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KERAMIK

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Virtual Conference

Abstracts

96. DKG-Jahrestagung
96th DKG Annual Meeting

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Grußwort der Programmkommission

Liebe Mitglieder der DKG und Freunde der Keramik, Chers amis de la céramique, Beste vrienden van keramiek,

hiermit laden wir Sie herzlich zur KERAMIK 2021, der 96. Jahrestagung der Deutschen Keramischen Gesellschaft (DKG), ein. Die Veranstaltung, die allen Keramikern und Keramikinteressierten offensteht, findet aufgrund der COVID-19 Pandemie virtuell statt.

„Virtueller Gastgeber" der KERAMIK 2021 ist das FZ Jülich. Mit 6.000 Mitarbeitern ist das FZ Jülich eine der großen Forschungseinrichtungen in Europa in den Bereichen Energie, Umwelt, Bioökonomie, Informationstechnologie und Hirnforschung. Hier werden Grundlagen geschaffen für zukünftige Schlüsseltechnologien. Durch interdisziplinäre Forschung entwickelt das FZ Jülich tragfähige Lösungen für komplexe und drängende Fragen der Gesellschaft. Keramische Werkstoffe sind dabei von zentraler Bedeutung und stehen stellvertretend als „hidden champions" für die Technologien der Energiewende.

Die internationale Ausrichtung des FZ Jülich ist durch den jährlichen Besuch von über 1.000 Gastwissenschaftlern aus der ganzen Welt belegt; so wie auch die DKG-Jahrestagung einen zunehmenden internationalen Bezug hat. Wir freuen uns insbesondere, Frankreich als Konferenzpartner der KERAMIK 2021 gewonnen zu haben und begrüssen ebenfalls, da Jülich in der EUREGIO liegt, alle unsere Kolleginnen und Kollegen aus den Nachbarländern Niederlande und Belgien!

Bis bald & À bientôt
Im Namen des Programmausschusses

Greeting by the Program Committee

Dear members and friends of the DKG, dear colleagues, Chers amis de la céramique, Beste vrienden van keramiek,

I would like to cordially invite you to CERAMICS 2021, the 96th Annual Meeting of the Deutsche Keramische Gesellschaft (DKG / German Ceramic Society). This event, which is open to all ceramists and those interested in ceramics, takes place virtually due to the COVID-19 pandemic.

„Virtual host" of KERAMIK 2021 is FZ Jülich. With almost 6.000 employees, FZ Jülich is one of the largest research institutions in Europe in the fields of energy, environment, bioeconomy as well as information technology and brain research. We lay the foundations for future key technologies and work to find viable solutions for complex and pressing societal issues through interdisciplinary research. Ceramic materials are of central importance in Jülich, as „hidden champions" for the energy transition technologies.

The international orientation of the research centre is evidenced by the annual visit of 1,000 guest scientists from all over the world. The DKG Annual Meeting is also becoming increasingly international, and in 2021 we will be paying special attention to activities in France. We look forward in advance to the intensive exchange and future cooperation with our colleagues from Germany's first economic European partner country. As Jülich is located in the EUREGIO, we would also like to warmly welcome our colleagues from the neighbouring countries Netherlands and Belgium.

See you soon & À bientôt
On behalf of the program committee

Prof. Dr.-Ing. Olivier Guillon
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Prof. Fabrice Rossignol promovierte 1995 an der Universität Limoges im Bereich der keramischen Prozesse und Oberflächenbehandlung. Von 1996 bis 1998 arbeitete er als Post-Doktorand an der Agency of Industrial Science and Technology (AIST) in Japan. Danach ging er in die Industrie und war von 1999 bis 2001 als Technischer Manager für die Firma Bosch tätig.

Im Jahr 2002 kehrte er als Mitarbeiter des französischen Nationalen Zentrums für wissenschaftliche Forschung (CNRS) in die Wissenschaft zurück und arbeitete am Forschungsinstitut für Keramik (IRCER) in Limoges, Frankreich. Von 2007 bis 2017 war er hier Teamleiter für keramische Prozesse. Das Team führt integrierte Forschungen durch, von der Pulversynthese bis hin zur Herstellung von Prototypen mit neuen oder verbesserten Eigenschaften. Hierbei werden verschiedene Formgebungs- und Festigungstechniken angewendet (z.B. Additive Herstellung).

Zurzeit ist Dr. Rossignol der stellvertretende Leiter des IRCER. Seine persönlichen Forschungsinteressen sind:
- Die Formgebung nanostrukturierter Keramik (Top-Down und Bottom-Up Ansätze)
- Die Entwicklung additiver Fertigungstechnologie (Tintenstrahldruck)

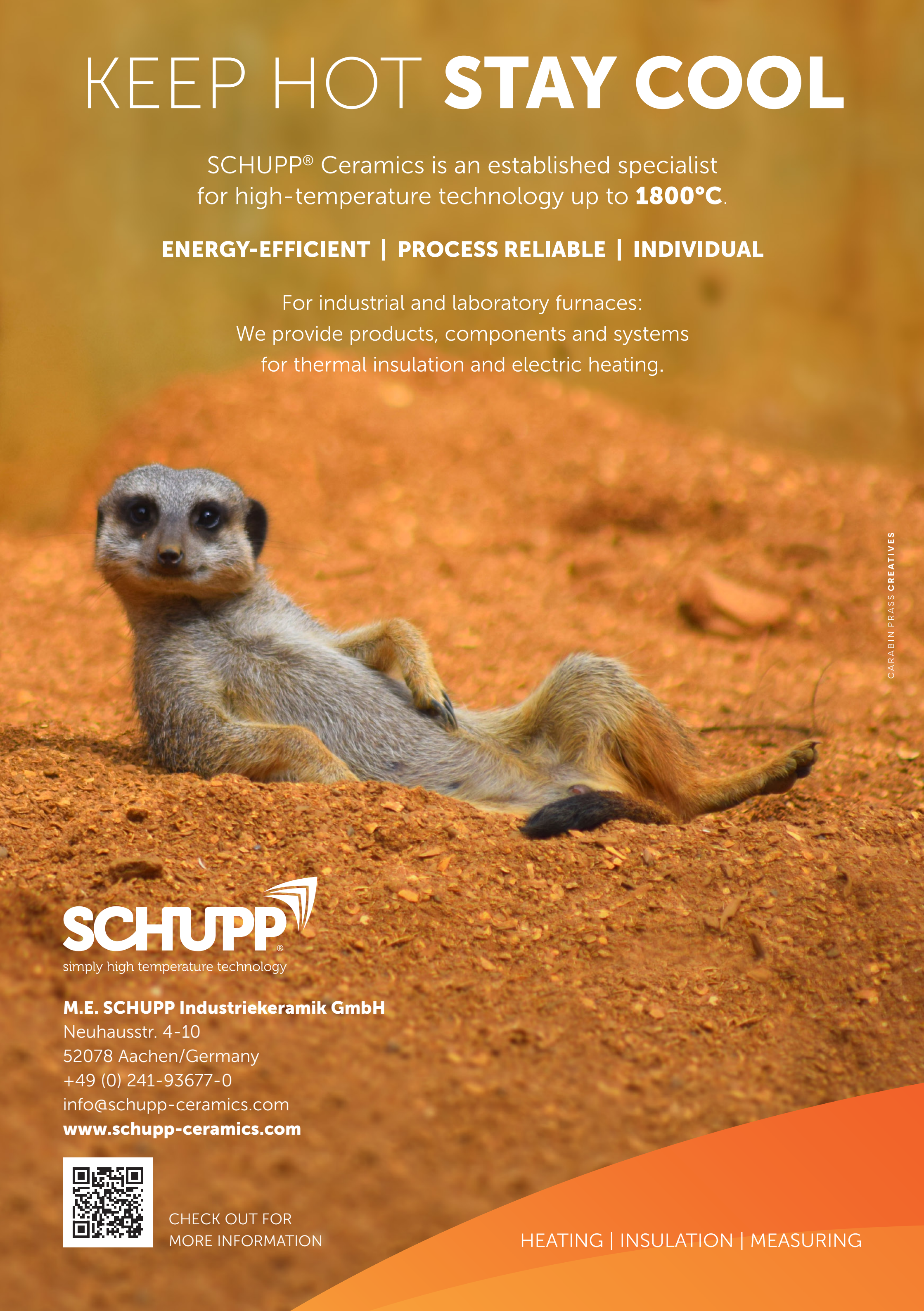
Ein Schwerpunkt seiner Forschung ist der Bereich Energie (z.B. geträgerte Katalysatoren für die Wasserstoff-Produktion).

Prof. Fabrice Rossignol *received his PhD in 1995 at the University of Limoges in the field of Ceramic Processes and Surface Treatments. He used to be a postdoc fellow in the Agency of Industrial Science and Technology in Japan from 1996 to 1998. He then joined the industry as technical manager for the Bosch Company from 1999 to 2001.*

In 2002, he returned to the academic field as a member of the research staff at the French National Research Council (CNRS), working in the Institute of Research for Ceramics (IRCER) in Limoges, France. From 2007 to 2017, he has been team leader of Ceramic Processes at IRCER. His team conducts integrated research ranging from powder synthesis to the fabrication of prototype objects with improved or new properties using various shaping (e.g. additive manufacturing) and consolidation techniques.

Dr. Rossignol is now Deputy Director of IRCER and his personal research interests are:
- *The shaping of nanostructured ceramics (top-down and bottom-up approaches)*
- *The development of additive manufacturing technologies (ink jet printing)*

One key application field of his research is energy (e.g. supported catalysts for H2 production).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Dr. Jens Eichler arbeitet seit 2014 als Senior Specialist im zentralen Forschungslabor der 3M Deutschland GmbH in Neuss. Er leitet dort eine Forschergruppe, die sich speziell mit den verschiedenen Anwendungsmöglichkeiten anorganischer Füllstoffe in polymeren Matrixsystemen befasst. Für seine Forschungsarbeiten wurde er intern mehrfach ausgezeichnet, unter anderem 2018 mit dem Corporate Award „Circle of Technical Excellence & Innovation" (CTE&I). Ein Ergebnis der Forschungsarbeiten sind bisher 13 Patente.

Nach seinem Studium der Materialwissenschaften an der Technischen Universität Darmstadt mit Promotion im Bereich Nichtmetallisch-Anorganische Werkstoffe absolvierte Herr Eichler einen neunmonatigen Aufenthalt als Post-Doc am Indian Institute of Science in Bangalore. In dieser Zeit befassten sich die Forschungsarbeiten mit verschiedenen Aspekten des mechanischen Werkstoffverhaltens von Oxidkeramiken. Mit dem Wechsel zu 3M Technical Ceramics in Kempten (früher ESK Ceramics GmbH) begann eine ca. zehnjährige Phase intensiver Entwicklungsarbeiten im Bereich der Nichtoxidkeramiken mit zahlreichen Veröffentlichungen. Der Wechsel in den Forschungsbereich von 3M eröffnete dann vor fünf Jahren eine neue Perspektive auf die Welt der Keramik in Form von funktionellen Füllstoffen in polymeren Matrixwerkstoffen.

Seit neun Jahren ist Jens Eichler Mitglied des Koordinierungsausschusses Hochleistungskeramik und war zusammen mit Herrn Voigt in den letzten vier Jahren dessen Co-Vorsitzender. Weiterhin hielt er von 2011 bis 2014 an der Hochschule Kempten eine Vorlesung zum Thema „Keramische Herstellverfahren".

*Since 2014, **Dr. Jens Eichler** has been working as a Senior Specialist in the Central Research Lab of 3M Germany GmbH in the city of Neuss. He has been leading a research group that deals with the different applications of anorganic fillings in polymer matrix systems – a highly specialized field. Jens Eichler has received multiple awards for his research work, i.a. the 2018 Corporate Award „Circle of Technical Excellence & Innovation" (CTE&I). As a result of his work, he holds 13 patents.*

After he studied the subject of materials science at the Technical University of Darmstadt and got his PhD in the field of non-metallic anorganic fillers, Dr. Eichler went to Bangalore for nine months and worked as a postdoc at the Indian Institute of Science. During this time, he did research on the different aspects of the mechanical material behavior of oxide ceramics. When entering 3M Technical Ceramics in Kempten (formerly ESK Ceramics GmbH), a ten-year phase of intense development work in the field of non-oxide ceramics started for him, with numerous publications. Five years ago, Dr. Eichler changed to the research department of 3M, which for him opened a new perspective on the world of ceramics in the form of functional fillers in polymer matrix materials.

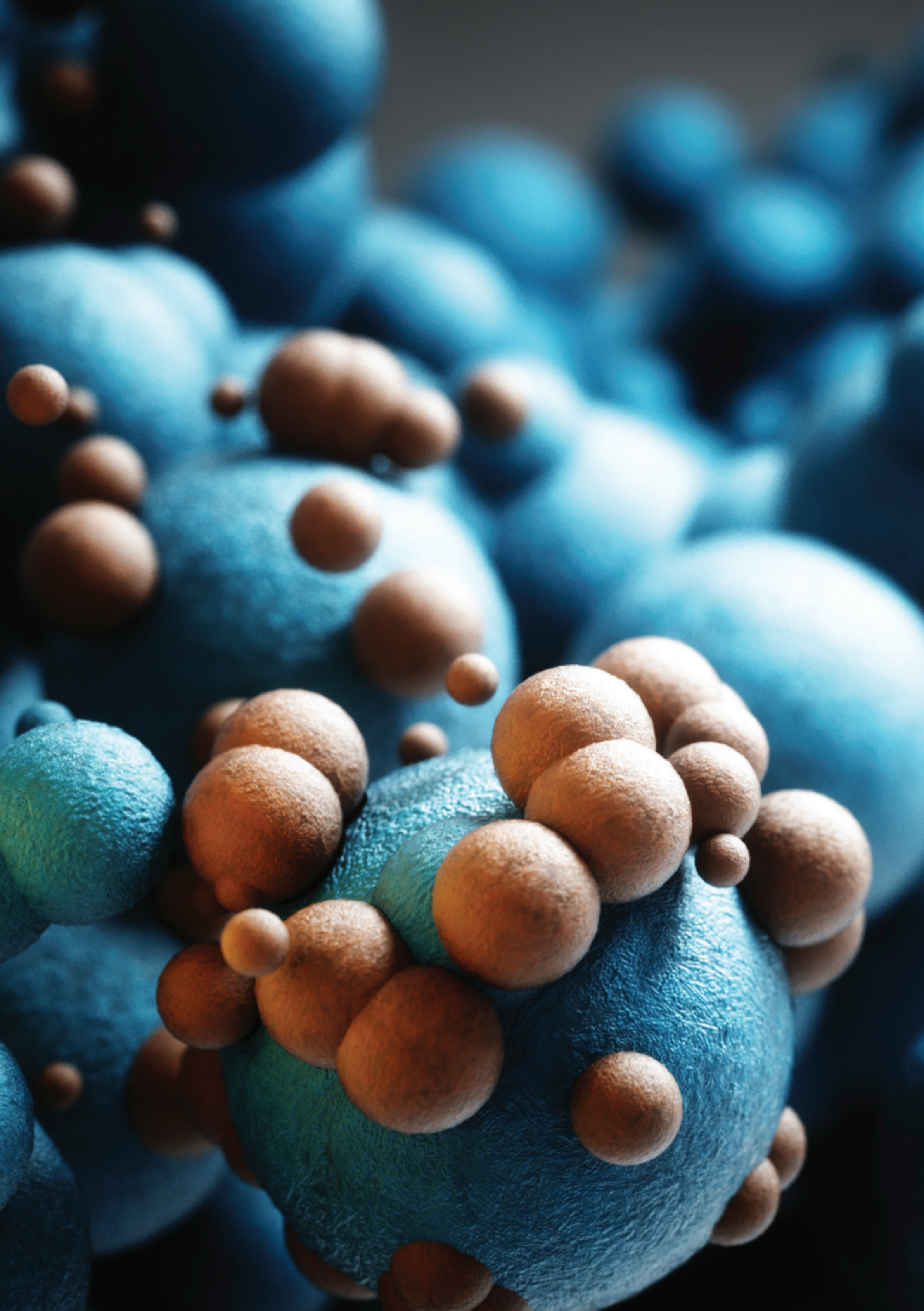
For nine years, Jens Eichler has been a member of the coordinating committee for high performance ceramics and, together with Mr. Voigt, has been its co-chairman for four years now. Further, he held a lecture on the topic of „Ceramic Manufacturing Processes" from 2011 until 2014 at the University of Kempten.

Prof. Dr. Joachim Mayer promovierte in Physik am Max-Planck-Institut für Metallforschung in Stuttgart. Im Jahr 1988 ging er als Postdoktorand an das Materials Department der University of California, Santa Barbara. Ab 1990 war er als Wissenschaftler und Gruppenleiter für den Bereich Analytische Elektronenmikroskopie am Max-Planck-Institut für Metallforschung in Stuttgart beschäftigt. 1999 erhielt er den Ruf an die RWTH Aachen, wo er seither das Gemeinschaftslabor für Elektronenmikroskopie leitet. Im Jahr 2004 wurde er zusätzlich zu einem der beiden Direktoren des Ernst Ruska-Centrums am Forschungszentrum Jülich ernannt. An der RWTH Aachen war er außerdem bis Ende 2019 der Sprecher des Profilbereichs Materials Science and Engineering.

Seine Forschungsaktivitäten beschäftigen sich mit der Anwendung moderner Verfahren der Elektronenmikroskopie zur Lösung materialwissenschaftlicher Fragestellungen in allen wichtigen Materialklassen. Hochauflösende TEM dient dabei zur Aufklärung der Struktur von Grenzflächen und Defekten, während analytische TEM es erlaubt, die chemische Zusammensetzung und die elektronische Struktur mit den funktionalen Eigenschaften zu verbinden. Neue Forschungsaktivitäten beschäftigen sich mit den Struktur-Eigenschaftsbeziehungen in funktionalen Keramiken für die Energie- und Informationstechnologie.

Prof. Dr. Joachim Mayer *received his PhD in physics at the Max-Planck-Institute for Metals Research in Stuttgart, Germany. In 1988, he joined the Materials Department at the University of California, Santa Barbara, as a postdoctoral research associate. In 1990, he moved back to the Max-Planck-Institute where he worked as a research scientist and group leader in the field of analytical electron microscopy. In 1999, he joined RWTH Aachen University to become professor and head of the common laboratory for electron microscopy. In 2004, he received an appointment as co-director of the Ernst Ruska-Centre at FZ Jülich. At RWTH Aachen, he was chairman of the profile area Materials Science and Engineering until the end of 2019.*

His research focuses on the application of modern electron microscopy techniques to solve materials science problems in all major classes of materials. The structure of defects and interfaces is elucidated with high resolution TEM and STEM techniques and analytical TEM makes it possible to link chemical composition and electronic structure to functional properties. Recent research activities aim at understanding structure-property-relationships in functional ceramics for energy and information technology applications.



Böttger-Plakette
Böttger Badge

Für Ihre herausragenden Verdienste um das Zusammenwirken von Industrie, Wissenschaft und Lehre

Die Böttger-Plakette wird seit 1929 an Personen für ihre herausragenden Verdienste um das Zusammenwirken von Industrie, Wissenschaft und Lehre verliehen.

Die Plakette erinnert an Johann Friedrich Böttger (1682 - 1719), der als Naturforscher, Werkstoffwissenschaftler und Alchemist 1707 das Böttger-Steinzeug und im Jahr 1708 gemeinsam mit Tschirnhaus und Ohain das europäische Hartporzellan entwickelte. 1710 wurde die Königlich-Sächsische Porzellanmanufaktur gegründet, deren Leitung der sächsische König August I. Johann Friedrich Böttger übertrug.

For your outstanding achievements in the collaboration between industry, science and teaching

The Böttger Badge has been awarded since 1929 to individuals for their outstanding achievements in the collaboration between industry, science and teaching.

The badge commemorates Johann Friedrich Böttger (1682 - 1719), who as a naturalist, materials scientist and alchemist developed the Böttger Stoneware in 1707 and in 1708, together with Tschirnhaus and Ohain, the European hard-fired porcelain. In 1710, the Royal Saxon Porcelain Factory was founded and the Saxon King August Ist conferred it's management to Johann Friedrich Böttger.

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Prof. Dr. Ingolf Voigt diplomierte auf dem Gebiet der Chemie an der Friedrich-Schiller-Universität Jena und promovierte 1993 in Jena und an der Friedrich-Alexander-Universität Erlangen-Nürnberg. Nach Eintritt in das Hermsdorfer Institut für Technische Keramik (HITK) war er dort als wissenschaftlicher Mitarbeiter, Gruppenleiter für „Keramische Membranen", Bereichsleiter „Keramische Schichten" und abschließend als stellvertretender Institutsleiter tätig. 2010 übernahm Ingolf Voigt die Abteilungsleitung „Umwelttechnik und Bioenergie" des Fraunhofer-Institut für Keramische Technologien und Systeme (IKTS), wo er seit 2013 als stellvertretender Institutsleiter und Standortleiter des IKTS Hermsdorf tätig ist.

Ingolf Voigt wirkt neben seiner stark industrieorientierten, wissenschaftlichen Arbeit am IKTS seit 2010 in der Lehre (Masterstudiengänge der Ernst-Abbe-Hochschule Jena, Friedrich-Schiller-Universität Jena im Bereich Werkstofftechnik und Chemie-Energie-Umwelt) mit, für die er 2018 mit einer Honorarprofessur der Ernst-Abbe-Hochschule Jena geehrt wurde.

