Silikofтал® HTT and Silikophen® P80/X was used as an adhesive system. After several scrubbing cycles of the coating on a sponge, the contact angle never decreased below 130°, showing high mechanical stability. The particles were investigated by x-ray diffraction (XRD), fourier transform infrared (FTIR) spectroscopy and the coatings were examined by scanning electron microscopy (SEM), atomic force microscopy (AFM), scratch test and contact angle measurements.

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