Ingolf Voigt ist Autor von mehr als 50 Fachartikeln und mehr als 200 Vorträgen auf nationalen/internationalen Tagungen, sowie Erfinder/Miterfinder von 19 Patenten (Patentfamilien). Im Laufe seiner Berufstätigkeit erhielt Ingolf Voigt mehrere Ehrungen, u. a. den Thüringer Innovationspreis für die Entwicklung keramischer Membranen, den IQ Innovationspreis Mitteldeutschland für Keramische Membranen mit Nanoporen zur energieeffizienten Bioethanolgewinnung, den Innovationspreis der Fördergesellschaft Erneuerbare Energien e.V. (FEE) für nanoporöse keramische Membranen zur Trocknung von Bioethanol, den Thüringer Forschungspreis für die Entwicklung keramischer Membranen zur Sauerstofferzeugung, sowie den Corporate Environmental Achievement Award der American Ceramic Society (ACerS) und den Joseph-von-Fraunhofer-Preis jeweils für die Entwicklung keramischer Membranen zur Nanofiltration und Behandlung von Wasser.

In seiner ehrenamtlichen Tätigkeit in Verbänden und Organisationen leitet Ingolf Voigt u.a. seit 2003 den Arbeitskreis Keramische Membranen und war von 2015 bis 2019 Vorsitzender des Gemeinschaftsausschusses Hochleistungskeramik von DKG und DGM. Seit 2015 ist Ingolf Voigt Vorstandsmitglied der DKG.

Prof. Dr. Ingolf Voigt *graduated in the field of chemistry at the Friedrich-Schiller-Universität Jena and obtained his PhD in 1993 in Jena and at the Friedrich-Alexander-Universität Erlangen-Nürnberg, which made him Dr. rer. nat. After entering the Hermsdorfer Intitute for Technical Ceramics (HITK), he worked as research assistant, group leader for ceramic membranes, division manager in the field of ceramic layers, and finally as deputy director. In 2010, Ingolf Voigt became head of the department of „Environmental Technology and Bioenergy" at the Fraunhofer Institute for Ceramic Technologies and Systems (IKTS), where he has been working as deputy managing director and as site manager.*

Apart from his heavily industry-oriented scientific work at IKTS, Ingolf Voigt has been teaching since 2010, for example the master's degree courses at the Ernst-Abbe-Hochschule Jena as well as Materials Engineering and Chemistry-Energy-Environment at the Friedrich-Schiller-Universität Jena. He was honored with an honorary professorship of the Ernst-Abbe-Universität Jena in 2018.

Ingolf Voigt is author of more than 50 specialist articles and more than 200 lectures held at national and international conferences, as well as inventor of 19 patents (patent families). In the course of his professional activity, Voigt obtained several honors, i.a. the Thuringian Innovation Award for his development of ceramic membranes, the IQ Innovation Award of Central Germany for ceramic membranes with nanopores for energy-efficient production of bioethanol and the Innovation Award of the Fördergesellschaft Erneuerbare Energien e.V. (FEE), a German development association for renewable energies, for nanoporous ceramic membranes for the drying process of bioethanol. Further, the Thuringian Research Award for the development of ceramic membranes for the production of oxygen, the Corporate Environmental Achievement Award of the American Ceramic Society (ACerS) for the development of ceramic nanofiltration membranes for efficient water treatment and the Joseph-von-Fraunhofer-Award for the development of ceramic nanofiltration membranes.

In his honorary work for various associations and organizations, Ingolf Voigt has been managing i.a. the Work Group for Ceramic Membranes and was chairman of the Community Committee for High Performance Ceramics of DKG and DGM. Since 2015, Ingolf Voigt has been a board member of the DKG.

Seger-Plakette
Seger Badge

Für herausragende wissenschaftliche Leistungen

Die DKG verleiht die Seger-Plakette seit 1929. Mit der Plakette werden Persönlichkeiten geehrt, die herausragende wissenschaftliche Leistungen auf dem Gebiet der Keramik erbracht haben.

Bereits der Name der Seger-Plakette birgt ein Stück DKG-Geschichte: Die Auszeichnung geht zurück auf Dr. Hermann August Seger (1839 - 1893), den deutschen Altmeister der Keramik und Erfinder des Segerkegels und des Segerporzellans. Die Schriften Segers brachten der deutschen Keramik weltweit hohes Ansehen.

Dr. Seger propagierte außerdem den Ausbau der Fortbildung und Lehre, damit tradierte Erfahrung allgemein zugänglich gemacht werden konnte. Denn bis zum Ende des Ersten Weltkriegs war es in Deutschland noch üblich, Erfahrungswerte in der Herstellung von keramischen Materialien wie ein Geheimnis zu hüten. Aus diesen Gründen wird Seger als einer der Pioniere der Keramik bezeichnet.

For outstanding scientific achievements

The DKG has been awarding the Seger Badge since 1929. The Badge honors individuals who have shown outstanding academic efforts in the field of ceramics.

The name „Seger Badge" already harbors a piece of history: The award goes back to Dr. Hermann August Seger (1839 - 1893), the German old master of ceramics and the inventor of the „Segerkegel", the pyrometric cone, and the Seger porcelain. Seger's publications brought high reputation to German ceramics worldwide.

Dr. Seger propagated the development of teaching and further training so that traditional knowledge and experience could be made generally accessible. Until the ending of World War I, it was usual practice in Germany to guard experience in the production of ceramic materials like a secret. For these reasons, Seger has been called one of the pioneers of ceramics.

Prof. Dr.-Ing. Walter Krenkel studierte Luft- und Raumfahrttechnik an der Universität Stuttgart und promovierte berufsbegleitend an der dortigen Fakultät für Luft- und Raumfahrttechnik. Von 1985-2004 entwickelte er am Institut für Bauweisen und Konstruktionsforschung des Deutschen Zentrums für Luft- und Raumfahrt (DLR) in Stuttgart neue Werkstoffe und Verfahren zur Herstellung von Leichtbaustrukturen für Thermalschutzsysteme von Raumtransportsystemen, für Turbinenbauteile der Energie- und Antriebstechnik und für neue Hochleistungsbremssysteme. Seit März 2004 ist Prof. Walter Krenkel Inhaber des Lehrstuhls Keramische Werkstoffe der Universität Bayreuth. Von 2006 bis 2012 wirkte er außerdem beim Aufbau des Zentrums für Hochtemperatur-Leichtbau (HTL) der Fraunhofer Gesellschaft in Bayreuth mit.

Hauptforschungsgebiete von Professor Krenkel sind die Entwicklung von hochtemperaturbeständigen Faser-Verbundwerkstoffen mit polymerer und keramischer Matrix, die Prozessentwicklung für neue Hochleistungskeramiken, insbesondere von Faserverbund- und Precursor- Keramiken (CMC und PDC), sowie die Qualifikation von Struktur- und Funktionsbauteilen aus keramischen Werkstoffen.

Professor Krenkel erhielt im Jahre 1996 den Industriekooperationspreis des DLR, 2002 den Karl Heinz Beckurts-Preis für seine wissenschaftlichtechnischen Leistungen auf dem Gebiet der keramischen Werkstoffe und 2010 den Bridge Building Award der American Ceramic Society. Professor Krenkel ist Mitglied der World Academy of Ceramics (WAC), Vorstandsmitglied der DKG und des Ceramic Composites sowie Fellow der Europäischen und Amerikanischen Keramischen Gesellschaft. Er ist Herausgeber von fünf Büchern über Keramiken und Verbundwerkstoffe, Autor und Co-Autor von mehr als 250 Publikationen und hält 40 Patente.

Prof. Dr.-Ing. Walter Krenkel *is professor of ceramics and chair of the Department of Ceramic Materials Engineering at the University of Bayreuth in Germany. He received his diploma degree (Dipl.-Ing.) and PhD (Dr.-Ing.) in aeronautics and aerospace from the University of Stuttgart, Germany. He worked as a research associate at the German Aerospace Center (DLR) in Stuttgart from 1985 to 2004. Walter Krenkel joined the University of Bayreuth as a full professor of ceramics in 2004. From 2006 to 2012 he worked also with Fraunhofer Gesellschaft and established the Center of High Temperature Materials and Design (HTL) in Bayreuth.*

The focus of Walter Krenkel's research are engineering solutions for the development of process routes that display technical and economic advantages over existing techniques for ceramics. Key developments of his research on ceramics and composite materials have been successfully applied in industry.

Walter Krenkel holds 40 patents, is author of more than 250 publications and has edited five books on ceramics. He received several awards like the Karl Heinz Beckurts Award in Germany and the Bridge Building Award from the American Ceramic Society. He is Fellow of the European Ceramic Society (ECerS), Fellow of the American Ceramic Society (ACerS), Academician of the World Academy of Ceramics (WAC) and Member of the Board of the German Ceramic Society (DKG) and of Ceramic Composites (CCeV).

Prof. Dr.-Ing. Jürgen Rödel erhielt 1983 ein Diplom in Werkstoffwissenschaften der Universität Erlangen-Nürnberg, einen PhD der UC Berkeley in 1988 und wurde 1992 an der Technischen Universität Hamburg-Harburg (TUHH) habilitiert. Er war Postdoktorand am National Institute of Standards and Technology (NIST) und der TUHH. Seit 1994 ist er Professor an der TU Darmstadt. Er ist ebenso Professor ehrenhalber der USTB und derzeit Gastprofessor an der Tsinghua-Universität (beide Peking) und dem Tokyo Institute of Technology.

Jürgen Rödel ist Autor oder Koautor von 315 referierten Publikationen mit einem h-Faktor von 63. Im Laufe der Jahre forschte er auf den Gebieten des Sinterns, der mechanischen Eigenschaften, der elektrischen Zuverlässigkeit, der Entwicklung bleifreier Piezokeramiken und der versetzungsdominierten Funktionalität in Oxiden. In Deutschland erhielt er die höchsten Auszeichnungen der DFG für junge (Heinz-Maier-Leibnitz Preis) und erfahrene (Leibniz Preis) Wissenschaftler und ist Mitglied der Deutschen Akademie der Technikwissenschaften (acatech). Ebenso erhielt er die Heyn-Denkmünze der DGM, den Sosman Award der American Ceramic Society und den Ferroelectrics Recognition Award der IEEE.

Prof. Dr.-Ing Jürgen Rödel *received a diploma in Materials Science from Erlangen-Nürnberg University in 1983 and a PhD from UC Berkeley in 1988. He received his habilitation in Materials Engineering from Technische Universität Hamburg-Harburg (TUHH) in 1992. He held postdoc positions at the National Institute of Standards and Technology and at TUHH. Since 1994, Rödel has been working as professor at TU Darmstadt. He is also honorary professor at USTB and currently a distinguished visiting professor at Tsinghua University (both Beijing) as well as specially appointed professor at the Tokyo Institute of Technology, Japan.*

Rödel is author or co-author of 315 refereed publications with an h-factor of 63. Over the years, his research has covered sintering, mechanical properties, electrical reliability, lead-free piezoceramics and dislocation-tuned functionality. In Germany, he received the highest awards of Deutsche Forschungsgemeinschaft (DFG): The award for young scientists (Heintz-Maier-Leibnitz Prize) and the award for senior scientists (Leibniz Prize). Rödel is a member of the National Academy of Science and Technology. He also received the Heyn commemorative medal of Deutsche Gesellschaft für Materialkunde (DGM), the IEEE Ferroelectrics Recognition Award and the Sosman Award of the American Ceramic Society (ACerS).

Lectures

C/C-SiC composites based on a short carbon fiber reinforced plastics containing silicon particles

AHMAD, Husam; STILLER, Jonas; PAESSLER, Erick; NESTLER, Daisy; KROLL, Lother; WAGNER, Guntram

The fiber reinforced ceramic matrix composite C/C-SiC has excellent high temperature properties and due to its low density a very high potential for the use in lightweight structures. The production of carbon fiber reinforced plastic (CFRP) by warm pressing in combination with a subsequent pyrolysis and liquid silicon infiltration is currently the most widespread and cost effective manufacturing process of C/C-SiC composites. The pyrolysis of the CFRP green bodies produces a C/C preform, which is then infiltrated with silicon to produce the SiC ceramic phase, whose amount predominantly determines the properties of the composite material. The injection moulding of the CFRPs offers an interesting alternative for the mass production of green bodies with complex geometry compared to the well-established warm pressing technique. This process can be basically divided into the main steps compounding and shaping. Due to the intensive mixing effect of the compounder, active or passive additives such as Si and SiC can be homogeneously incorporated into the matrix during the compound production. These additives should positively influence the proportion of the ceramic SiC phase and thus the properties of the ceramic matrix composite. In addition, this could enable to omit the external siliconization. In this contribution, the addition of nano silicon to the CFRPs to produce short fiber reinforced C/C-SiC through in-situ C-Si reaction will be presented and discussed

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In vitro bioactivity of silicon oxycarbide-based bioactive glasses modified with Ca and Zn for bone regeneration

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Bioactive glasses have attracted attention in tissue engineering research due to their ability to bond to bone tissue during their dissolution process, besides they present osteoinductive and osteoconductive properties. Incorporation of therapeutic ions is considered an interesting approach to enhance the properties of the glass, for instance, in terms of osteogenic or antibacterial behaviour. Zn is a metallic ion known for having a role in the formation, mineralization, development and maintenance of healthy bones; additionally it has antibacterial properties. Alkali-free bioactive glasses based on silicon oxycarbide (SiOC) have been considered an alternative to melt-derived silicate glasses due to their ability to retain an amorphous structure at high temperatures. In this study, silicon oxycarbide glasses were produced via thermal conversion (pyrolysis) of a polymer-source precursor chemically modified with Ca and Zn. In vitro activity assessment was performed by incubating the samples in simulated body fluid and characterized by means of scanning electron microscopy (SEM), Fourier-transform infrared spectroscopy (FTIR), X-ray diffraction (XRD) and ICP-OES. Additionally, cell viability studies were performed using osteoblast precursor cell line MC3T3-E1 and antibacterial properties were evaluated against S. aureus and E. coli bacteria. The results show the potential bioactive behaviour of SiOC modified with Ca and Zn for application in bone regeneration.

Rare-earth doped SrF_2 as a low fading material for decay time dosimetry

ARNOLD, Michael

A recent approach to measure electron radiation doses in the kGy range is the use of phosphors with an irradiation dose-dependent luminescence decay time. However, applicability of the previously investigated material $NaYF_4$:Yb^{3+},Er^{3+} is limited as it shows pronounced fading. Therefore, in this work a modified SrF_2 synthesis is presented that results in SrF_2 nanoparticles co-doped with Er and Yb that is shown to be a promising dosimeter material. Upon excitation with a 980 nm laser a typical upconversion luminescence is observed. For SrF_2: 2mol% Er, 2mol% Yb a dose dependency of the luminescence decay time is observed. However, of utmost importance is the very low fading effect of the luminescence decay time that is as low as 5% in the 30 days after irradiation and beyond. This makes this material attractive for electron beam dosimetry in spite of lower upconversion intensity compared to $NaYF_4$.

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Treatment and classification of carbon fiber reinforced polymers and their recycling potential in carbon-bonded refractories

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The global demand for carbon fibers for use in carbon composites is increasing rapidly in recent years. Carbon fiber reinforced polymers (CFRP) amount to almost 83% of the entire carbon fiber need in 2017. Therefore, the recycling of carbon fiber composites represents a crucial issue. Most of the recyclable CFRP material arises out of worn-out parts from lightweight automobiles, aircraft, and wind turbines along with production waste.

In order to reuse CFRP waste as carbon fibers in carbon-bonded refractories, several processing steps are required. Hence, the aim of the study is to develop suitable separation and treatment techniques for CFRP fractions and their application into carbon-bonded alumina bricks. Carbon-bonded alumina is often used for functional components for continuous casting of steel. Thus, the high annual production of crude steel offers a tremendous possibility to reuse CFRPs from worn-out parts.

Separation of fibers includes mechanical and thermal treatment as well as comparable classification according to fiber length. Carbon-bonded alumina bricks containing fibers of different lengths and treatments were analyzed regarding their mechanical properties such as modulus of rupture and Young's modulus, both at room and high temperature. Increased high-temperature strength and Young's modulus were achieved with raw CFRPs of particular length. With the addition of long heat-treated carbon fiber bundles, fracture at maximum load might be decelerated. This study provides a straightforward characterization and treatment procedure for reusable CFRPs as well as the potential to recycle treated and raw CFRPs of different lengths in carbon-bonded alumina ceramics.

Formal analysis of UO_2 densification in spark plasma sintering

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Spark plasma sintering (SPS) of uranium dioxide is still a recent achievement. Little information is available on the sintering mechanisms in the open literature. In this work we perform experiments at constant heating rates (CHR) and varying pressure, with the aim to calculate the apparent activation energy for densification, Q_{act}, and the stress exponent, n. In order to avoid the complications given by the variation in the stoichiometry when sintering UO_{2+x} (as typically done in the practice), particular care was given to limit the experiment to nearly stoichiometric $UO_{2.00}$. The proposed method was compared with the master sintering curve analysis (MSC) for the calculation of the apparent activation energy. The obtained values are Q_{act} = 96 kJ/mol for CHR (in a range of theoretical density between 50% and 75%) and Q_{act} = 100 kJ/mol for MSC. The stress exponent results n = 1.4, which is compatible with densification by grain boundary diffusion and grain boundary sliding. It was confirmed that the apparent activation energy for densification in SPS is lower compared to what typically reported for conventional sintering. To the best of our knowledge the literature reports only one study providing Q_{act} for SPS of UO_2 (Q_{act} = 140 kJ/mol) and there are no comparable results for hot pressing (HP). The perspective is a comparison between SPS and HP in order to distinguish the pressure and electric field effects.

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CeraLink: An antiferroelectric capacitor for power electronics

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Proceeding electrification including automotive power electronics demands increased power density, miniaturization, and elevated operating temperatures. One crucial component to define the cost, size, energy efficiency, and reliability of power electronics is the DC link capacitor. For this purpose, CeraLink^{TM} capacitors are perfectly suited due to their high capacitance density, high operating temperatures, low inductance, as well as low losses in operation. CeraLink^{TM} is based on the ceramic material PLZT (lead lanthanum zirconate titante) and characterized by a high energy storage capability due to the antiferroelectric phase. This presentation provides an overview of material and device requirements. Optimization capabilities as well as constraints will be outlined to further advance the capacitor technology based on antiferroelectric materials. Especially, methods to study the process of resistance degradation are addressed to continuously improve the overall reliability as well as the predictability of the device life time.

Reaction mechanism of co-sintered oxidic cathodes for all solid-state lithium batteries

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Li ion conductive ceramics provide additional options for solid state battery. With high ionic conductivity, chemical stability and non-flammability, the Li^+ conductive phosphates are good candidates for improved safety and energy density. Most promising phosphate ceramic is LATP ($Li_{1.3}Al_{0.3}Ti_{1.7}(PO_4)_3$) with Li ion conductivity > 10^{-3} S/cm.

The LATP ceramic can be used as electrolyte as well as catholyte creating the percolating, Li-ion conductive network in a composite cathode. To achieve an optimal Li ion transport in the solid electrolyte and at the interface between solid electrolyte and cathode material, the densification by sintering at elevated temperatures is needed. This processing step leads to the formation of phases at the grain boundaries between both materials, which could interrupt the ionic conduction paths and reduce the capacity of the cathode.

In this study, the reactivity of LATP and commercially available NCM622 ($LiNi_{0.6}Co_{0.2}Mn_{0.2}O_2$) as cathode material is investigated. A mechanistic understanding of reaction mechanism in the relevant temperature range up to 700 °C is addressed. For this purpose optical dilatometry, electron microscopy, X-Ray diffraction and ToF-SIMS are used for characterization the phase formation and microstructure.

The obtained results help to develop the strategies for composite cathodes for co-firing with electrolyte with enhanced chemical material compatibility.

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Model based design of ceramic springs- function and reliability optimization

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The mechanical failure of ceramics- as a brittle material- shows a statistical behaviour and should be analysed on basis of corresponding concepts, as are a weibull distributed strength, weakest link component failure assumption, principal stress related equivalent failure stress and consideration of sub-critical crack growth. Although a corresponding methodology for probabilistic reliability evaluation exists for a longer time, it is seldomly used in the everyday design process of elementary ceramic parts, which are commonly evaluated via a simple strength assessment. Reasons can be found in the complexity of the postprocessing procedures, which are no integral part of most standard FE-Codes, a restricted access to existing specific statistical postprocessors (CARES/Life, STAU, ...) and- most important- the time-consuming and expensive characterisation process of the necessary reliability parameters.
To make a contribution for a more common use of that concepts in the default ceramic component design we present a prototypic workflow for the case of ceramic compression springs. It is based on Finite Element Analysis with standard codes (COMSOL, ANSYS) and a self-developed, external statistical postprocessing tool (Mathematica). Necessary reliability parameters had been obtained for Si_3N_4 and ZrO_2 material variants by experimental characterisation. A simple engineering Excel-tool for spring design has been developed based on a database from a comprehensive FEM-variant analysis campaign. Spring prototypes had been practically manufactured and tested.

Studies on binder jetting of alumina honeycomb structures

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Binder jetting (BJT) is a well-known powder bed based additive manufacturing process. BJT of ceramics results in a relatively low density of green part and so a high sinter density is difficult to achieve. Therefore, BJT is preferred for manufacturing of porous ceramics which are applicable e.g., as filter components, catalyst support or bioactive implants. Within the scope of the project ImProVe (Zwanzig20 AGENT-3D_IMProVe, supported by Federal Ministry of Education and Research) the feasibility and limits of binder jetting for alumina components were investigated. Using a granulated alumina powder (d_{50} = 0,7 µm) different honeycomb test structures with a cell number of 200 cpsi and wall thicknesses of 1,0 mm and 0,5 mm were printed. The size of the components varies between 30 mm and 80 mm diameter. All components could be cleaned well from powder residues and could be handled well. During debinding, the components remained stable and could be sintered under air at 1640 °C. The sintered honeycomb samples were measured using a 3D-profilometer which allows the comparison of target geometry (based on CAD data) and actual size. After variation of sinter support, defect-free honeycomb structures could be manufactured with good matching to the target geometry. These results and the measured open porosity of 51 % and compressive strength of 11 MPa are promising with respect to the manufacturing of complex, porous alumina components via binder jetting.

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Exploring new concepts to design damage tolerant layered ceramics

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Advanced ceramics incorporate multifunctional design to produce high-performance applications in the fields of biomedicine, automotive engineering, electronics, energy and mechanical engineering. The combination of ceramics with other materials (metals, polymers or other ceramics) has enabled the fabrication of hybrid systems with exceptional structural and functional properties. However, a critical issue affecting the functionality, lifetime and reliability of these systems is the initiation and uncontrolled propagation of cracks in the brittle ceramic parts, yielding in some cases very high rejection rates of component production. In this work, design concepts are presented that combine different approaches used in current ceramics engineering to obtain highly reliable ceramic materials with enhanced fracture resistance. It is demonstrated that tuning the location of "protective" layers within a ceramic multilayer architecture can increase its fracture resistance by five times (from 3.5 to 17 MPam1/2) relative to constituent bulk ceramic layers, while retaining high strength (~500 MPa). The use of tailored residual stresses in embedded layers can act as an effective barrier to the propagation of cracks from surface flaws, providing the material with a minimum design strength, below which no failure occurs. Moreover, by orienting (texturing) the grain structure, similar to the organized microstructure found in natural systems such as nacre, crack propagation can be controlled within the textured ceramic layers. A combination of experiments and modelling is here presented, showing the potential of multi-phase layered architectures in the design of future ceramic components with enhanced damage tolerance.

Solid oxide cells: Oxygen ion conductor electroceramics for the energy market: readiness and open challenges

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Solid Oxide Cells (SOC) are electrochemical devices based on Oxygen ion conductor ceramics which are used to generate power from gaseous fuels (Solid Oxide Fuel Cell) and to convert power into gas (Solid Oxide Electrolyzer). Thanks to the high temperature operation, reforming and water-shift reactions also occur within such devices, offering fuel flexibility (H_2, LPG, biogas, ammonia) in SOFC mode as well the capability to generate syngas by co-electrolysis of steam and CO_2, in both cases with high conversion efficiencies and good stability with time. SOLIDpower, a world leader of SOC technology, has developed and commercialized m-CHP appliances in Europe, cumulating up to date more than 40 Mio. hours of field operation, and is currently focused to ramp up production and to expand its product portfolio into commercial and industrial applications.

The paper will provide an overview about readiness of SOLIDpower' stack technology. Particularly, stack lifetime and robustness improvements will be discussed for both fuel cell and electrolysis operation, including update about thermal cycling tolerance, power modulation, endurance in electrolysis and co-electrolysis operation up to 15'000 h. Furthermore, setup and ramp up of an industrial automated production plant of 25 MW per year will be presented. Recent results related to stack power scaleup from 1.5 kW up to 6 kW (single tower) and 25 kW (module with 4 towers) will be presented based on tests performed in DLR Labs. in fuel cell mode and in CEA Labs in electrolysis mode.

Finally, open challenges related to cost reduction and further lifetime improvement for electrolysis and SOFC/SOE reversible operation will be discussed.

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On the importance of surface and interface contaminants in β-tricalcium phosphate bone graft substitutes

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Doping calcium phosphates (CaPs) has been very popular since the publication of Gibson et al (1999) reporting the synthesis of Si-substituted hydroxyapatite. Generally, the main aim of this approach is to modify CaPs biological response by adding a biological relevant anion or cation in the CaP crystal structure. In other words, CaPs are used as carrier for inorganic drugs, and the release of these inorganic drugs occur during CaP resorption. Unfortunately, the incorporation of foreign ions in the CaP crystal structure modifies the crystal shape and solubility, and as such, it is not possible to univocally attribute a biological effect to the release of the incorporated inorganic drug. Indeed, it could also be due to a change of surface topography or a change Ca or phosphate release. Another difficulty related to CaP doping is associated with the determination of the location of the doping agents. Most studies conclude from a change of crystal lattice parameters that all inorganic drugs are incorporated in the crystal lattice even though part of the ions could be located at grain boundaries. The aim of this communication is threefold: (i) briefly summarize this field of research, (ii) describe and illustrate the difficulties to produce pure calcium phosphate powders and sintered blocks, and finally (iii) present some in vitro data demonstrating the link between processing conditions (incl. sintering), purity / doping, and osteoclastic resorption. It will be in particular shown that the sintering atmosphere, surface impurities due to phosphate evaporation during sintering, and the Ca/P molar ratio affects the osteoclastic response. Data on Sr and Cu-doping will also be presented.

Segregation-controlled densification and grain growth in rare earth doped Y_2O_3

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Cation doping of Y_2O_3 is established for tailoring densification and grain growth during sintering. However, the segregation of doped cations to the grain boundary and its impact on sintering is still not completely understood. It can be driven by either electrostatic effects due to charge mismatch with the host lattice or by elastic effects induced by ion size mismatch. While being caused by the reduction of the total energy, segregation itself impacts diffusion and kinetics of grain boundary migration through the solute drag effect.

In this study, we utilize two isovalent dopants (Gd^{3+} and La^{3+}) aiming on to exclude the impact of charge mismatch onto segregation. When sintering doped Y_2O_3 via FAST/SPS, Gd^{3+} does not significantly affect densification, while La^{3+} resulted in a strongly reduced sintering activity. Furthermore, the grain growth kinetics during sintering reveal a decrease of the grain growth coefficient, with La^{3+} having the stronger impact. Aberration corrected TEM studies reveal that no structural change was induced by doping. Chemical analysis showed strong segregation of La^{3+} to the grain boundary, which was not observed for Gd^{3+}. Obviously, La^{3+} segregation causes significant decrease of grain boundary migration rates through solute drag and slower sintering kinetics due to reduced grain boundary self-diffusion. The study hints on the importance of the elastic contribution to cation segregation and its influence on to grain growth and sintering kinetics.

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Searching for new photovoltaic absorber materials: properties of triple perovskite $Ba_3CuNb_2O_9$

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Ferroelectric photovoltaics is attracting a lot of attention since ferroelectricity has been proven for metal halide perovskites whose efficiency has increased rapidly over the past two decades. While ferroelectric domains could be advantageous for efficient charge carrier separation some fundamental problems of metal halide perovskites will be difficult to overcome. Their use of toxic materials and degradation issues motivate to seek for new efficient, earth abundant, non-toxic and stable absorbers for thin film solar cells in the field of oxidic perovskites.

As the field of material combinations is broad, the present study, starting from band structure simulations of $Ba_3CuNb_2O_9$, shows how important material properties can be obtained by thin-film processing with adequate characterization methods. The main focus was placed on basic requirements for absorbers such as band gap, absorption coefficient and charge carrier mobility.

Thin films with a thickness of several hundred nanometres of the perovskite $Ba_3CuNb_2O_9$ were processed by pulsed laser deposition. Furthermore, bulk samples and thin films have been characterized regarding fundamental material properties by Rietveld refinement of neutron diffraction data, UV-VIS-NIR spectroscopy, as well as SEM-EDXS.

Predicting the behaviour of refractory linings - up to the fracture and beyond

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High temperature Industrial processes rely on refractory linings to protect the equipment and reduce energy losses. As a direct consequence, one face of refractory linings usually experiences high and fluctuating temperatures, while the other face remains significantly colder. In use, considerable steady and transient thermal gradients thus arise within linings, accordingly causing thermal stresses. Especially during the first heating cycle of a lining, when the temperature rises at the hot face, substantial thermal stresses emerge. There are two ways for a refractory lining to deal with them: Fracture, as soon as the strength of the material is exceeded, or creep, notably at the hot face. Both relieve excessive thermal stresses. In practice, and depending on the temperature, those two processes occur simultaneously and thus influence each other.

Due to their complex microstructure and their specific way of failure, the behaviour of refractory linings in use is difficult to predict. Above all, it is not the initiation of fracture (that occurs anyway), but the ability of refractory materials to sustain damaging under stress that distinguishes well-designed refractory products. New methods for determining thermomechanical properties of refractories at high temperature, coupled with novel simulation concepts, allow identifying weak points of refractory linings under thermomechanical load as well as predicting zone at higher risk of damaging. It could be demonstrated that, in castable refractories, rather splintered tabular grains promotes higher resistance to fracture by enhancing the tensile strength of the material, as well as that a finer, more reactive matrix promote a better resistance to thermal stresses induced damaging.

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Constrained sintering in Al_2O_3 and $MgAl_2O_4$ based ceramic laminated composites

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Ceramic laminated composites (CLCs) have been developed to improve the fracture toughness of monolithic structural ceramic materials. Using co-sintering, tape-cast layers of different materials are combined to obtain highly reliable composites. The individual layers exhibit anisotropic shrinkage during sintering, due to alignment of non-spherical particles during tape-casting. Co-sintering of heterogeneous multilayer ceramic laminates may lead to unacceptable distortion or to premature fracture. The failure is induced by internal stresses resulting from mismatches in thermal expansion and sinter shrinkage between layers. The Skorohod-Olevsky model for viscous sintering was modified in order to take into account anisotropic shrinkage. Implemented in an FE environment it has been used to predict the residual stresses and to simulate laminate deformation. Material and laminate data for simulation and validation included, among others, the shear viscosity and creep activation energy that have been determined using viscosimetry experiments, measurement of elastic constants at elevated temperature using impulse excitation, determination of grain size and dilatometry measurements to determine sinter shrinkage evolution and macroscopic curvature of single layers, bilayers, and 3-layer laminates. While residual stress simulation delivers qualitatively reliable results, the predicted densification and deformation are in good agreement with experimental data.

Influence of Nd-substitution on (La,Ca)FeO_3 perovskites with regard to application in solid oxide fuel cell cathodes

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Mixed ionic and electronic conducting perovskites from the series (La,Sr)(Co,Fe)$O_{3-\delta}$ are promising candidates for a broad variety of applications, such as cathodes for solid oxide fuel cells (SOFCs), anodes for solid oxide electrolyser cells (SOECs), electrochemical gas sensors or catalysts. These materials show superior performance in the short term, but often lack long-term stability under application-relevant conditions (several thousand hours of usage, presence of critical contaminants), preventing their broad market introduction. Recent studies showed that substitution of Ca for Sr and Fe for Co improved the oxygen exchange kinetics by a simultaneous enhancement of the long-term stability against SO_2. In order to optimize these materials, the influence of the A-site substitution by partially replacing La with Nd was investigated, considering the crystal structure, distribution of cations, and important material properties like thermal expansion coefficients and electronic conductivity. Based on these results, structure-property relationships for the Nd-substituted series ($La_{0.8-x}Nd_xCa_{0.2}$)$FeO_{3-\delta}$ with 0<x<0.6 were derived. All investigated materials show favourable properties for the above-mentioned applications.

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Modelling of the densification under pressure of ceramic parts placed in a powder bed

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Spark Plasma Sintering (SPS) is a rapid sintering method of ceramics. It combines the application of uniaxial pressure and Joule effect heat treatment through the passage of a high intensity electric current within the mould and (possibly) in the powder to be sintered. This technology has many advantages: the ability to quick densify a large series of materials, often leading to improved end-properties, thanks to better microstructure control. The thermal cycle is shorter leading to energy savings per unit of material produced. More recently, hybrid heating modes, allowing to reduce temperature gradients in large parts, have broaden its field of applications. However, there is a strong limitation in the shapes that can be produced (simple forms, classically achievable by uniaxial pressing), which clearly reduces the scope of the SPS.

The aim of this talk is to present a SPS densification method for complex shape ceramic parts, without any modification of the tools and equipment. The green samples are placed in a powder bed in the classical die used in SPS. Different powder beds have been tested (graphite and silicon carbide) on different materials (alumina and tungsten carbide).

The force exerted on the granular medium and on the surface of the part to be sintered is simulated by a digital model, based on the Janssen model conventionally used to measure the granular pressure in a container. This allows rapid simulation of the force field around a part surrounded by a bed of powder in the SPS tool. In the presentation, we will describe the optimization of the operating conditions which was carried out first on small samples of simple shape, then on parts with complex geometry to demonstrate the feasibility of the proposed sintering process.

Hybrid additive/subtractive manufacturing system to prepare dense and complicated ceramic parts

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As the conventional binder jetting method is limited in terms of achievable density and finish surface, a new concept of hybrid subtractive/additive manufacturing system has been recently developed. The system relies on a modified binder jetting machine which integrates a slurry-based deposition system and a pulsed laser. The slurry-based deposition system is a spray device which can be an alternative to the recoating blade traditionally used to spread layers of coarse powder. By using slurry containing fine ceramic grains, thin layers of highly packed powder can be obtained which is highly beneficial to improve the sintered density of printed parts. The other limitation of binder jetting is the rough finish surface. Thanks to the smallness of the spot size, a pulsed laser is a good option to refine the contour of each printed layers thus enhancing the overall part resolution and smoothness.

The present work exhibits some preliminary results obtained using this new concept of hybrid machine. Parts made in alumina have been produced demonstrating the advantages and current limitations of the new approach. Alumina powders have been processed to change the particle size, distribution and morphology in order to assess the influence of these characteristics on printing behavior. Furthermore, process parameters were studied such as layer thickness, deposited amounts of binder on each layer, spreading speed of recoater. This work also reports the results of the first tests using the laser to refine the borders of each printed layer. The influence of this treatment on the finished surface of the sintered parts is highlighted.

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Can indentation cracks describe residual/internal stresses in Al_2O_3/ZrO_2 laminates?

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The strongly bonded laminates typically alumina/zirconia (Al_2O_3/ZrO_2) layers can form relatively high residual/internal stresses reaching in some cases the strength of materials. The effective management of residual stresses via proper design/architecture can result in a combination of high fracture resistance with high strength and stiffness. The presence of compressive residual stresses formed in Al_2O_3 layers can suppress and deflect cracks propagating through the layers. The easy to use and very common method of how to estimate fracture behaviour in ceramic materials is indentation technique. The residual stresses are in literature also commonly investigated by this method. In case of laminates with strongly bonded layers is the crack path in the vicinity of interface governed by both the elastic properties and the internal stress field of individual layers. The laminates alternating Al_2O_3 and ZrO_2 layers with various layer-thickness ratios ranging from 0.1 to 3 were used to investigate the effect of residual stresses and influence of crack formation pattern on the crack path development. The indentation surface cracks observed in various alumina-zirconia laminates exhibit the same crack deflection at the interface independently on the level of internal stresses. This finding was explored in more detail and compared with the crack deflection observed on the fracture surfaces of bending specimens. The observed complicated crack path was explained experimentally by 3D reconstruction with the support of numerical simulations.

Piezo-ceramics and piezoelectric MEMS optimization by SPS: advantages of a protective layer

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The choice of the elaboration process for piezoelectric PZT-based ceramics is crucial for ease of implementation, energy and cost savings, and decisive from the point of view of densification and piezoelectric performances. The combination of screen-printing and Spark Plasma Sintering is particularly promising for low temperature single step sintering of thick layers of PZT on metal substrates for MEMS (Micro-Electro-Mechanical Systems). To date, few studies have reported the sintering of multilayer by SPS for microelectronics applications and the different issues identified are mainly related to interfaces. SPS of functional oxides usually involves several hours of subsequent annealing at temperatures well above 800ºC. In the case of PZT, this annealing is crucial to remove oxygen vacancies and then to restore good electromechanical properties. We will show here that using a protective carbonate layer to avoid a post annealing is an efficient way to obtain in one step performant piezoelectric ceramics. Then, the transfer of the sintering process to screen-printed PZT layers on steel substrate will be presented. Finally, we will conclude by highlighting the flexibility of the SPS in terms of set-up design and sintering environment which makes this process particularly suitable for sintering functional materials.

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Debinding and sintering of dense oxide ceramic structures made with fused deposition modeling

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Shaping and thermal processing of ceramic structures with fused deposition modeling (FDM) is promising, but still a challenging technique. Achieving an optimal compromise between the thermoplastic feedstock properties in terms of 3D printing and debinding behavior is not trivial. We studied Mullite, Al_2O_3, ZrO_2, Al_2O_3-ZrO_2 ceramic feedstocks based on Ethylene Vinyl Acetate (EVA) and stearic acid as a binder system. Different EVA grades and stearic acid concentrations were investigated. Our objective was to produce flexible and strong filaments, which can be used in commercial FDM printers with 2.8 and 1.75 mm filament size. An addition of stearic acid significantly influences the rheological properties and improves printing and the debinding behavior, but also makes solidified filament more brittle. Therefore a fine adjustment of the binder composition is needed to avoid the brittleness of filaments and still maintain good shaping possibilities. We will show how printing parameters have to be optimized to achieve fully dense 2.5 mm ceramic disc structures. Large 3D parts were made to study the debinding and sintering processes. Excellent fusion between the different layers of 3D printed parts was achieved without defects at interfaces. Thermal analysis with GC-MS characterization of decomposition gases was used to better understand the binder removing mechanisms and to optimize the debinding program. Pure thermal removing of the binder in air is challenging, due to the formation of dense skin during the first stages of EVA decomposition. However, dense structures with a thickness of 5 mm can be already achieved by following our approach. Using additonal solvent debinding process step, 3D printed parts with a thickness of 10 mm can be achieved. Microstructure and mechanical properties of sintered structures were also investigated.

Thermal sprayed coatings of the Al_2O_3-TiO_2 system

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Materials from the Al_2O_3-TiO_2 system are used for the preparation of thermally sprayed coatings for many decades and have a big practical importance. Typically feedstock powder compositions containing 3, 13 and 40 % TiO_2 are used. These powders are prepared by different processes, such as fusing & crushing, agglomeration & sintering. However, the interactions between Al_2O_3 and TiO_2 , in particular the formation and decomposition of Al_2TiO_5, occuring during feedstock powder preparation and during spraying are porly studied so far. The influence of Al_2TiO_5 on the coating properties is also poorly known. In this contribution, the state-of-the-art of knowledge of this type of coatings is reviewed. Microstructures and phase compositions are linked with the mechanical, tribological, electrical and corrosion properties of the coatings.

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Polyanionic materials at the positive of Na-ion batteries

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Polyanionic materials are intensively studied as promising positive electrode materials for Na-ion batteries thanks to their high stability and fast ionic mobility within their structural framework. Among those polyanionic materials, $Na_3V_2(PO_4)_2F_3$ and $Na_3(VO)_2(PO_4)_2F$ are the two most attractive ones due to their high voltage for two Na+ ions extraction and their high theoretical energy densities: 500 mAh.g^{-1} and 495 mAh.g^{-1}, respectively. These two compositions are indeed the two end members of a family of compounds described with the general formula $Na_3V_2(PO_4)_2F_{3-y}O_y$ where $0 \leq y \leq 2$. We will first discuss the stability of these active compounds in aqueous media and in enlarged potential window, and the performance of optimized carbon-coated stoichiometric $Na_3V_2(PO_4)_2F_3$ in a large panel of cycling conditions (at different rates, temperatures etc.). Then, we will show how cationic and anionic substitution have been widely explored with the target to reversibly de-intercalate and re-intercalate the third Na^+ ion from/in the structure.

Manufacturing, characterization and testing of novel temperature-resistant dual-bell nozzle structures based on liquid silicon infiltration

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Ceramic matrix composites (CMCs) are considered as the most promising materials to replace super alloys in common rocket propulsion systems due to their excellent mechanical and thermal properties at high temperatures and low material densities. To demonstrate the capabilities of CMCs, a test campaign for novel dualbell nozzle concepts which includes the test of two fiber-reinforced nozzle extensions were performed. Both extensions were designed and manufactured with different phenolic resins. The green bodies were produced by wet filament winding. To reduce delaminations, the layers were built employing multi-angle fiber architecture. While one of the nozzle preforms remained in the CFRP state, the other one was ceramized to the C/C-SiC state via liquid silicon infiltration. The influence of the winding process on the resin systems was investigated in analytical procedures. To characterize the material, detect flaws and show the contour accuracy, computer tomography scans have been carried out at different stages of the developmental process. Furthermore, the quality of the material formed was determined by microstructure analysis using SEM. In preparation for the gas flow test, the nozzles have been subjected to internal pressure tests which show the helium leak rate and the permeability of the structures. The structural integrity and thermal stability were tested by hot gas firing tests under laminar and separated flow conditions.

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Smart biomaterials for tissue engineering of complex tissues

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The biofabrication of 3D biomimetic tissue analogs, which accurately mimic the properties of native tissue samples, have an enormous potential in biomedical applications (drug discovery, cancer research, regenerative medicine,…). The main challenge in tissue engineering is the creation of functional complex tissues such as bone, cartilage, muscle, adipose tissue,… Strategies to engineer these tissues can be categorized in top-down, scaffold-based approaches and bottom-up, developmental biology inspired approaches. During this lecture, we will first focus on the application of biomaterials/scaffolds in top-down tissue engineering. In the second part, we will specifically evaluate the role of smart biomaterials in the field of bioprinting complex tissues. Combining both scaffold-based and developmental biology-based tissue engineering will have a synergistic effect on the fabrication of 3D tissue analogs. Cellular building blocks with self-assembling properties and mimicking the tissue of interest are combined with cell instructive biomaterials. The cellular building blocks are either tissue-specific or vascular. High quality homocellular building blocks were already generated that form tissue-specific cellular building blocks ((fibro)cartilage, bone, adipose tissue,…). These tissue-specific spheroids can be combined with vascular spheroids providing the capillary like network. 3D bioprinting of spheroids in photocrosslinkable hybrid hydrogels was performed. The tunable characteristics of smart biomaterials can influence outgrowth, fusion and phenotype of the spheroids.

High production yield synthesis of garnet type $Li_7La_3Zr_2O_{12}$ for all-solid-state li-ion batteries: Understanding decomposition thermodynamics

DERMENCI, Kamil Burak

Garnet type LLZO solid electrolytes are considered as a promising solid electrolyte candidate as they show considerably high ionic conductivity, outstanding stability against ambient atmosphere and a wide electrochemical stability range. However, stabilizing garnet type LLZO requires high temperatures and long heat treatment times which can cause extensive Lithium losses. Hence, the powder bed technique was usually used and the production yield is rather low.

During the synthesis of LLZO solid electrolytes, a tremendous effort has been conducted for lowering the heat treatment temperatures to develop a more feasible process. Pechini-derived method is a convenient option to synthesize garnet type LLZO solid electrolytes in a powder form by maintaining a high production yield. However, very little work has been conducted to enlighten the insights of Pechini-derived LLZO production.

In this study, the Pechini-derived LLZO xerogels – the precursors of garnet LLZO – was obtained starting from the aqueous solution of $LiNO_3$, $La(NO_3)_3$, $ZrOCl_2$ salts. Up to 40 mole % of $Zn(NO_3)_2$ was added as a stabilizing agent. To analyze thermodynamics of LLZO xerogel decomposition, thermogravimetry was used and TG/DTA was performed using scan rates between 5 – 40 K/min under air atmosphere. Thermodynamic parameters, the changes in Gibbs Free Energy (ΔG), Enthalpy (ΔH) and Entropy (ΔS), as functions of degree of decomposition were assessed and the effect of the stabilizing agent was discussed.

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Electrical properties and cation distribution in NTC thermistor Fe-Ni-Mn spinel oxides

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The majority of commercial NTC thermistors ceramics is based on substituted spinel-type $NiMn_2O_4$. The operating temperature of most of these materials is limited to about 120 °C; moreover, they exhibit a low long-term stability of the electrical properties at elevated temperatures. $NiMn_2O_4$ is stable between 730 °C and 920 °C only. Above this temperature it decomposes into NiO and a Mn-rich spinel, whereas at lower temperatures $NiMnO_3$ and Mn_2O_3 are formed. Moderate cooling regimes lead to formation of metastable $NiMn_2O_4$ spinel. Substituted $NiFe_zMn_{2-z}O_4$ spinels exhibit improved thermodynamic stability and electrical properties. We report on the preparation and electrical characteristics of thermistor oxides and multilayer NTC thermistors.

To understand the electrical properties of the spinel oxides, the cation distribution was investigated using in situ neutron powder diffraction (NPD) from 25 °C up to 900 °C. Rietveld refinements of NPD data allow to extract a temperature dependent inversion parameter v, which describes the distribution of cations between tetrahedral and octahedral sites of the spinel structure. In addition, the Seebeck coefficient was measured as function of temperature. Using a modified Heikes' formula, the disproportionation p of manganese was analyzed from Seebeck data. Combining both results allows to derive a cation distribution model $(Ni^{2+}_{1-v}\ Mn^{2+}_{p}\ Mn^{3+}_{v-p})_{tetrahedral}\ [Ni^{2+}_{v}\ Mn^{3+}_{2-v-p}Mn^{4+}_{p}]_{octahedral}$. The same methodology was used for Fe-substituted spinel oxides. Finally, the temperature dependent cation distribution is discussed in terms of the influence of manganese in various valences on the stability and electrical properties of NTC thermistor ceramics.

Simulation of sintering o photonic structures with discrete element method

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In this contribution, the use of the discrete element method (DEM) has been proposed to investigate sintering-induced structural deformations occurring in reflective thermal barrier coatings (rTBC). The rTBC are highly porous structures with three-dimensional highly-ordered macro pores, also called inverse opal photonic crystals. In experiments, they show crack formation and opening of the structure due to sintering effects, although an application as TBC requires stable macroscopic mechanical, photonic and structural properties at high temperatures.

The investigated structures have been represented as a set of interacted primary particles, and then the influence of the initial arrangement of template particles on the stability of the structures has been analyzed. The material transport, densification, as well as formation of defects and cracks have been compared for various case studies. The results of the studies carried out have shown differences in material transport within the structure, leading to the formation of defects, e.g. initial cracks on the surface and within the structure at the struts. It has been observed that defects start predominantly at the former connection points between template particles and propagate, leading to the formation of interpore cracks. At later stages, the intrapore cracks with lengths exceeding 0.7 µm are formed.

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Micro- and macrostructural design of ceramic composites applied to advance technologies in various industries

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The process of developing new products that will advance the technology for specific industry needs is at its core based on applying one or more 3M technology platforms. Ceramics "Ce" is one of these platforms in the 3M periodic table. Ceramics make the difference in many industry fields ranging from steel & metal processing, automotive & electronics all the way to cosmetics & dental applications. One common term is the micro- or macrostructural design that integrates the specific advantages of ceramic materials into a composite or article. Based on this general observation specific application examples will be given that span the full breath of opportunities ceramics have to offer, some of which might not be obvious from a traditional ceramic material perspective.

For example, ceramics play an important role in thermal management from highly thermally conductive solutions (e.g. cooling of battery systems) up to thermal insulation under extreme conditions (e.g. containing the effect of a battery thermal runaway). For thermal management high aspect ratio filler particles like hexagonal boron nitride applied to microstructural design in a polymer matrix will increase thermal conductivity. Macrostructural design on the other hand based on ceramic fibers will help to protect passengers from heat and particle extraction in the event of an automotive battery failure. Just two of many examples where ceramics make the difference.

Rehydroxylation of ceramic tiles and its influence on curvature effects after firing

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The development of roller kilns reduced the firing times and the energy consumption in tiles production. However, a sintering time of only a few minutes does not enable a complete thermal conversion of clay minerals, resulting in disequilibrium of sinterphases. Time-dependent rehydroxilation of transition phases (X-ray amorphous clay minerals) by environment moisture absorption results in deformation of the ceramic product on a middle to long term basis. Standard production controls at the kiln exit fail to assess this effect, which is more noticeable with large-format products. Countermeasures in production like a process-controlled pre-curvature in pressing are accompanied by high process uncertainty and high storage costs. These rehydroxilation reactions of ceramics and the associated mass gain occur in two distinct stages. Stage I is characterized by moisture equilibration, whereas Stage II reactions refer to long-term chemical rehydroxylation. In order to gain more detailed information on the influence of raw materials and ceramic mixes, experiments were performed and evaluated using RBA, DTA-IR, FTIR, mass gain after autoclave treatment. It has been observed that mass gain is directly related to calcination temperature and phase composition. Clays from different deposits with identical mineralogical composition (as analyzed) show different rehydroxilation behavior under firing.

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Dislocation-based plasticity and crack formation in oxides: a case study on strontium titanate

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In light of the rising topic of dislocation-based functionality of oxides, the dislocation-based mechanical behavior, for instance, dislocation plasticity and potentially crack formation induced by dislocations is also drawing increasing attention. Dislocations are line defects and the main carrier of plastic deformation. Understanding the dislocation-based mechanics in oxides can play a critical role in assessing the materials' mechanical and operational reliability. In this talk, we present an approach to evaluate the incipient dislocation plasticity and crack formation using nanoindentation. In addition, the concept of "defect chemistry engineering" is proposed for the first time, based on which the dislocation plasticity of the oxide crystals can be tuned, as will be demonstrated in single-crystal $SrTiO_3$. Two methods, i.e., via stoichiometry change of the Sr/Ti ratio, and by reduction treatment to increase oxygen vacancy concentration, have been validated to modify the dislocation nucleation and dislocation motion. These are both keys to dislocation plasticity. Our approach may also serve as a potential method to increase the fracture toughness of the material when appreciable dislocation plasticity can be achieved.

Triple-phase ceramic 2D nanocomposite with enhanced thermoelectric properties and its application in thermogenerators

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Sol-gel process was used to produce heavily doped misfit-layered cobaltate $Ca_3Co_4O_9$, which by subsequent heat treatment in suitable atmosphere self-assembled into two-dimensional (2D) nanocomposite as confirmed by high-resolution transmission electron microscopy (HRTEM). Nanocomposite was preserved in sintered ceramic, which showed enhanced thermoelectric properties parallel to the cold-pressing direction as applied to the green body. This went along with the preferential alignment of multiphase misfit-layered oxide platelets. HRTEM confirmed 2D nanoslabs of three individual thermoelectric oxides, which contributed synergistically to the thermoelectric properties of the nanocomposite. Different sintering procedures were applied: hot pressing (HP), spark plasma sintering (SPS) and pressureless sintering in air or oxygen. In all cases, the isothermal electrical conductivity and the Seebeck coefficient increased. In the oxygen-sintered ceramic, the power factor was enhanced to 8.2 µW cm^{-1} K^{-2} at 1073 K in air. According to Ioffe plots, i.e. thermoelectric power factor versus electrical conductivity, 2D nanocomposites where chosen to manufacture prototype thermogenerators (TEGs) by combining them with doped indium oxide (Ge,Mn,Zn:In_2O_3; Sn,Al:In_2O_3). The electric current-power characteristics of TEGs hint to the primary importance of the power factor compared to the thermoelectric figure-of-merit when a high electric power output is desired.

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UltraHigh temperature sensors based on boron carbide composites

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Boron carbide is frequently used for wear resistant components, ballistic protection and neutron absorption because of its high hardness, low specific weight or high neutron absorption efficiency. Furthermore, boron carbide combines a unique spectra of electrical and thermal properties. There are the outstanding high Seebeck coefficient (> 300 µV/K) and a moderate electrical (1 S/cm) and thermal conductivity (20 W/mK) at room temperature. Those properties perfectly match the requirements of a thermocouple which can be used even at extremely high temperatures of more than 2000 °C. First products of boron carbide – graphite thermocouples had been commercialized about 30 years ago but they were not kept on the market.

A new approach to an improved product is described here. The materials of the thermocouple comprises only boron carbide composites which differ only in their exact composition. This allows to shift the melting point above 2400 °C and additionally produce a precise temperature signal by the sensor. The specific manufacture and design of the thermocouple reduces the thermal mass of it considerably compared to the thermocouple of. Consequently, the thermal impact to the process is minimized as well. Principal solutions of the assembly are improved to maximize the operation time and the reliability of the thermocouple, too.

Additive manufacturing with preceramic polymers

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Preceramic polymers convert into nanostructured Si(X)OCN (X = Al, Ti, Zr, etc.) ceramic materials by high temperature pyrolysis. Additive manufacturing (AM) technologies are generally more established for polymeric materials than for ceramics; therefore, employing preceramic polymers in such systems can be a successful alternative for the fabrication of 3D components with complex shapes. Different types of additive manufacturing (AM) techniques were employed, with particular focus on direct ink writing (DIW) and digital light processing (DLP). Advantages and disadvantages of the different techniques will be discussed, together with examples of materials and proposed applications. SiOC components were fabricated, as well as structures of advanced silicate ceramic phases, including bioceramics, starting from preceramic polymers (e.g. silicones) and inert or reactive fillers. Ceramic matrix composites (CMCs) with aligned reinforcement were also developed exploiting the shear stresses generated by the DIW process itself.

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Comprehensive optimization of debinding processes in ceramic production

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Debinding is a critical step in the processing of ceramics. In particular when ceramic components are large or complex or have a high binder content. To prevent component failure during debinding the processes are usually carried out very slowly in industry. Thus, there is a huge potential for saving time and energy through the optimization of the time-temperature cycles. A first step towards tailored debinding cycles is to construct time-temperature profiles with a constant debinding rate from thermogravimetric measurements. However, a comprehensive optimization requires much more details to be taken into account: gas formation and temperature gradients cause internal stresses, local temperature is modified by the exothermic or endothermic binder decomposition and material parameters like gas permeability, heat conductivity and mechanical strength vary as a function of temperature and degree of debinding.

In this work, we present a validated method for systematic and universal debinding process optimization accounting for all mentioned phenomena. The method is based on experimental data on the process kinetics and state-dependent material properties as input for an FE model of debinding in the considered component geometry. The risk of green body damage is assessed in the model with respect to temperature cycle, furnace and firing stack as well as component geometry. After experimental validation, the optimized debinding cycles can be transferred to industrial production.

NBT-based high temperature capacitor materials

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There is an increasing need for electronic components for sensing and controlling purposes in harsh environments. Hence, the properties of the electronic circuitry shall not vary over a large temperature range with maximum temperatures beyond 200°C. Many ceramic materials used for capacitor applications undergo phase transitions, which increases permittivity, and the electric loss rises with temperature. Therefore, it is difficult to obtain a stable temperature independent capacitance. Recently, promising lead-free solid solutions of sodium bismuth titanate (NBT) have been presented, which have unique properties rendering them excellent materials for possible capacitor applications. However, the high loss is still a major problem. Therefore, the defect chemistry for such a critical high-temperature component has to be well known. In this study the defect chemistry of $Na_{0.5}Bi_xTiO_3$-$BaTiO_3$-y$CaZrO_3$ (NBT-BT-yCZ) is investigated. NBT-BT-CZ exhibits an extremely high temperature stable permittivity range. The materials show a significant overlap of a large temperature-independent permittivity range and the loss factors can be optimized far below 0.02. NBT-BT-20CZ shows an overlapping regime of -70 °C up to 370 °C. Both, permittivity and loss factor are so encouraging that further capacitor requirements were investigated. It was possible to show that all materials can compete with the energy density of a commercial X7R. Furthermore, NBT-BT-20CZ reaches an excellent energy efficiency of 98%.

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Suspension flame sprayed metal doped calcium phosphate coatings with antibacterial properties for infection prophylaxis

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High Velocity Suspension Flame Spraying (HVSFS) has been successfully employed to produce a wide variety of calcium phosphate and bioglass based coatings for metal bone implants. The HVSFS process has proven to be capable to process all kinds of these biomaterials resulting in dense and well adherent coatings on all types of metal substrates. In recent years the focus has gradually shifted to a group of degradable bioceramic coatings, as they offer a faster osseointegration of the endoprosthetic structure. A common problem that occurs after the implantation of coated implants is the risk of infection due to the presence of bacteria which can result in severe post operative inflammation reactions associated with a high risk to lose the implant. In a novel approach, metal ions with known antibacterial properties, such as silver (Ag) or copper (Cu), are incorporated into the coating as a metal dopant to reduce the risk of inflammation.

In this study, metal doped coatings based on HA, tricalcium phosphate, bioglass and a calcium potassium sodium phosphate were suspension flame sprayed using modified suspensions containing additional metals or metal salt based precursors. Metal doped coatings were evaluated regarding their microstructure and phase composition, as well as their in-vitro behavior. The presence of metal and metal oxide particles in the coating were detected using micro-Raman and scanning electron microscopy. To evaluate the biocompatibility, a live/dead-assay viability / cytotoxicity study based on MG-63 cells was performed. Results did not give evidence for any cytotoxic reaction.

Thermal spraying of oxide-ceramic friction surfaces for composite brake rotors with reduced fine dust emissions

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Pollution emissions are a crucial topic in automobile business and public transport with distinct political impact up to driving restrictions in metropolitan areas. Beside the NO_x emission problem fine dust is the most important issue. Recent research showed that most emissions from the transport sector are not coming from combustion engines but from brake and tire wear (non exhaust emissions). The paper presents a novel brake rotor engineering concept made by thermal spraying and focused on lightweight, improved brake performance and reduced fine dust emissions. The industrially widespread and cost-effective thermal spray processes are used to apply high-performance ceramic coatings onto aluminum rotors. The result is a layer composite component consisting of a wear- and corrosion-resistant friction surface that ensures the required braking performance and of the FEM-optimized base rotor design, that meets the strength and lightweight requirements.

The present coating composite design separates the friction surface and substrate material, offering the possibility of tailored tribological systems for individual brake requirements, keeping the overall manufacturing cost in an acceptable range. Furthermore a considerable decrease in brake particle emissions can be achieved. Future potential applications and advantages especially for brake systems in the context of new electric mobility concepts will be discussed.

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Pressure slip casting of spider bricks, and their corrosion behaviour during ingot casting

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Pressure slip casting is a near net shaping manufacturing method. Several advantages are prospected by application of pressure slip casting technology for production of coarse-grained refractories, like i.e. no finishing of the refractory components and the generation of smoother surfaces. Using the example of a spider brick -a manifold in the ascending ingot casting process in the steel metallurgy-the development and optimization of coarse-grained slips will be presented. For example, the spider brick must fulfil harsh requirements relating to thermal shock and corrosion resistance. For the steel industry, it offers advantages such as a denser surface which reduces corrosion by the steel melt. In summary, higher stability of the spider brick leads to better steel cleanliness.

To cast such a complex shape, a binder system to obtain dimensionally stable bodies without deformation, cracks, and cavities, as well as an optimal grain size distribution, had to be found. Additionally, the component homogeneity, casting time, and thermal shock resistance have been optimised in order to successfully test the spider brick under real ingot casting conditions at 1500 °C for 45 minutes at the Deutsche Edelstahlwerke Specialty Steel GmbH steel shop at Siegen. After the industrial test, the corrosion behaviour has been evaluated.

Analysis of sintering by means of thermoanalytical methods

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The IKTS offers a variety of instrumented furnaces on a pilot plant scale and thermoanalytical methods on a laboratory scale that are used to optimize sintering processes. Changes in the properties of the materials of interest are analyzed in dependence on temperature, time and atmosphere, and other process parameters (component size, furnace workload, ...). The influence of these parameters on the product properties are determined. The results enable the control and optimization of technical processes on a production scale.

When producing ceramics precise knowledge of the shrinkage behavior during sintering (and thus also of the change in density) is of particular interest. Information about the beginning and the end of critical processes allow the optimization with regard to energy-efficient and cost-effective production. They are obtained through thermodilatometric examinations on a pilot plant and laboratory scale.

The results can be used to describe the kinetics of the processes by Kinetic Field of Response, Master Sintering Curve or Thermokinetic method, respectively. This offers the possibility of assessing temperature-time profiles without time-consuming laboratory-scale experiments or pilot plant trials. This is demonstrated by characterization, modelling and optimization of the thermal processes during production of High-Performance Ceramics (Si_3N_4, Al_2O_3, ZrO_2, ...).

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Creation of reaction bonded silicon carbide by binder jetting

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Binder Jetting, classically also known as three-dimensional printing, is one of the most efficient additive manufacturing technologies to create large and complex shaped ceramic parts. It enables the production of prototypes as well as final products, which may not be realized by established shaping techniques. One main drawback of the technology is the immanent porosity of printed green bodies, due to dry powder deposition methods. This usually prevents the creation of parts with material properties which are technically sufficient.

In contrast to the vast majority of technical ceramics, powder bed porosity is not an obstacle for the production of components made of reaction bonded silicon carbide (RBSiC). Instead a porous network is a prerequisite for the liquid silicon infiltration (LSI) process which follows the creation of green bodies. However, green part porosity as well as microstructural inhomogeneity have to be kept on a very low level to be able to produce technical components by binder jetting.

The presentation will give some insights into material and process development, which was key to enable the production of three dimensional printed RBSiC with excellent properties. Today we can exploit process-related advantages of additive manufacturing providing a new dimension of constructive design potentials and address demanding market segments of lithography, metrology and thermal process technology.

Supercrystalline nanocomposites: Boosting and controlling the mechanical behavior of these new multifunctional materials

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Supercrystalline nanocomposites have emerged in the last decade within the broader field of colloidal crystals. They consist of inorganic nanoparticles, surface-functionalized with organic ligands and arranged into periodic structures, very reminiscent of atomic crystals. The combination of nano-sized building blocks and their long-range order arrangement leads to a whole set of emergent functional properties, with potential applications in optoelectronics, magnetic devices, catalysis, and more. A major obstacle towards the full exploitation of these materials into devices, however, is the lack of information on their mechanical behavior, accompanied by usually poor mechanical properties. An important improvement in this direction has recently been made by inducing the formation of a crosslinked network of covalent bonds in the organic phase that interfaces the nanoparticles, via heat treatment at mild temperatures. This same covalent network alters the deformation behavior of the nanocomposites, as established via multiscale ex- and in-situ studies. Many analogies with atomic crystals emerge, even if length scale and interactions between building blocks vary significantly. Future directions for the development of supercrystals with tunable behavior are outlined.

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Unconventional low temperature sintering process of ceramics and multimaterials: the hydro/solvothermal sintering

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The development of new high performance advanced materials faces the challenge of implementing low temperature densification processes to overcome current technological limitations and to be consistent with sustainable development goals to put our planet back on the path towards sustainable development. In this context, the hydro-/solvothermal sintering (HSS) has recently emerged as a major opportunity to develop new and/or optimized materials that respond to today's scientific, technological and related socio-economic issues.

As in the case for the Cold Sintering Process (CSP), HSS is based on dissolution-precipitation mechanism induced by a pressure solution creep as a driving force for the densification but CSP and HSS differ from each other due to the balance between thermodynamics and kinetics that strongly influences the predominance of the involved elementary steps. In this thermodynamically controlled process, the hydro-/solvothermal conditions promote a strong enhancement of the reactivity at the interfaces and also represent a powerful lever to tune the nature of the precipitated phase. In this way HSS appears as chemically-driven at the interfaces.

All the potentialities of HSS will be presented mainly from the results obtained on silica used as a common theme to study the sintering of amorphous (silica) or metastable (alpha-quartz) materials, to design and fabricate 0-3 type functional nanocomposites. A description of the current understanding of chemical and mechanical-chemical mechanisms necessary for densification will be proposed. Finally, opportunities and challenges to expand the method more generically to other systems will be discussed and illustrated by recent results.

Molten salt shielded synthesis (MS3): a novel synthesis route for non-oxide ceramics in air

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Synthesis of non-oxide ceramics typically requires high temperature (> 1000 °C) due to their covalent atomic bonds, and protective atmosphere – commonly argon or vacuum – to avoid the spontaneous oxidation. Furthermore, conventional synthesis routes, such as solid state reaction (SSR) or self-propagating high temperature synthesis (SHS), produce a dense or porous ceramic block, which needs to be ground and milled into fine powder for further processing. The milling process introduces several complications for large-scale powder production, as contamination of the powders, high wear and operating costs with longer milling times. Additionally, industry and society are demanding novel sustainable processes, where less energy is consumed.

In this work, we will present a novel synthesis route for non-oxide ceramic powders, which is referenced as Molten Salt Shielded Synthesis or MS3. MS3 is carried out at lower temperatures than conventional synthesis routes, in air instead inert atmospheres, and does not require any milling step to obtain fine and loose powders. As a result, the process is environmentally friendly, easy and attractive for industry. MS3 is based on the encapsulation of the starting precursors in a salt (KBr) to prevent the oxidation, followed by a molten salt synthesis process at high temperature in air. Finally, the pellets are washed in hot water and filtered to obtain the final powder. Synthesis of different ternary transition metal compounds (MAX phases such as Ti_3SiC_2, Ti_2AlC and Cr_2AlC) will be shown, as well as the possibility for other non-oxide ceramics such as TiC.

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Osteoinduction and bone regeneration by synthetic biomaterials

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Intrinsically osteoinductive biomaterials have received interest as promising alternatives to autograft in treating large, clinically-relevant bone defects. A majority of osteoinductive biomaterials developed so far is based on calcium phosphates (CaPs). While the ability of osteoinductive CaPs to induce de novo bone formation and regenerate large bone defects has been proven in various studies, the clinical applicability of CaP-based bone graft substitutes is still limited by their brittleness, difficulty to control their degradation behavior and complex handling properties. In this lecture, recent attempts to improve some of these properties without compromising the osteoinductive potential will be discussed. Moreover, studies aiming to understand the interactions between osteoinductive biomaterials and the biological system at gene- and protein level, as well as in vivo will be presented. The role of chemical, physical and mechanical properties of the material in the process of osteoinduction will also be discussed as well as the approaches to use this fundamental knowledge to design improved bone graft substitutes.

Characterisation of phosphate bonding in high-temperature ceramics by solid state NMR

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The reaction sequence of phosphate-bonded high-temperature ceramics during bonding progress is not clearly predictable. Consequently, the phosphate components have an unspecific mode of action. This research results here give a detailed insight into phase transformations of phosphate binders within an Al_2O_3-$MgAl_2O_4$-ceramic after thermal treatment of up to T = 1500 °C using several inorganic (di)-hydrogen phosphates as binding agents. Via comprehensive solid-state NMR structural analyses, conversion processes from hydrogen orthophosphates to amorphous phases and poly- and metaphosphates to high temperature phases can be clarified. The phosphate binding activities mainly results from chemical and adhesive interactions of a crosslinked, three-dimensional aluminium phosphate network with the refractory body. Especially amorphous phases are identified to effectively impact the formation of a phosphate bond. At T > 800 °C, phosphates react with $MgAl_2O_4$ to form magnesium orthophosphates. Based on this understanding, phase formation can be directly correlated with developing properties, such as strength or resistance.

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3D printing with technical ceramics SiSiC in art & design

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Since 1996, the engineer, artist and university lecturer has been realizing artistic and design-related cooperations with industrial companies. Recently two projects for large format 3D printing with SiSiC were developed in close cooperation with the company Schunk Ingenieurkeramik GmbH. Specifics in the design work and the realization in the powder bed printing process are reflected by means of examples from this cooperation. The development of the required analog-digital process chain is shown, which initially takes place via steps such as 2D sketches, plastic models or 3D scans, digital designs and small plastic-printed models. Furthermore, necessities and special features of the powder bed printing process with the extremely hard silicon carbide and its further processing are shown. It shows that digital design and 3D printing open up exciting new design possibilities compared to analogue processes, but also imply some limitations. Experimentation, rethinking and rethinking are required in order to exploit and expand technical possibilities - a synergy effect for both project partners.

Microstructure and hydrothermal ageing of alumina-zirconia composites modified by laser engraving

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Laser technology is used for different applications such as joining and cutting as well as structuring and marking of surfaces. Orthopedic implants are marked by laser engraving to ensure product traceability according to ISO13485. In this study the influence of a solid-state laser on surface properties of two alumina-zirconia composites (ATZ; ZTA) is analyzed. The laser was used to mark the ceramic. A good visibility and readability as well as no negative influence on the properties and application performance of the ceramic is indispensable. Both ceramics were lased in two different states: as green and sintered body. Different laser parameters were used. The laser engraving was done by two commercially available marking stations with solid-state lasers made of Nd:YAG and Nd:YVO_4. The laser engravings were analyzed regarding the macrostructure (LSM), phase content (XRD), microstructure (SEM, FIB-Tomography, EDS) and ageing behavior. In the lased zone the color changed from white to brown-black. The higher the zirconia content and the higher the energy density the darker the color. Higher energy density also led to deeper engravings. Phase analysis showed a change in XRD pattern at the (101) tetragonal peak of the zirconia which might be linked to a lattice deformation or presence of a solid solution layer. The resistance to hydrothermal ageing wasn't much affected by the laser. SEM and FIB-tomography analyses showed a formation of slight surface cracks. Just below the surface a kind of solid solution layer of Al-Zr-O was detected. With XPS oxygen vacancies were found which might be responsible for the color change. Additional analyses like GIXRD, burst strength of lased hip heads as well as detailed XPS analyses are in progress.

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The use of humic substances as rheological additive

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In the course of the ZIM Project ZF4268907-AG7 (in cooperation with the company Humintech GmbH/DE), the effect of geogenic and processed humic substances on the processing properties in ceramic clays has been investigated. It has been shown that particularly sodium humate, produced by Humintech GmbH/DE, exhibits suitable properties. The achieved liquefaction of ceramic slurries is comparable with that obtained with commercially available liquefiers. In addition to the good liquefaction of ceramic raw materials and slurries, also an increase of the dry bending strength has been observed. This positive influence of the humic substances on the processing properties of ceramics could presumably be explained with the formation of stable clay-humus complexes. For better quality control, the new DIN EN ISO 19822 standard for the determination of humic fractions was tested and successfully established.

Manufacturing of SiC/SiC composites based on colloidal C-SiC slurries

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The demand for lightweight materials applied in aeronautics, space and gas turbines is increasing. Therefore, SiC-based Ceramic Matrix Composites (CMCs) with their outstanding thermo-mechanical performance and low density, are promising candidates compared to conventional Ni-based superalloys. Colloidal-based SiC/SiC manufacturing routes are described, which are based on carbon and silicon carbide containing powder slurries and thereby avoid the costly and time-consuming pyrolysis step or gas deposition steps of conventional SiC/SiC processing routes. The development of an aqueous C-SiC slurry is briefly explained and four different manufacturing routes, namely two prepreg and two direct infiltration routes, will be addressed and compared. Each route can be divided into the following three-steps. The first step is a multilayer coating on the SiC-fibers to enable the weak interface concept and hence achieving a damage tolerant fracture behavior of the resulting composite. Two different coating systems, applied by atomic layer deposition (ALD) or chemical vapor deposition (CVD) were used. In the second step, the fabrics are impregnated with the slurry followed by a consolidation, which differs for each manufacturing route. Finally, the greenbody is infiltrated with molten silicon using the LSI- process (liquid silicon infiltration) to obtain a dense and homogeneous SiC/SiC composite. The influence of the fiber-coating and the manufacturing route on the microstructure and the mechanical properties of the SiC/SiC will be discussed in detail.

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Thermoelectric properties of composite ceramics based on $Ca_3Co_4O_9$ and large plate-like oxides

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Energy harvesting is a topic with increasing importance due to limited resources. Especially the vast amount of wasted thermal energy in multiple processes makes the conversion into electrical energy via thermoelectric generators an attractive option. However, the efficiency and performance of the latest thermoelectric materials demand further research. Regarding high-temperature applications, an excellent chemical stability in air as given by oxide-based ceramics is a key feature. Within the material class of thermoelectric oxides, the $Ca_3Co_4O_9$ is well-known for its high power factor combined with a low thermal conductivity. To enhance the thermoelectric properties of $Ca_3Co_4O_9$, we investigated the influence of individually adding perovskite-type oxides such as the mixed ionic-electronic conductor $La_2NiO_{4+}\delta$ or the large bandgap semiconductor $Na_2Ca_2Nb_4O_{13}$ to obtain sintered composite ceramics.

For the preparation of the perovskite-type oxides, molten-flux syntheses were carried out, as it allowed control over the crystal shape. All three oxides are characterized by layered structures and therefore anisotropic transport properties. To benefit from the preferred orientation in a textured ceramic, the added oxides were synthesized as large anisotropic plate-like crystals in the size of several microns, which preferentially orientate when the green body is pressed uniaxially. An extensive analysis of the sintered composite ceramics was performed and textured ceramics with enhanced thermoelectric properties were obtained.

Texturing 3D-printed alumina ceramics through templated grain growth

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Texturing the microstructure may enhance the structural and/or functional properties of polycrystalline ceramics. For instance, nacre-like alumina has been fabricated through different processing routes (e.g. tape casting), using Templated Grain Growth (TGG) as a strategy to obtain high degree "morphological" and "crystallographic" texture. However, a limitation of these methods is the rather simple geometries that can be fabricated, e.g. discs or plates. Additive manufacturing techniques have proved successful in fabricating 3D monolithic structural and functional ceramics of complex geometries. In this work, we explore the possibility of fabricating textured microstructures using the 3D-printing based on lithography-based-ceramic-manufacturing (LCM). A commercially available α-alumina slurry was employed to fabricate the equiaxed alumina (EA) reference material. For textured alumina (TA) samples, the slurry was modified with α-alumina templates. TGG during sintering led to textured grains with preferential crystallographic orientation. Density, microstructure, and the effect of sintering time on grain growth were analysed in TA and compared to EA samples. The use of LCM-process for template alignment together with TGG during sintering for grain growth resulted in a high degree of grain orientation in alumina, reaching a Lotgering Factor of approximately 80%, with relatively low porosity (approx. 7%). The alignment of templates was facilitated through the torque occurring when the slurry is distributed after every printing step as well as the immersion of the building platform into the slurry bed. This approach opens the path for 3D printing textured ceramics with complex architectures.

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Fabrication of composite cathodes for all-solid-state lithium batteries

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All-solid-state lithium batteries are promising candidates for futures energy storage systems, promising high energy and power densities while being inherently safe. However, the ceramic processing of oxide and phosphate based batteries and cell manufacturing using Li-metal anodes still poses several challenges. For co-sintering of mixed cathodes, the focus is on maximization of thicknesses and minimization of secondary phase formation to achieve the desired properties (high conductivity, low interface resistance, high active material loading, etc.). For Component manufacturing, tape casting is a promising technique for scalable and low-cost production of ceramic sheets, but the process needs to be optimized for each material and materials combination. The sintered foils should be dense, flat, mechanically stable and electrochemically active.
We report on the manufacturing of both oxide (garnet) and phosphate (NaSICON) based all-solid-state batteries with co-sintered mixed cathodes and Li metal anodes. On the one hand, co sintered Ta-substituted LLZ as solid electrolyte is combined with $LiCoO_2$ obtaining very high areal capacities of 1.63 mAh/cm^2 with an active material loading of 13 mg/cm^2. On the other hand, Tape-casted and co-sintered cathodes composed of $Li_{1.5}Al_{0.5}Ti_{1.5}(PO_4)_3$ and $LiCoPO_4$ or $LiFePO_4$, were fabricated having thickness between 120 to 300 µm. A thin layer of LIPON electrolyte was deposited on the top of tape casted cathodes using physical vapor deposition to ensure chemical and electrochemical compatibility with metallic Li as anode.

Phase and microstructure evolution of polymer-derived SiZr(B)CN ceramic nanocomposites

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A zirconium and a zirconium / boron containing single-source precursor were synthesized via modification of a commercially available polysilazane with tetrakis (dimethylamido) zirconium (IV) (TDMAZ) as well as with both TDMAZ and borane dimethyl sulfide complex, respectively. The transformation of the precursors into SiZrCN and SiZrBCN ceramics as well as the thermal evolution of their phase composition and microstructure were studied. Interestingly, the as-prepared SiZrBCN ceramic was single-phasic and fully featureless; whereas SiZrCN exhibited the presence of amorphous ZrC_xN_y particles within an amorphous SiZrCN matrix. Heat treatment at higher temperatures induced crystallization processes in both prepared ceramics. Thus, at temperatures beyond 1500 °C, cubic ZrC_xN_y, β-Si_3N_4 as well as β-SiC were generated. It has been shown that the incorporation of B into SiZrCN suppresses the crystallization of ZrC_xN_y and impeded the reaction of SiN_x with C. The liquid single-source precursors presented here were used to coat ZrB_2 powders. Subsequent cross-linking, ceramization and hot-pressing of the coated UHTC powders at 1800 °C resulted in dense monolithic ZrB_2/SiZr(B)CN composites, indicating SiZr((B)CN being an efficient additive system for the densification of ZrB_2.

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Development of solid electrolyte cell by spray coating

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The European KEROGREEN project aims to develop sustainable aircraft kerosene based on water and CO_2 powered by renewable electricity. An important step in this production process is the oxygen separation after the plasmolysis of CO_2 to create a CO stream for Fischer-Tropsch synthesis. The oxygen separator is similar to a solid oxide electrolyte cell and it consists of a plasma electrode, an electrolyte and an oxygen electrode. In this work, the electrode layers are applied by spray coating on yttria-stabilized zirconia (YSZ) planar substrates, which serve as the electrolyte layer. For the electrodes the most promising La-based perovskite materials were selected based on their conductivity and ability to suppress the CO to CO_2 back reaction at the plasma side, and produced by CerPoTech. The coating parameters such as atomization pressure and coating speed were optimized to obtain uniform coatings and to control the thickness. The suspension formulation was adjusted to improve the adhesion and the quality of the coating. Profilometry and microscopy were used to determine the coating thickness and porosity. Furthermore, electrochemical impedance spectroscopy were carried out.

Several YSZ discs were successfully coated with an oxygen and plasma electrode layer and then thermally treated up to 1100 °C for 4h. The coatings show a good adhesion to the YSZ discs and look rather uniform. Both electrode layers are porous and are about 10 µm thick. The total electrodes impedance determined at 800 °C in dry air was reduced to 10 Ohm by using an extra interlayer to prevent interactions.

Influence of the microstructural characteristics of boron nitride coatings on oxidation/corrosion resistance and mechanical properties of SiC/BN/SiC composites

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In the latest generation of ceramic matric composites (CMCs) that are being considered for aeronautical applications, BN is becoming the main interphase material used between SiC-based fibres and matrices. The oxidation/corrosion resistance under moist air at high temperature and the mechanical behaviour at room temperature of model SiC/BN/SiC samples with different microstructures and geometries were investigated.

In the case of a thin CVD BN coating with a typical interphase thickness of 0.5 µm, the oxidation resistance is not very dependent on the microstructure (and therefore on the structural anisotropy) of BN. At 800°C, a slowdown in oxidation is observed due to the formation of a protective borosilicate glass in the confined space between the two SiC layers. However, this protection becomes limited at 1000°C.

Composite macroscopic behaviours obtained from tensile and shear tests were correlated with the interfacial bond properties obtained from push-out tests. They depend on the BN interphase thickness and are, in contrast to oxidation, highly affected by the increase in its degree of crystallization. The location of the debonding in the interfacial complex during material mechanical damaging and its mechanisms of ruin were determined by scanning and transmission electron microscopy. Obtaining a gradient of the interphase structure controlled by temperature variation during deposition allows, for instance, a debonding in the middle of the interphase. The improvement and/or protection of this interphase material for CMCs therefore remains both necessary and possible.

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Sintering of two phase structures – challenges and opportunities

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For advanced ceramics composites, precise microstructural tailoring is the most essential prerequisite for successful commercial use. This holds particularly for components made out of composites with complex hierarchical structure and high demands of mechanical and functional performance. Although huge improvements are achieved in the past with respect to knowledge as well as technology, precise microstructural control during "conventional" sintering (=without applied pressure and/or electric fields) is still a challenge as several processes act typically simultaneously. In the presentation, some strategies aiming at reliable components will be presented based on three examples of recent research, (i) interpenetrating metal-ceramic composites (ii) fiber reinforced Ox-Ox composites, and (iii) photonic structures based on 3 dimensional ordered porosity. Thereby, the focus will be on alumina or alumina-based compounds. The results are reasoned considering theoretical predictions as well as the sintering activities of the constituents. Additional emphasis will be given to Discrete Element Modelling (DEM) taking thereby into account the particulate structure - in particular the particle-particle and particle- pore coordination. Finally, some outlook will be given addressing future developments.

Microwave sintering of additively manufactured yttria stabilised zirconia

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Stereolithography (SLA) is an additive manufacturing technique where a 3D object is printed by adding material layer-by-layer in a vat. Each layer is selectively photocured by a UV beam, which allows the manufacturing of complex-shaped parts with high-resolution. In the case of ceramics, SLA leads to high density green parts compared to other additive manufacturing methods. However, as SLA is an indirect additive manufacturing technique, green parts must be thermally debinded and sintered. Usually, this second processing step is achieved in conventional furnaces and is time-consuming. Besides that, Microwave (MW) sintering is a quick and efficient way to sinter ceramics, which allows the achievement of a fine microstructure due to short thermal treatment time, which usually leads to better mechanical properties in comparison with conventional sintering.

In this study, yttria stabilized zirconia (YSZ; Lithoz LithaCon 3Y 230) bars (3 × 4 × 20 mm) were printed using a Lithoz CeraFab 8500 machine and the samples were conventionally debinded. Samples were subjected to MW sintering and compared to conventionally sintered samples. Conventionally sintered (2 h @ 1450 °C) YSZ led to 98.6 % relative density parts. The elastic modulus, the Vickers hardness and the 3 points flexion resistance were 243 GPa, 14.5 GPa and 1280 MPa, respectively. Similar values were obtained for MW sintered (5 min @ 1430 °C) samples. The effect of sample preparation (grinding, polishing) was also investigated in term of flexural strength. Finally, it is shown that coupling SLA and MW sintering led to a 24% time-saving without weakening the mechanical properties. Therefore, this approach could be a promising way to produce customized ceramics in short time.

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Cool-SPS: opportunities for fragile functional materials and beyond

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The use of materials requires the control of their shaping (dense or porous materials, spherical or anisotropic particles…), frequently achieved through the elaboration of ceramics by conventional, high temperature sintering. Ferroic materials are especially concerned, being mostly used as ceramics, the quality of which is crucial. In this view, Spark Plasma Sintering (SPS) is a particularly efficient densification method that allows for the elaboration of many types of high-quality ceramics, and has proven extremely versatile concerning the chemical nature of the processed materials. This contribution aims at highlighting the Cool-SPS approach, which allows efficient sintering at low temperature by balancing SPS processing parameters (pressure,…), and mobilizing chemical reactivity. The proof of concept of Cool-SPS will be presented, highlighting the expected and unexpected benefits of the approach, and the complexity of some underlying sintering mechanisms. Brief accounts of the densification of classic ferroelectrics by Cool-SPS, and of the elaboration of functional molecular ceramics will follow. Additional examples (hydrates and more...) will highlight the broad potential of Cool-SPS, yet to be fully explored. To conclude, the opportunity to develop more sustainable functional materials and processes associated with Cool-SPS will be discussed.

Ceramic materials for tribological applications - state of the art and new challenges

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A quite large part of the technical applications of structural ceramic materials and components are related to their tribological properties, as high-performance ceramics are generally very resistant even under corrosive conditions and at high temperatures. Whereas until a few years ago, wear resistance and the possible long service life of ceramic components were the most important aspect, today possibilities of friction minimization are considered to be much more more important, since the reduction of friction losses directly improves the energy efficiency in mechanically moving components.

This paper discusses the tribological objectives and the general tribological requirements for ceramic materials from today's point of view, and shows the results of own current work as well as some published results, which show the possibilities for reducing friction with ceramic materials – down to superlubricity (friction coefficients <0.01). This opens up new perspectives for current applications and future applications for ceramic materials.

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Processing of advanced porous materials for energy & environmental applications

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Advanced porous materials and structures are used in different energy and environmental applications, such as in gas separation & storage, air filtration, energy conversion & storage or water treatment. The choice of materials and smart structuring into multilayer or into hierarchical porous structures is crucial to achieve high performance in gas separation and storage. Here, we report the processing of asymmetric ceramic oxygen transport membranes (OTMs) and of hierarchical porous materials for upgrading & storage of biogas in swing adsorption processes. The fabrication of planar or tubular asymmetric OTMs at DTU Energy is based on the optimization of the shaping of support, membrane and catalytic layers, their multi-layering and co-firing to achieve membranes with improved microstructures and performance. Examples for the optimization of these processes include the use of of sacrificial templates (pore former) in extrusion, phase inversion tape casting and optical dilatometry.

The upgrading of biogas, which is the separation of CO_2 from CH_4, and the subsequent storage of biomethane can be achieved by swing adsorption processes. New concepts for the structuring of zeolites and metal organic frameworks (MOFs) into hierarchical porous nanofiber structures have been evaluated, utilizing electrospinning and in-situ growth techniques. Correlations between the processing, the microstructure of the resulting hierarchical porous structures and some key performance parameters, such as compression strength, CO_2 and CH_4 uptake, selectivity and mass transfer properties will be presented.

Modelling of the highly nonlinear electrical behaviour of zinc oxide varistors

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ZnO varistors (variable resistors) are among the most used surge arresters in the electronics industry today. They protect sensitive electronic components from short-term overvoltage events which can occur, for example, during switch-on or switch-off processes, electrostatic discharges, lightning strikes, etc. The key property required for such applications is the highly nonlinear electrical behaviour of the grain boundaries in the polycrystalline ZnO structure. The grain boundaries block the current flow until a critical voltage is reached. By exceeding this critical voltage, the grain boundaries (and thus the varistor) become conductive within nanoseconds. This effect is reversible and can be used to safely discharge overvoltages. Even though modern ZnO varistors are very reliable, they have some disadvantages. With time and load, they age and conduct leakage currents or can cause a short circuit after absorbing too much energy. At the Institut für Struktur- und Funktionskeramik at the University of Leoben, unique measurement tools and simulation techniques have been established and combined to study ZnO varistors on the microscopic scale and to predict and understand their electrical behaviour. These methods include a micro 4-point probe setup to measure currents across individual grain boundaries, or micro lock-in infrared thermography to image very small heat sources (current paths), and full 3D models of the ZnO microstructure to calculate local current and power densities. In this talk, it is shown how the microscopic current-voltage characteristics of ZnO grain boundaries can be used in a simulation to reproduce the macroscopic behaviour of real varistors, allowing a deeper understanding of the fundamental processes.

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A new automotive application for ceramic matrix composites (CMC): C/C-SiC based piston rings for internal combustion engines (IC-engine)

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Alternative energy and the transition away from fossil fuels is one of the core subjects of current politics. In the automotive industry the internal combustion engine, however, remains the most commonly used power unit. In terms of improving its environmental compatibility, the current research goal is to increase its efficiency and service life while maintaining or even reducing the fuel consumption.

The DLR develops a ceramic fiber-reinforced piston ring, which should contribute to fuel savings and reduced piston wear. In order to investigate fundamental questions of feasibility and functionality, piston ring prototypes made of C/C-SiC (carbon fiber reinforced silicon carbide) were developed in cooperation with the DLR Institute of Structures and Design and DLR Institute of Vehicle Concepts. For a ceramic piston ring the mounting is one of the most restricting requirements, as the material is usually not flexible and fiber reinforcement has to be carefully adjusted in order to provide elastic behavior. Within the project, variants of C/C-SiC with different preforming technology were developed. Weaving-, winding- and tailored fiber placement-technologies were used for preform manufacturing. The mechanical examination of the samples was carried out according to ASTM C1323 - 16 for ceramic C-rings and the results were promising.

In addition, the work provides further insights into the expected running behavior of the ceramic piston rings and how economic production can be achieved. Further research now aims at examining the effects on the energy consumption and service life when utilized in IC-engines.

Production of fine-grained iron oxide particles by pulsation reactor technology and investigation of the dispersing character of the produced particles

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It is well known that each technical field of application requires specially selected materials with defined properties. Iron oxide particles are widely used materials for many different applications, such as pigment for paints and colors, coating on magnetic tapes, catalyst or main component in energy storage systems. 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 iron oxide particles with specific properties have been thermally synthesized by the pulsation reactor. This work describes the specifications of the applied pulsation reactor as well as the characteristic properties of the iron oxide particles produced by using the pulsation reactor. During the synthesis of iron oxide in the pulsation reactor, the effect of characteristic parameters of the process, such as temperature, residence time and reactor configuration on the physico-chemical characteristics of the produced particles were investigated applying different analytical methods. The temperature of the reactor, as one of the main parameters of the process, varied from 500°C to 900°C. This allowed the adjustment of the specific surface area (SSA) of the resulting Fe_2O_3 up to 37 m^2/g. The high SSA results with a very fine primary particles size of about 30 nm could be realized. According to this work, it was also concluded that the PR technology could reach a wide range of iron oxide powders with tailored properties in a continuous process. The effect of properties of the different precursors as well as process parameter of the reactor on the dispersing behavior of the prepared particles in aqueous media was tested and optimized.

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Fiber-matrix interfacial characterization of composite materials: investigation by single fiber push-out tests

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The mechanical properties of composite materials, e.g. the damage tolerant fracture behavior of ceramic matrix composites, relate to the fiber-matrix interfacial properties. These strongly depend on the fracture toughness of the fibers and the interface as well as on the surface properties of the fibers. The micromechanical single fiber pushout test represents a versatile method to measure these values. A rigid indenter-tip axially loads an individual fiber, which is oriented perpendicular to the surface of a thin composite sample. This induces fiber matrix debonding and displacement of the loaded fiber relative to the surrounding matrix. Evaluation of the load-displacement curve allows extraction of interfacial parameters, i.e. the interfacial fracture toughness, which characterize the bonding of fibers and matrix. It is shown that fiber surface treatment as anodic oxidation and sizing strongly influences the interfacial fracture toughness in carbon fiber reinforced epoxy resins. This is also measured in carbon fiber reinforced phenolic resin, which represents the intermediate state of C/C-SiC. The effects of interfacial toughness in the polymer state on the overall properties of the final C/C-SiC material will be discussed.

Antiferroelectric-ferroelectric phase transitions in sodium niobate

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Antiferroelectric ceramics exhibit unique electrical properties, which originate from the complex antiparallel arrangement of the local dipoles. Typically, a ferroelectric phase with a comparable free energy to the antiferroelectric phase exists and it can be induced by different external stimuli (electric field, temperature, stress). Compositions in which this transition is reversible exhibit characteristic double polarization loops and could be used for high-energy storage capacitors or electrocaloric cooling. Besides the well-known lead-based compositions, several lead-free systems based on sodium niobate ($NaNbO_3$) were recently reported.
This work explores the properties of the antiferroelectric-ferroelectric phase transition in the model system $NaNbO_3$. In polycrystalline samples this transition can be induced by grain size or external electric field. Structural evidence for temperature- and grain-size-induced transitions is presented and related to macroscopic properties, such as dielectric permittivity and polarization. The second part explores the electric-field-induced transition. The transition into the ferroelectric phase is confirmed by X-ray diffraction and piezoelectric measurement, while the amount of the phase is determined by ^{23}Na Nuclear Magnetic Resonance spectroscopy. The behaviour is found to be irreversible and the phase transition is decoupled from the polarization switching process.

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Properties of coarse-grained Nb-Al_2O_3 and Ta-Al_2O_3 refractory composite materials for high temperature applications

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Over the last decades high-temperature materials have been investigated to a very large extend. This is caused by the increasing demand for long-term stable materials, for instance in metallurgical industry and energy sector, to improve performance and efficiency. Different attempts have been made to fulfil the requirements for different applications. In this context, coarse-grained composite materials of refractory metals and ceramics seem to be a promising option for such applications due to their high melting points and advantageous combination of properties. The coarse-grained attempt is considered to improve high-temperature creep resistance and thermal shock behaviour. Furthermore, recycling potential of these new materials is also a key target, since responsible use of resources is an important issue currently gaining more and more attention.

This study deals with the properties of a new generation of non-shrinking coarse-grained refractory Nb- and Ta-Al_2O_3 composites based on a two-step processing of pre-synthesized coarse grains of Niobium, Tantalum and alumina from fine-grained starting powders. To get a better understanding of these materials, first investigations were conducted to find out about the relation between starting powder compositions, processing parameters and the resulting microstructure, as well as their influence on electrical and mechanical properties.

Sustained delivery of antimicrobial and anticancer compounds via biodegradable hollow mesoporous silica capsules

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Hollow mesoporous silica capsules (HMSC) have aroused tremendous interest in the field of drug delivery due to their unique properties such as high biocompatibility, large surface areas and high loading capacities due to a protected hollow core. In this work, HMSC were synthesized using a hard iron oxide template which was coated with silica through sol-gel synthesis. Afterwards the iron oxide core was removed by acidic leaching. For testing their capability as drug delivery vehicle, a hydrophilic antibiotic (Ciprofloxacin) and a hydrophobic anticancer compound (Curcumin) were loaded and a pH dependent release at 37°C was monitored via UV-Vis spectroscopy. Ciprofloxacin-loaded HMSC were also tested towards E.coli revealing a complete growth inhibition over 18 hours. In addition, the intensive use of all kinds of antibiotics has led to an increase in multi-drug resistant bacteria and shows the necessity for alternative treatment options against bacterial infections. Therefore, metallic nanoparticles such as Cu, Ag and ZnO were incorporated into HMSC through the reduction of their metal salts. The formation of rattle-type metal HMSC was proven by TEM. To determine the controlled leaching of metal ions, free Ag, Cu and Zn ions were complexed with dithizone and quantified via UV-Vis analyses. Moreover, INT assays revealed that all particle types exhibit a strong antimicrobial effect against B. subtilis as well as E. coli, demonstrating their promising potential as antibiotic alternative in the future.

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Towards the control of nanoparticle shaping: Electrical field parameter analysis and particle packing optimization

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Since the beginning of the 19th century, the motion of electrically charged colloidal particles in an electrical field has been known. Particularly in the shaping of nanoscale particles, electrophoretic deposition (EPD) is used to produce homogeneous, defect-free green bodies with high theoretical density. Due to the independence of the particle size from the particle velocity and thus the deposition rate, the EPD has great potential especially for shaping nano-sized particulate systems. In contrast, in conventional molding processes such as powder pressing or slip casting, the small particle size and the associated very large particle surface area result in lower green densities and longer process times.

The presentation will give an overview of the updated control parameters of creating compact, nanoparticulate shaped bodies using EPD. Thereby the particle packing is strongly dependent on the electrical field parameters and the colloidal suspension preparations. By using the membrane method, the EPD process can be designed variably. The experimental results show the potential of the shaping method for structure generation down to extremely small scales and summarize some promising and upcoming applications in the field of ceramic nanotechnology.

Optimization of the filling shoe design for finely dispersed, ceramic granules in die filling

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Process speed and component quality in uniaxial dry pressing are essentially affected by the process step of die filling. Using analytical methods, Fraunhofer IKTS Dresden was able to identify the influencing factors on the granulate side as well as the influences of die geometry and process control on the dosing behavior of ceramic granules and the filling result. The investigations were carried out using a practice-oriented filling demonstrator "FlowD" which was developed at the Fraunhofer IKTS Dresden. The air replacement has a significant influence on the dosing behavior during die filling of ceramic granules and require closer analysis. For visualization and evaluation of the granulate-air interaction, a modular fill shoe was integrated into the demonstrator. It is equipped with various internals for deaeration of the cavity or the fill shoe, adjustment of the filling level in the fill shoe and for fluidizing the granulate bed. High-speed and computer tomographic images are used to evaluate filling characteristics, the settling behavior of the granules in the die, and the resulting bulk structure. For ceramic model granules with defined filling and flow properties, filling phenomena caused by venting of the cavity or fill shoe and fluidization of the granule bed are demonstrated. The use of custom-tailored internals led to a significant increase in the final filling degree δ_{max} for all the granules investigated, while keeping the process time constant. In addition, the potential of combining several methods to achieve optimum filling results was demonstrated.

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Multi material additive manufacturing of ceramics by material jetting

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Over the last years, additive manufacturing gained more and more importance. The reason for this is the possibility to produce complex geometries with lesser material loss and high precision as well as surface quality. Additionally, additive manufacturing enables the printing of multi-materials. Multi-material additive manufacturing allows the creation of parts with spatially varying material properties. Thus, the properties can be designed according to the functionality requirements during the application. In this presentation, a material jetting process based on the ejection of aqueous ceramic suspensions by thermal inkjet printheads is presented to fabricate ceramic multi-material parts. The key factors during suspension development and the requirements to print multiple ceramic suspensions simultaneously are shown and demonstrated on alumina-zirconia composites with complex material transitions. SEM analyses of these composites' microstructure lead to the conclusion that the material jetting process is capable of producing dense and defect-free multi-material parts with material variations on a drop-wise scale. In the future, a voxel-based design approach should be applied to control the material composition in all three dimensions to use the full potential of material jetting.

Sample preparation of nano-powders for particle size determination

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The use of increasingly finer starting powders up to nanopowders can also be observed in the field of ceramics. Their advantages are, for example, lower activation energy, an increase in strength of the sintered products or unique optical properties. However, handling and characterization of ultrafine powders is much more difficult. The main reason for this is the very high adhesive forces both between the nanoparticles themselves and when in contact with other surfaces. Therefore, submicron- and even more so nano-particles tend to agglomerate and their separation into primary particles during sample preparation prior to particle sizing is a major challenge. A representative measurement sample is obtained only when it no longer contains agglomerates. The evaluation of the dispersion process and a decision on whether it was successful thus gains in importance for the reliability of the measurement results of particle sizing.

The lecture shows possible procedures by means of examples and gives hints on possible sources of error in the dispersion of ceramic submicron- and nano-powders. It is shown that successful granulometric characterisation of fine powders requires both an improved dispersion technique and very often an effective combination of two or more measurement methods.

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Thermomechanically loaded O-CMC reinforced metal pipes - from laboratory scale towards power plant components

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Metal components, e.g. steel pipes are very common in power plants and can resist high temperatures up to 700 °C and internal pressures up to 350 bar. However, their lifetime is limited due to creep deformation after > 100.000 h in service. In order to overcome this drawback, a creep resistant oxide CMC (Ceramic Matrix Composites) jacket was applied resp. wound around this pipe. The CMC can carry the creep induced loads and a compressible interphase compensates the CTE mismatch between the metal pipe and the ceramic jacket. Early laboratory studies showed that the lifetime of this metal-ceramic-pipe was increased by the factor of more than four. Within this work an alumina fiber reinforced CMC with siloxane-based Si-C-O matrix was further studied at application related loads within the laboratory and in a power plant. All experiments in the laboratory runs between 500° C and 625 °C with partially internal steam pressures up to 290 bar, in order to check a possible lifetime extension of jacketed metal pipes. During the extended laboratory tests > 5000 hours, the desired lifetime extension was confirmed again. Without a ceramic jacket, the metallic component fails after less than 1000 h. Investigations of the microstructure show the long-term compatibility of the siloxan-based SiO_x-matrix with the alumina fibers. After transferring the knowledge to a CMC jacket on a steel pipe (Ø > 300 mm) in a power plant, the CMC was studied after five years under loads up to 530 °C and 255 bar. No significant degradation and sintering effects between the fiber and the matrix are detected in SEM and XRD, which means, no embrittlement occurs.

Electrochemical properties of silicon carbide-bonded diamond materials

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Silicon carbide (SiC)-bonded diamond materials are developed at Fraunhofer IKTS, being a novel class of super-hard and wear resistant ceramics. A relatively simple way to fabricate these compound materials is by the silicon infiltration of diamond preforms. This process route also allows the adjustment of the microstructure of the material as well as of its conductive properties. However, the electrochemical behavior of these innovative SiC-diamond compounds is not yet well investigated. These properties, in turn, are of high practical interest, for example for EDM-cutting, concerning the corrosion behavior or applications as wear-resistant electrode in technical processes. Comprehensibly, the electrochemical properties are strongly affected by the microstructure and conductivity of the SiC-diamond materials. In this work the authors present the preparation of SiC-bonded diamond materials with varying electrical properties, using silicon of different purities as well as of boron-doped and non-doped diamonds. The compound materials were investigated by cyclovoltammetry, potentiostatic experiments and electrochemical impedance spectroscopy in aqueous electrolytes with different pH values. Chosen results of these electrochemical investigations are presented and related to the microstructure of the according specimen.

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Grain size dependence of electric field induced strain in barium titanate

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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. Even though, for a continuous miniaturization of dielectric devices the grain size has to be reduced. The fundamental mechanisms behind the electromechanical behavior are still not fully understood, but it is known that BT exhibits excellent electromechanical properties with a large electric field induced strain. Therefore, with proper tailoring it might be interesting for actuator applications.

In this work, the influence of the grain size distribution in dense BT on the piezoelectric response was investigated. Several different approaches were used to achieve a broad range of grain sizes: Including, fine grained BT powder for small grain sizes in the sub-µm range, two step sintering, quenched sintering, and hot isostatic pressing. As a result, the grain sizes vary over three orders of magnitude.

The microstructure was characterized using electron microscopy in order to determine the average grain size. The electromechanical behaviour was characterized using a commercial system equipped with an interferometer to measure the polarisation and strain hystereses. The response of the crystal structure to an applied electric field was determined with ***in situ*** synchrotron diffraction. Our results show that the electromechanical behaviour varies substantially with varying grain size. A sophisticated texture analysis using the STRAP method allows to understand the electric field induced response. The results show a behaviour that deviates from that previously described in the literature.

Chemical expansion: a challenge for processing and operation of high-temperature solid oxide cells (SOC)

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For functional oxide ceramics, the loss of lattice oxygen leads to a lattice expansion that is commonly called "chemical expansion". A coefficient of chemical expansion (CCE) can be defined analogous to the coefficient of thermal expansion (CTE) as the change of lattice parameter a with the change in oxygen deficiency δ. Chemical expansion is particularly notable for functional oxides used for oxygen exchange applications such as membranes for gas separation or air electrodes for solid oxide cells (SOC), since a high oxygen surface exchange coefficient is linked to a weak bond between the metal cations and the oxide anions.

The challenges associated to processing and operating multilayered structures (such as SOCs) that include materials showing chemical expansion are discussed on the basis of two relevant materials classes: ceria and cobaltites. Doped ceria crystallizes in the fluorite structure and is primarily used for diffusion barrier layers and electrolytes, whereas cobaltites such as $La_{1-x}Sr_xCo_{O3-\delta}$ are used as air electrodes due to their high mixed ionic-electronic conductivity and rapid oxygen surface exchange kinetics. Relevant cases where the materials show chemical expansion will be discussed for both material classes, including electro-chemo-mechanical expansion for ceria as an electrolyte material. The restraints on the processing or operation conditions arising from the expansion mismatch between different layers will be emphasized.

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Influence of cooling lubricants during milling of ceramic matrix composites

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Ceramic Matric Composites (CMCs) are a new generation of lightweight and high thermostable materials designed to overcome the limitations of monolithic ceramics. As their use in technical applications increases, the need for a more effective processing arises. A few studies have been published so far on the influence of mechanical machining on component quality. Majority focus on parameters such as cutting speed and feed rate. Little is known, though, about the effect of cooling lubricants. To this end, the present work investigates the influence of different cooling/ lubrication techniques on specific quality and process parameters when milling CMCs. The cutting forces of the process, as well as the resulting surface roughness were examined. Thermogravimetric analysis (TGA) were additionally carried out to evaluate the content of cutting fluid residues in the workpiece after machining. The study was performed on three different fiber reinforced ceramics: C/C-SiC, SiC/SiC, Al_2O_3/Al_2O_3. The cooling lubricants for the experiment include a water-miscible (wmb) cutting fluid emulsion— good cooling effect — and a non-water- miscible (nwmb) cutting oil— good lubricating effect —. The wmb cutting fluid was supplied through flood cooling system, while the nwmb cutting oil through minimum quantity lubrication (MQL). As third case study, dry machining was chosen. The tool used was a polycrystalline diamond milling cutter specially designed for machining of CMCs. The results show that lubrication provides no clear benefits over dry machining when milling CMCs, but cooling is highly recommendable since cutting forces and surface topography are improved. TGA reveal that those CMCs with higher porosity show more cutting fluid residues.

Influence of laser surface texturing on the flexural strength of Al_2O_3 and Si_3N_4

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Laser surface texturing (LST) has been well known for many years as an effective method to improve the tribological properties of high-performance ceramic components. However, its influence on the flexural strength of these ceramics were rarely investigated. The first objective of this work is to determine the flexural strength of ceramic samples with polished and laser-structured surfaces. In addition, this work aims to propose the potential explanations for the strengths difference among various surface conditions. Two kinds of high-performance ceramics, Al_2O_3 (Degussit AL23) and Si_3N_4 (SN-HP), were studied in this work. Provided as polished plates, the surfaces of ceramic plates were laser-structured with same micro-dimples but in different densities. Afterwards, the polished and laser-structured ceramic plates were laser-cut into disc-shaped samples. After carrying out of the biaxial ball-on-3-balls bending test, the flexural strength of Al_2O_3 and Si_3N_4 with different surface conditions were determined. Furthermore, the Weibull theory was applied to analyze the strength distribution of different surface conditions. The results show that LST helps to increase the Weibull modulus as well as the flexural strength of both ceramics. Observing the surface of ceramic samples using SEM (scanning electron microscopy), it was found LST removed pores beneath the tensile stressed surface. Moreover, compressive residual stress was found in the laser-structured ceramic surface by applying XRD (X-ray diffraction) measurement. This indicates LST is a potential way to locally improve the reliability of ceramics.

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Strategies to optimize thermoelectric energy conversion of a TMLTEG based on substituted calcium manganite

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Thermoelectric generators (TEG) convert waste heat into electric energy and may be applied to power self-sustaining sensors and transmitters in sensor networks. Commercially available TEG are based on doped Bi_2Te_3 or PbTe and are predominantly assembled in manufactory. Compared to this, transversal multilayer thermoelectric generators (TMLTEG) consist of a cost-efficient, nontoxic and environment-friendly oxide ceramic and a small amount of a metal. The device is resistant to harsh environmental conditions (high T, high or low $p(O_2)$) and is, as are all TEG, maintenance-free. Applying the ceramic multilayer technology, TMLTEG can be manufactured as monolithic devices in large quantity. This may create advantages in regard to the price of a TEG. Though, power output and energy conversion efficiency of the device must be increased.

Substituted $Ca_{1-x}A_xMn_{1-y}M_yO_{3-\delta}$ is a potential candidate for use in TMLTEG. It was used exemplarily to investigate different strategies to optimize thermoelectric energy conversion efficiency of thermoelectric materials. The impact of ceramic powder preparation, sintering conditions, substitution and preparation of composite materials on the thermoelectric properties of the calcium-manganite will be presented. Suitability of the applied strategies are assessed with regard to thermoelectric performance increase of the ceramic material. Furthermore, the experimental results are discussed along with parameters of a TMLTEG computed by 3D FEM-simulations and conclusions for further optimization of the oxide ceramic are drawn.

The ball-on-three-balls test: New developments and comparison with other biaxial strength tests for ceramics

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The strength of ceramic products that are available as (thin) discs or plates is preferably measured using biaxial strength tests. Several methods have been proposed for this purpose. For the Ball-on-three-Balls (B3B) test, specimens with 4 ≤ D/t ≤ 20 are positioned on three balls which are free to roll and loaded by a fourth ball. For the calculation of the maximum stress on the tensile side of the specimen results of FEM analyses and a fitted analytical expression is available for many specimen size variations. In the standardized Ring-on-Ring test, specimens with a diameter to thickness ratio D/t ≈10 are supported on a ring and loaded coaxially with a smaller ring. An even load distribution under the loading ring is ensured by using compliant interlayers between loading ring and specimen. For the determination of various dental ceramics, the Piston-on-three-Balls test is suggested. For this test, discs with a D/t »10 are supported on three fixed balls and loaded by a flat piston with a diameter similar to the specimen thickness. This test is only standardised for a narrow range on specimen diameters. For these two tests the stress at failure in the centre of the tensile face of the disc can be calculated form the test geometry and the load at failure using relations that were analytically derived. For these three test situations it will summarised which assumptions were made for the stress calculation and to what extend they are realised in the actual tests. Possible issues arising during the experimental implementation are addressed. Strength values of various ceramics obtained with these test methods will be compared on the basis of Weibull statistics.

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3D printing of ceramic bionanocomposites for bioprocessing applications

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Biotechnological processes offer great potential for resource-efficient, environmentally friendly production and purification of chemicals or pharmaceuticals. A key to innovation in this field is the development of new materials featuring pore structures at different length scales with embedded cells, enzymes or other biofunctional properties. However, it is challenging to design materials that are fully biocompatible and that can be used in high-throughput and continuous processes under flow conditions. A facile way towards complex 3D ceramics is liquid feed extrusion printing of hydrogel/ceramic nanocomposite inks, which combine the gelling properties of an organic hydrogel component with the structural properties of a percolating particle network. An example for this is the use of cross-linked alginate with the added reinforcement of alumina particles. Optionally, the printed parts can be sintered at high temperatures to transform the nanocomposite into a pure ceramic. Based on this approach, we recently developed a biocompatible processing route for ceramic/hydrogel nanocomposites in which we embed living bacteria or yeast cells. The outcome is a highly stable bionanocomposite material with minimal shrinkage, highly increased structural and mechanical stability over pure hydrogels, as well as excellent biocompatibility. Most importantly, the viability, accessibility and productivity of the embedded cells can be demonstrated in model studies. Accordingly, this method shows great potential for producing macroscopic bioactive materials for biotechnological applications.

Thermal barrier systems damage in the presence of thermal gradients

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To model life to spallation of currently used Thermal Barrier Coating (TBC) systems, the robust identification of damage evolution is a key issue. The current work focuses on developing a model that includes the effects of thermal gradients both on progressive damage and on lifetime of TBC. For that purpose, a first part is devoted to the development of an innovative experimental methodology. In the as-received state, artificial interfacial debonded areas known in size and localization are introduced by LASER shock (LASAT) on AM1/(Ni,Al)-Pt/YSZ-EBPVD specimens along YSZ/TGO interface. Then, thermal cycling is applied to the coupons with or without thermal gradients. At several times during testing, non-destructive techniques are used to monitor the progressive evolution of blister from the initial debonded area. Such an approach is promising because it gives access to (i) a measure of the rate of crack propagation, which can be correlated to interfacial damage and (ii) an estimation of the energy release rate. Doing so, a fatigue-like damage model, where the interfacial delamination is driven by energy release rate, can be identified. A second part is dedicated to the numerical modeling of the multi-layered TBC system to estimate energy release rate. The approach consists of explicit modeling of the multilayered TBC system : top coat, oxide, bond coat and substrate, using 2D plane strain assumption. Explicit modeling of the blister enables us to access stresses at the vicinity of the crack tip and hence stress intensity factors that are one way of evaluating the energy release rate. Another way is contour integral evaluations around the crack tip, that use virtual crack extension methods.

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Restoration of historically valuable porcelain artifacts by ceramic additive and subtractive manufacturing

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Preliminary results of a nationally funded research project between Fraunhofer IKTS, KI Keramik-Institut GmbH and the associated partners Porzellansammlung Dresden and Staatliche Porzellan-Manufaktur Meissen GmbH are presented. Goal of the project is the development of a material-specific restauration method for historical artifacts by means of additive and subtractive manufacturing technologies applied to traditional porcelain. The disadvantages of synthetic substitute materials are that they are not translucent and tend to yellowish discoloration after long-term exposure to daylight. For that purpose, different AM methods for ceramics (VPP, FFF, MMJ, and PBF) are investigated. Fragments of historical artifacts were reconstructed and transferred into CAD data. For each AM method specific semi-products - light-curable suspensions, filaments, thermoplastic feedstocks, and granules - must be prepared, and process parameters must be developed. Following challenges have to be solved – first, the complex structure of the fracture surface can hardly be reconstructed as well as built according to the lower resolution of the AM technologies. For that reason, the fracture surface shall be adjusted to the existing artifact by subtractive post-treatment. Second, distortion of the built fragment during sintering must be avoided and, third, the glaze shall be adjusted in the degree of whiteness or color to the original artifact and must not wet the fracture surface during glaze firing.

Elaboration of lead-free piezoelectric thick films by aerosol deposition method

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Some piezoelectric devices (sensors, energy-harvesting devices…) need the deposition of a ceramic material on a suitable substrate (thickness 10-100 µm). Currently, only screen-printing appears as the possible method for their elaboration and allows complex patterning suitable for piezoelectric devices. Unfortunately, this process needs sintering after shaping and thus a thermal treatment. In order to solve this problem, CTTC and IRCER have developed an innovative printing method of thick films ceramic coatings, based on the Aerosol Deposition method (ADM or AD).

The principle of ADM is to spray a gaseous aerosol of dry powder particles onto a substrate through a supersonic nozzle. The kinetic energy of particles allows obtaining directly dense thick ceramic coatings. Although the consolidation mechanisms are not fully understood yet, ADM is an efficient way for the manufacturing of dense films at room temperature, allowing ceramic coatings onto multiple kinds of substrates including metals or flexible materials like polymers.

This talk focuses on the deposition of lead-free piezoelectric for energy harvesting applications. The selected materials is sodium bismuth barium titanate 0.94 $Na_{0.5}Bi_{0.5}TiO_3$ – 0.06 $BaTiO_3$ (NBT-BT). In a first step, we developed our own solid-state synthesis route. Then we optimized the deposition parameters onto metallic substrates (Kovar). Afterwards, we investigated the structure, microstructure and properties of the obtained thick films. Finally, we demonstrated the possibility of energy harvesting for a simple prototype device. The ability to obtain thick films at room temperature, without post coating sintering or thermal annealing, is the most benefit of this method.

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Electrodes and electrolyte for Na-ion batteries: Fundamental and practical aspects

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Li-ion batteries (LIBs), as energy storage devices, are key enablers for the decarbonisation of both the transport and power sector. Owing to more than 25 years of intensive research in LIB materials and engineering, in addition to the discovery of new Li-resources, LIB technology has witnessed cost drops in the recent years. Yet, considering the growing demand on LIBs in the market, the need for developing sodium ion batteries (NIBs) that utilizes more sustainable materials, as a complementary energy storage device to Li-ion is essential. To this end, several prototype Na-ion batteries using various chemistries based on sodium layered oxides/ polyanionic compound such as $Na_3V_2(PO_4)_2F_3$ or prussian blue analogs as positive electrode and hard carbon as negative electrode have already been demonstrated. Nevertheless, all these chemistries suffer with poor specific energy in comparison to their lithium counterparts, the major limitation towards commercialization of NIBs. Moreover, the development of suitable electrolytes to ensure the cycle life and safety of the NIBs also remain as the major bottleneck, hence defining a future challenge. In order to understand the origin of these aforementioned limitations, we have studied the poly anionic $Na_3V_2(PO_4)_2F_3$ and several sodium layered oxides systems and bench marked their performances in practical Na-ion full cells together with optimization studies on Na-ion electrolytes. The results will be discussed by considering the fundamental aspects such as structural-stability-electrochemical performance relationship of the electrode materials and their interfacial reactivity with the electrolyte, hence stability of the electrode- electrolyte interphase.

Discrete element simulation for ceramic: from compaction, to sintering and fracture

MARTIN, Christophe

This talk reviews recent advances in simulations based on the Discrete Element Method (DEM) applied to ceramic materials. Starting from a particulate material, ceramic powders are typically pressed and then sintered. During compaction, defects may arise due to density heterogeneities, spring-back effects upon unloading or ejection. Similarly, sintering may also lead to defects such as cracks when constraints arise. Once the part is sintered, its mechanical behavior, and in particular its fracture behavior is of course of interest. The DEM is naturally suited to model compaction and sintering as particles are explicitly modeled. DEM is also a powerful method to simulate the fracture behavior of dense or porous ceramics as it naturally allows to follow the topological modifications (branching, bifurcation) that come with fracture.
We illustrate the capacity of DEM on ceramics with several examples using our in-house code dp3D, which is dedicated to materials science. In particular, we show how such simulations can be effectively coupled to X-ray tomography images that can be used as input for initial realistic microstructures. The crushing of porous agglomerates that include a central defect, the fracture of partially sintered electrodes and of nacre-like alumina offer examples of application of dp3D on fracture. Constrained sintering also provides a good example on the possibilities of a discontinuous approach to tackle crack growth (or healing) problems with DEM.

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Advanced TEM of interfaces and defects in functional ceramics

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Functional ceramic materials have to be developed in an integrated fashion, where design, synthesis, characterization, modelling and behaviour in applications throughout their life cycles will be considered in a concerted manner. Novel functionalities of materials will have to be adaptive and materials will have to adjust their characteristics in response to external stimuli. These ambitions are providing challenges in experimental characterization, as only the recording of multiple signals on the atomic scale can give full insight in the relevant properties. The aim of our present high resolution TEM activities is to provide quantitative materials data on the atomistic and nanometer scale that can be used for virtual materials design and to understand of structure property relationships. In the present lecture, several examples from our recent work will be discussed which are related to processes which are of high societal relevance in energy materials, membrane technologies and functional materials for future information technology.

Solid oxide cells based on fuel-electrode (FESC) and metal supports (MSC): Similarities and differences

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The functional layers of an SOC (solid oxide cell) – anode, electrolyte, and cathode – require a mechanical support for handling, stacking, and stable operation. Any of the aforementioned layers can in principle play this role in addition to its electrochemical functionality, and even non-electrochemical active supports are possible. Thus SOCs exist as anode- (ASC), electrolyte- (ESC), cathode- (CSC), metal- (MSC), and inert-supported (ISC) cells. This presentation highlights the materials and microstructural development of FESCs and MSCs, thereby comparing processing technologies, microstructures of the individual layers, and the electrochemical performance of the cells. In principle, cells based on fuel electrode-supports can be sintered under ambient oxygen partial pressure while MSCs must be sintered under vacuum or inert/reducing environment to suppress metal oxidation. Thin-film techniques like physical vapor deposition play a more crucial role for MSCs than for FESCs, but can partly be applied if the cells are operated at lower temperatures. And especially the air electrode needs different sintering conditions for FESCs and MSCs, as the perovskites used do not withstand the reducing conditions needed for the metal support. State-of-the-art FESCs and MSCs show, irrespective of the above mentioned boundary conditions, similar performance data.

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Texturing of calcium cobaltite for thermoelectric applications by pressure assisted sintering

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Thermoelectric materials can convert waste heat directly into electrical power by using the Seebeck effect. Calcium cobaltite (CCO) is a promising thermoelectric p-type oxide for energy harvesting applications at temperatures above 500 °C. The properties and morphology of single-crystal CCO are strongly anisotropic because of its layered crystal structure. Electrical and thermal conductivity are higher in the in-plane direction. By aligning the plate-like grains, this anisotropy can be assigned to poly-crystalline components.

In this study, the combination of tape casting and pressure-assisted sintering is used to texture and densify large scale components (50 cm^2). The influence of powder preparation and applied pressure during sintering on texturing and thermoelectric properties is investigated.

The analysis of XRD pole figures revealed that tape casting already leads to highly textured CCO. By pressure variation during sintering, the microstructure of CCO can be tailored either toward maximum power factor as required for energy harvesting, or toward maximum figure of merit as required for energy recovery. Low pressure leads to a porous microstructure and maximum figure of merit. Higher pressure leads to full densification and maximum power factor. The electrical and thermal conductivity of CCO seems to depend on both texture and sinter density.

Electric field distribution on ceramic samples during dielectric breakdown strength testing

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The dielectric breakdown strength of ceramics strongly depends on the test conditions. Thus, standardized test procedures and thorough documentation are indispensable. However, during dielectric strength testing the breakdown often occurs near the electrode edge or even outside the specified electrode area. This behavior is similarly observed for printed and cylindrical electrodes. The aim of the presented study was to calculate the electric field strength distribution in a ball-on-plate testing setup for metallized samples and to correlate the field distribution with the observed breakdown locations. Small misalignments in the test setup were also considered in the simulations. Furthermore, the field strength at the breakdown location should be compared to the experimentally determined dielectric strength. Therefore, Finite Element Models of several test conditions with varying printed electrode areas and sample thicknesses were created and electrostatic calculations of the electric field distribution were performed. The simulation results were compared to experimental data. Alumina (96%) was used as test material. The calculations show that the electric field strength maxima match the experimentally observed locations of breakdown. Without any fitting of the model, the maximum calculated field strength is in reasonable agreement with the experimental dielectric strength. The FE analysis is a helpful tool to understand the observations in experimental dielectric strength testing.

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Tailoring of microstructure by current-rate flash sintering of 10 mol% gadolinium doped ceria

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Flash sintering was discovered in 2010, where an electric field is applied to a powder-pressed specimen. The flash is signaled by a sharp rise in the current flowing through the sample. The current is then controlled by switching the power supply from voltage-to-current control; the current is preset to a limit. However, due to the inertia of the power supply the power density curve usually shows a spike during the transition from voltage to current control. This rapid spike in the power enhances the risk of hot spot formation or inhomogeneous densification of the specimen.

To overcome this drawback, flash sintering has been carried out entirely in the current control mode, the method is also known as current-rate flash sintering. In this experiment, the current is applied from the beginning and raised at a constant rate until the desired current density is achieved. The electric field is self-generated by the electrical resistance of the specimen. The onset of flash is signaled by a peak in the electric field profile. The power density profile revealed a steady growth, avoiding a spike. Surprisingly the densification, which occurred continuously, was related to the instantaneous value of the current density regardless of the current rate. The current-rate flash sintering is, therefore, promising for better control of the flash sintering process.

In the current work, we demonstrate the application of the current-rate experiment to 10 mol. % Gadolinium-doped Ceria (GDC10). The results were compared to experiments carried out in the voltage-to-current mode. The microstructure of the current rate sintered specimens was more uniform than in voltage-to-current experiments. Moreover, the microstructure could be tailored by changing the current rate.

Ceramic Multimaterial Components and Process Hybridization

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The presentation will give an overview over the opportunities and challenges of hybridization of materials and shaping processes. Motivations for hybridization are manufacturing of multifunctional components with property combinations like electrical conductive/electrical isolating, magnetic/nonmagnetic, ductile/hard, metallic gloss/white color, or porous/dense. Moreover, reduction of the number of processing steps, production cost and time is a driving force. The main challenge for co-processing of various ceramics or ceramics in combination with certain metals is the inevitable common thermal treatment, i.e. co-debinding and co-sintering. Beside comparable coefficients of thermal expansion (CTE) the compound partners must show an adjusted shrinking behavior and must allow a common sintering process at all.

A choice of material combinations basing on oxide and non-oxide ceramics, sintered glasses and metals will be introduced as well as co-shaping processes, like 2C injection molding, hydrid processes, like inmold-labelling, inmold-embossing, and ceramic Additive Manufacturing methods allowing for multimaterial approaches. The presentation will point to a spectrum of possible applications of material compounds developed by process hybridization during the last years.

Interface investigations will give insight into the bonding mechanisms between different materials.

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Processing of ceramic fibers, functional coatings and catalytically active ceramics based on specially tailored commercial oligosilazanes

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Since the first commercial production of silazanes in the 1980s, prices have fallen remarkably. Meanwhile, these precursors are commercially available on a ton scale at a reasonable price from Merck KGaA (Germany), but the variety of different silazanes is strongly limited, which prevents further distribution as for many applications a tailored precursor is required. However, the most common silazanes (Durazane types 2250, 1800 and 1033) are liquids but possess reactive groups like Si-H, Si-vinyl, N-H, and Si-N bonds suitable for precursor modifications.

This presentation gives an insight in our activities to tailor the commercial oligosilazanes for special applications. The use of tetra-n-butylammoniumfluoride (TBAF) as catalyst leads to selective cross-linking via N-H and Si-H groups resulting in a meltable polysilazane suitable for processing of ceramic SiCN fibers. Using the same catalyst, the reaction of the oligosilazanes with trifluoroethanol leads to F-modified polysilazanes for coating applications. Furthermore, a carbon rich C/Si_3N_4 fiber with extraordinary oxidation stability was developed using an acrylonitrile/oligosilazane hybrid polymer. Finally, the reaction of various metal-aminopyridinato complexes with silazanes led after pyrolysis to catalytically active M@SiCN ceramics suitable for hydrogen generation and storage.

These examples demonstrate that based on only a few basic silazanes many applications can be realized, due to a smart chemical tailoring of the precursors in combination with suitable processing methods.

Comparative study of suitable preparation methods to evaluate irregular shaped, polydisperse nanoparticles by scanning electron microscopy (SEM).

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Reliable characterization of materials at the nanoscale regarding their physio-chemical properties is a challenging task, which is important when utilizing and designing nanoscale materials. These materials pose a potential toxicological hazard to the environment and the human body. For this reason, the European Commission amended the REACH Regulation in 2018 to govern the classification of nanomaterials, relying on number-based distribution of the particle size.

Suitable methods exist for the granulometric characterization of monodisperse and ideally shaped nanoparticles. However, the evaluation of commercially available nanoscale powders is problematic. These powders tend to agglomerate, show a wide particle size distribution and are of irregular particle shape. Zinc oxide, aluminum oxide and cerium oxide with particle sizes less than 100nm were selected for the studies and different preparation methods were used comparatively.

First, the nanoparticles were dispersed in different dispersants and prepared on TEM-supported copper grids. Furthermore, individual powders were deposited on carbon-based self-adhesive pads. In addition, the samples were embedded by hot mounting and then ground and polished.

The prepared samples were investigated by scanning electron microscopy (including the transmission mode STEM-in-SEM) and Dynamic Light scattering. The software package ImageJ was used to segment the SEM images and obtain the particle sizes and shapes and finally the number-based particles size distribution with size expressed as various descriptors.

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Thermal modelling of field enhanced sintering

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Electrical fields with small and large field strength can enhance the sintering substantially. The most commonly used approach so far is the field assisted sintering (FAST) using small electrical fields. When using ceramics, most of the current is flowing through the die which can cause large thermal gradients in particular for large specimens. Furthermore, rather large temperature gradients inside the oven (water cooled metallic walls) imply large heat losses during the process. The influence of different thermal insulations on both phenomena was investigated using an electro-thermal coupled finite element simulation (ANSYS). The power consumption could be lowered by a factor of 5 using CFRC spacers sintering zirconia. The temperature became very homogeneous in specimens up-to 50 mm in diameter.

Flash sintering which uses significantly higher field strength requires yet a much more fundamental understanding, before in can be used for manufacturing ceramics. One aspect is the onset of flash sintering as a function of oven temperature and applied field as well as the stability of the sintering behavior. Using a dynamic bifurcation model based on the heat balance during the process, it was predicted if a material flashes at all and at which field dependent temperature. Furthermore the stability was predicted using perturbation theory which can be used to define safe process windows.

Donor and acceptor like self-doping by mechanically induced dislocations in bulk TiO_2

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The functional properties of ceramics are classically tailored by designing point defects and interfaces. Dislocations as heavily charged nanoscale line defects have so far been underappreciated as a means to tune functionality but are finding increasing attention today. To modify electrical properties of rutile (TiO_2), a semiconductor with wide bandgap prevalent due to its essential applications; for example, gas sensors and as a photocatalyst in solar cells, defect engineering via chemical doping has an important role. However, often the solubility limits of the dopant restricts this method for tailoring material properties significantly while it increases material complexity.

Here, we demonstrate the possibility to induce equivalent conductivity enhancements akin to conventional chemical doping by mechanically introduced dislocations. We combine the understanding of mesoscopic dislocation structure and its behavior at elevated temperatures, which results in highly arranged dislocation bundles in single crystals. By controlling the mesoscopic structure of dislocations we are able to both enhance ***and*** reduce conductivity. These changes are documented by temperature and oxygenpartial pressure dependent conductivity measurements. In this way, the prospect of dislocations as "self dopant" is presented, where the additional design parameter of the dislocation arrangement renders them potentially superior to conventional chemical doping strategies.

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3D-printed sacrificial polymer moulds for prototypes and small series suitable for ceramic injection molding

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As any production engineer knows, the path from a desired part design to a part design which is ready for production can be anything but straight. Often, several iterations are required for testing of different aspects of part design and production process. For this qualification of injection molded components or for production of small series cheap molds are essential. The invention of single-use 3D-printed polymer molds potentially enables both, design freedom and fast design realization of additive manufacturing without limiting the wide material selection of injection molding. In this new approach, called Freeform Injection Molding (FIM), a UV-curable, dissolvable polymer is used to 3D print molds or mold inserts for injection molding. Using a dissolvable mold material removes all mold design considerations regarding draft angles, ejector pins and split lines allowing AM-like part designs which are simply impossible with conventional split tools. In this paper specific design, properties and machine mounting of polymer molds made by DLP stereolithographic method will be demonstrated. Further, the process performance of 3D-printed polymer molds with alumina CIM feedstock is evaluated in terms of finished part integrity, dimensions and surface finish.

Use of the laser diffraction to investigate the packing of ceramic materials

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Many ceramic components are produced by dispersing and compacting powdered ceramic in to the shape required (green body). This is then heated to just below the melting point of the material, at which temperature sintering occurs and the particles become bonded together.

How well this process works depends on several factors, but one of the more important ones is the particle size distribution and shape of the powdered material. The particle size defines the time and temperature required to attain full density during sintering (fine particles require shorter sintering times due to their high surface area). A direct relationship also exists between the size and the pore size observed in the green body.

Large particles pack inefficiently leading to large pore sizes. These pores are found to remain during sintering, thus reducing the strength of the final product. Packing can be improved by reducing the size of the powder. A polydisperse ceramic powder can provide an advantage as small particles can fill the voids present between the larger particles, thus reducing the pore size. The presence of large agglomerates must be avoided as these can lead to defect formation during sintering, as agglomerated grains tend to grow more quickly than well-dispersed products. Again, this reduces the strength of the fired product.

In the past, many different techniques have been used to verify the particle size distribution of ceramic powders. Of these laser diffraction offers particular advantages due to its ease of use and fast analysis times in seconds. The dynamic range covered by modern laser diffraction systems allows both well–dispersed and agglomerates to be easily detected.

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Synthesis, evolution of crystallization kinetics and biological performance of sol-gel derived 1393 bioactive glass powders

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In this study, 1393 bioactive glass ($53SiO_2$-20CaO-$6Na_2O$-$4P_2O_5$-12K $_2O$- 5MgO in wt. %) powders were prepared using sol-gel method. The influence of temperature and length of thermal treatment was evaluated using DSC, XRD, TEM and X-ray nano-computed tomography (nano-CT). The glass transition and crystallization parameters were evaluated under non-isothermal conditions using DSC. The temperature and time of the thermal treatment strongly influences on the formation of the crystalline phases and at the crystallization temperature (750 °C) combeite crystalize as a main crystalline phase. The amorphous and crystalline bioactive glass samples were subjected to in-vitro mineralization to assist a comparative study of the adhesion capabilities of the mineralized layer formed on crystalline and amorphous glasses. Higher dissolution and mineralization ability of amorphous bioactive glasses was observed as compared to its crystalline counterpart. The preliminary results reveled that synthesized bioglass is non-cytotoxic to MG-63 cells. The initial results provide a platform of possibility of tailoring the crystalline phases by controlling the nucleation and growth of crystalline phases via thermal treatments. It is the first time that this kind of material was investigated by XRM at lab scale. This novel technique enables the 3D visualization of features in the nanometer range, giving a suggestion of the distribution in the sample of the parts composing the sample.

Lifetime assessment of self-supporting alumina-rich flame-sprayed compounds

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Based on stable crack growth experiments and biaxial strength investigations, the objective of the study to be shown was the prediction of the lifetime of self-supporting flame-sprayed Al_2O_3- and Al_2O_3-ZrO_2-TiO_2-compounds under constant sub-critical loads. Both materials were further compared regarding their flexural strength, energy release, and sub-critical crack parameters. Here, distinct differences could be observed. In the contrary, the characteristic lifetimes only barely deviated from each other. The conclusion was drawn that the service lifetime under constant load application is governed by the characteristic flame-spray microstructure, albeit advantages in the flame-spray processing of Al_2O_3-ZrO_2-TiO_2 are still given, attributed to its lower melting temperature.

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Cold sintering of highly dense ZnO with nanometer sized grains and investigation of mechanical properties

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In the present work, cold sintering of ZnO was studied in a field assisted sintering (FAST/SPS) device. For process optimization, a systematic parameter study was conducted aiming to investigate how densification is influenced by the primary particle size, the agglomeration of the starting powder, pressure, the aqueous phase, the dwell time and the sintering atmosphere. The same ZnO powder, conventionally sintered at 900 ºC for dwell time of 1 h was used as a reference, and the parameter study revealed that – at a primary particle size (of starting powder) below 100 nm – the degree of agglomeration has a higher impact on cold sintered densification than particle size. Optimization of the processing parameters produced a material with > 99 % relative density with negligible grain growth (< 200 nm). No influence of atmosphere was observed on cold sintering. In contrast to literature studies, a defect free grain boundary structure was achieved even for cold sintered samples, without using any post thermal treatment, as confirmed by microstructural analysis (scanning and transmission electron microscopy). A mechanical study (nanoindentation and micro-compression testing) on highly dense, cold sintered samples gave 5.5 GPa ± 0.5, 100 GPa ± 5 and 1.2 GPa for the hardness, modulus and compressive strength, respectively. This was significantly stronger than the reference, conventionally sintered sample, which showed a hardness of 4.8 GPa ± 0.6, modulus of 109 GPa ± 6 and compressive strength of 0.8 GPa.

C/SiC hybrid brake disc with metal support body for e-mobility

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Future mobility concepts like electric powered vehicles demand for new braking technologies and brake disc concepts. Due to the technological progress regarding the recuperation capabilities of electric vehicles, friction brakes are merely needed for complementary braking and more importantly for emergency braking manoeuvres. Consequently, new brake disc designs are needed in which for example the mass of the brake discs can be reduced. Due to the fact, that the brake discs in electric powered vehicles aren't used as frequently, the corrosion of the brake discs and brake pads are problems which have to be coped with. Thusly the use case for brake discs for electric powered vehicles is very different compared to state-of-the-art brake discs.

A new concept in the form of a metal-ceramic hybrid brake disc is propagated for the use in E-Mobility. It consists of an aluminium carrier body which is lined with ceramic friction segments on the friction surface of both sides. For the friction segments a short fibre reinforced CMC (C/SiC) is used. The outlined concept allows for a light-weight, corrosion resistant and economically viable emergency brake with outstanding friction properties for the use in E-Mobility.

A potential use case of a mid-class sedan with a mass of around 1.8t and maximum travelling speeds of up to 200 km/h is taken as a basis for the design and construction of a metal-ceramic hybrid brake disc prototype, which was tested on the dynamometer test bench at the University of Bayreuth under emergency braking conditions. Different characteristic values like wear, COF and different temperatures were measured. Furthermore, possible joining methods were evaluated and thermomechanical characterisations of different joints were conducted.

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ATZ bioceramics made by injection molding for use in joint endoprosthetics

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Alumina and YTZP ceramics have been established in joint endoprosthetics since 1970s and 1980s. Zirconia rich dispersion ceramics made of alumina and YTZP are known for high strength, high fracture toughness and low wear rates for decades too. Thus, alumina toughened zirconia (ATZ) was introduced to the endoprosthetics market by Mathys in 2007.This article summarizes the first investigations for manufacture of a ceramic knee implant. Product development began with formulation of clinical needs. After defining the requirements for ATZ on basis of standards (ISO 13356, ISO 6474-2) feedstock development for ceramic injection molding started. A debinding process and thermal treatment - sintering and HIP - were carried out. High purity and the removal of any porosity are mandatory. The whole process was accompanied by the determination of properties such as green density, microstructure, chemical composition, density after sintering and HIPing and investigation of the phase composition. Parallel to the material development, a suitable injection molding tool was developed too and the first sample parts were geometrically investigated.A suitable ATZ feedstock was prepared. It was used to produce green parts with densities of 54.1 to 54.6% theoretical density and 98.2 to 98.3 % theoretical density after sintering and HIP. The chemical analysis met requirements. By adjusting the thermal process, the microstructure could be adjusted so that grain sizes for Al_2O_3 in the range between (0.33 - 0.45) µm and for YTZP in the range between (0.31 - 0.46) µm were produced.The first knee implants made of ATZ ceramic with a homogeneous microstructure were successfully produced by injection molding.

Prediction of crack initiation in ceramic laminates designed with residual stresses

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Structural ceramics are brittle materials with high strength. However, they often contain intrinsic flaws, such as pores, which may act as a stress concentrator during mechanical loading. Cracks usually initiate at such flaws and their unstable propagation follows, causing "catastrophic failure".

One way of increasing the resistance to the propagation of cracks is combining ceramic materials with different thermo-mechanical properties in a layered architecture. Using the strain mismatch between materials during cooling from sintering, residual stresses may be introduced in the layers. The in-plane compressive residual stresses, which "shield" the propagation of cracks, are counter-balanced with tensile residual stresses. Consequently, edge cracks at the free surface, tunnelling cracks in the bulk, and/or delamination of layers at their interfaces may form. The location, size or shape of such cracks cannot be known a priori, nor predicted with the linear elastic fracture mechanics.

In this work, we demonstrate how edge crack and/or tunnelling crack formation in laminates may be assessed using the finite fracture mechanics (FFM). This approach utilizes a "coupled criterion" (CC), which requires that the stress and energy at the potential location of the crack simultaneously exceed the inherent strength and fracture toughness of the material. A parametric finite element analysis of crack formation in layers of varying thicknesses and residual stresses has been conducted, demonstrating, that for certain combinations of thickness and stress, crack initiation may be prevented.

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Laser beam melting of ceramics

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Selective Laser Melting (SLM) is a direct additive manufacturing technology which uses a high power-density laser to melt and fuse powders together. Functional components with high structural integrity are produced routinely in the industry using this technology. If SLM is an almost mature shaping technology for metals and polymers, it is still in its infant age for ceramics. One significant challenge for the ceramist is to determine how to use this technology to produce high performance parts. SLM of ceramics can be categorized as direct or indirect. The indirect approach involves laser melting of a sacrificial organic binder in a polymer/ceramic composite powder to produce "green parts". This requires a subsequent debinding step followed by sintering to produce the final part. On the contrary, in direct SLM the laser is used as a localized and energetic heating source which melts the ceramics at a very high temperature. In the present talk, state of the art in SLM of ceramics is reported with a special emphasis on recent results showing that oxides and carbides can be processed in commercial SLM machines. Powder key factors to fabricate alumina, alumina/zirconia and Si/SiC 3D parts are discussed. Current limitations of the approach and examples of applications are described.

Impact of service conditions on performances of refractories in steel ladles

POIRIER, Jacques

The effect of service conditions is important on the refractory performances of steel ladles. This study surveys the effects of different operationnal factors on the damage of refractories: the slag composition, the stirring by porous plugs and the vessel rotation. The different mechanisms of corrosion and spalling are described in detail and industrial solutions to improve the refractory performance are suggested. The corrosion of MgO-C refractories are different depending on the slag composition. i) With CaO-SiO_2 slag, the dissolution of MgO is predominant. The wear rate depends on the under saturation in MgO of the slag and is reduced when silico-aluminates of CaO or MgO precipitate at the slag-refractory interface. ii) With CaO-Al_2O_3 slag, corrosion results from the slag infiltration and erosion of the matrix. The microstructure of the matrix is therefore essential. For the MgO-C qualities with Al, the dissolution of spinel controls corrosion and a slag enriched in Al_2O_3 is less aggressive. Corrosion also depends on the flow rates of the slag and is significant slowed down if the stirring is less strong and if suspended solids increase the slag viscosity. iii) A few amount of FeO in the CaO-Al_2O_3 slag gives rise to an additional reaction which involves Mg vapors formed by the reduction of MgO by C. A protective layer of MgO is formed at the hot face. iv) With rich FeOx slag, the carbon oxidation becomes significant and promotes erosion/corrosion of the matrix. High levels of FeOx (> 30%) have a corrosive effect. At lower levels (10-15%), the iron oxides promote the formation of an magnesio-wustite layer. The impact of the rotation of ladles is also crucial. It is the case for the walls and the bottom for which slag impregnation and spalling are strongly favoured.

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Extending the range of oxide and non-oxide ceramic nanomaterials

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Many ceramic materials rely on high temperature synthesis and processing, which trigger large grain growth and preclude the production of nanostructured materials. Because nanoscaling can modify, if not enhance properties such as hardness, catalysis, thermoelectric energy conversion or electrocatalysis, it is of utmost importance to search for pathways to design nanostructured ceramic materials from solids that belong traditionally to the field of high temperatures and of solid-state reactions. We will discuss our recent advances in the search of such challenging materials by designing nanocrystals within inorganic molten salts as high temperature liquid media. These liquids are thermally stable, possess large solvating ability, and fasten reactions compared to solid-state reactions. Such features enable triggering a wide range of reactions under kinetic control, thus leading to original nano-objects that can in turn be used as building blocks for further processing of ceramic nanostructured bulk materials. We will focus on nano-objects showing properties different than those of bulk solids with perovskite oxide nanocrystals for spin transport and electrocatalysis, metal borides and silicides for electrocatalysis applied to energy conversion in fuel cells and water electrolyzers.

Plastic deformation of polycrystalline perovskite $SrTiO_3$: Shaping and introducing dislocations for functionality

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A range of ceramics becomes ductile at elevated temperatures allowing dislocation-based plastic deformation. This gives the opportunity to shape ceramics and to introduce dislocations to tune their functional properties. As a promising approach, tuning electrical conductivity by inducing dislocations has been demonstrated for example for $SrTiO_3$ single crystals with deformation of several percent achieved. For polycrystals, however, it is much less proven and for many materials it is still unclear, to which extent this plastic deformation can be used as a high-temperature process to tune the microstructure with dislocations. Here, we reveal high degrees of deformation for polycrystalline $SrTiO_3$ and specify a parameter range in which this can be obtained. We demonstrate that deformation of several percent is achievable in only a few minutes at 1150 °C and also contrast deformation in the polycrystal with deformation in single crystals. By analyzing the samples and determining dislocation densities with high voltage electron microscopy, the process can be clearly linked to dislocation movement. A detailed discussion of the mechanical behavior at elevated temperature is presented including stress-strain curves at different temperatures and different strain rates. Hence, a parameter range in which plastic deformation is possible is benchmarked and characterized by activation energy and activation volume allowing inter- and extrapolation of the parameter field. This foreshadows the feasibility of high-temperature deformation as a process step allowing to tune functional properties of perovskite ceramics with dislocations.

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Grain growth in electric field: Influence of defects, AC and DC fields in strontium titanate

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Sintering and grain growth behaviour under the influence of electric fields were studied in vast detail during the last decades. New synthesis and processing routes were established using that knowledge, such as SPS and flash sintering. The present study contributes to the topic by investigating the impact of an electric field on the microstructure evolution in strontium titanate for external DC and AC electric fields. We took a closer look at the grain growth behaviour of dense ceramics with a special focus on defect chemistry and space charge layers. Well-defined model experiments and the use of alumina shielded electrodes to prevent joule heating were conducted and a solid dataset was established by varying temperature, AC frequency and the electric field strength. The local microstructure evolution was analysed by SEM. We will explain the effects of the DC electric field on grain growth by combining the knowledge on field-free microstructural evolution, defect chemistry and polarization. Electromigration of oxygen vacancies locally enhanced the grain boundary mobility and thereby leading to a gradient in the grain size. Finally, we apply low frequency AC fields and expand the model with some basic thoughts on kinetics and other possible defect related mechanisms, such as oscillating of defects on different length scales.

Development of AlN powder systems for additive manufacturing by vat photopolymerization

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Efficient cooling solutions require optimized substrate and thermal management materials with high thermal conductivity accompanied by a suite of additional, situationally relevant properties like corrosion resistance or electronic insulation.
Aluminum nitride (AlN) is one of many intriguing candidates for thermal management applications due to its high thermal conductivity of up to 250 W/m.K and low thermal expansion in addition to electrical insulation. Owing to many challenges in processing the material, primarily the hydrolysis of AlN powder and, in turn, the incorporation of oxygen in the AlN lattice, limiting its thermal conductivity, most industrially available AlN parts come in very basic shapes. Using lithography-based ceramic manufacturing (LCM), more complex geometries will become accessible.
AlN powder systems of various compositions were used to produce conventionally shaped parts by cold isostatic pressing (CIP). Using those parts, processing parameters like powder composition, sintering temperature, time and atmosphere were evaluated to obtain high thermal conductivity samples. The most promising parameter combinations were then used to produce and sinter additively manufactured (AM) parts with complex geometries.
Based on the comparison of properties like densification, thermal conductivity and microstructure of CIP and AM samples, it can be demonstrated that LCM is a promising solution for the manufacture of complex shaped AlN parts.

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Sequential manufacturing of highly functionalized ceramic components for rapid heating and cooling

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Ceramic materials are characterized by excellent chemical and physical properties, especially at high temperatures, and are therefore predestined for applications under harsh environmental conditions. Thickfilm technology is an established technology to produce robust ceramic substrates for electronic circuits, power electronic modules and sensors. By applying thickfilm technology on ceramic components of various shapes their range of functionality can be significantly expanded. For the manufacturing process, a sequential approach is used. It is based on different process steps for the realization of the ceramic substrates and the subsequent functionalization. Different material combinations are available. The presentation will demonstrate the current possibilities concerning the materials and technologies for creating ceramic components especially by sequential manufacturing. Furthermore, the functionalizing of various shapes by thick film technology will be addressed - starting with flat substrates and tubes for ceramic heaters with operating temperatures up to 800 °C. Even more challenging is the integration of heating and cooling structures in one ceramic component. This can be done by complex additively manufactured ceramic structures. First results of a structure for a PCR cycler will be presented. Further developments focus on the modularization of this structure. This work is supposed to contribute to more efficient PCR-Testers, which are so crucial in detecting the covid-19 virus.

Oxide and non-oxide CMCs – applications, perspectives and need for development from a manufactures point of view

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The company Schunk Kohlenstofftechnik GmbH has been involved in the production and development of Ceramic Matrix Composites (CMCs) since the mid-1980s. In addition to the continuous further development of the established C/C materials, which are produced at Schunk in particular by means of pressing and winding technology, the latest developments and projects cover a wide range of materials in the field of CMCs: Oxide Fibre reinforced Ceramics (OFCs) are being developed e.g. for an application in the field of industrial heat treatment, the primary shaping of non-ferrous metals or mechanically loaded parts in the exhaust area of hot gas turbines. New non-oxide ceramics in the form of silicon carbide fibre-reinforced silicon carbides (SiC/SiC) represent an interesting material substitute for metallic superalloys in the hot gas area of turbines in particular. From a national perspective, material development for these SiC/SiC materials is still far from complete and lags behind in international competition. Based on the existing CVI/CVD process know-how and experience in the field of CMC at Schunk, these gaps are to be closed in the next few years and marketable products are to be developed.

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New zirconia-based ductile composites for biomedical applications: opportunities and challenges

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The use of stabilized-zirconia in the biomedical field has been motivated by its high mechanical resistance coupled with excellent biocompatibility, wear and aesthetics features. Particularly, yttria-stabilized zirconia (3Y-TZP) can withstand strengths of more than 1 GPa and was used in manufacturing less-invasive femoral heads (HIP joint) until 2001, when a huge number of devices failed in recently implanted patients. Nowadays, 3Y-TZP is emerging as an alternative to titanium (dental implants) or other restorative dental ceramics and glasses. However, 3Y-TZP ceramic is brittle and can undergo low temperature degradation (LTD), which could lead to micro-cracking and a loss of strength, as was the case with 3Y-TZP femoral heads. Therefore, our current research is focusing on novel strategies for zirconia stabilization and processing in order to design composites with good mechanical properties, a perfect stability in-vivo (no LTD) and a certain degree of ductility (CAD-CAM machining) for long-lasting spinal and dental implants applications. In this work, after introducing main zirconia drawbacks and the state of the art in the orthopedic/dental fields, the processing and mechanical characterization of new composites consisting of ceria-stabilized zirconia (Ce-TZP) and two second-phases (16 vol.%) will be presented. Moreover, the tetragonal (t) to monoclinic (m) zirconia phase-transformation which lead to a mechanical behavior similar to metals (transformation induced plasticity or TRIP) will be deeply analyzed. The future of such composites will certainly rely on our capacity to process, with scalable methods, ultra-fine and homogeneous powders and materials.

Ceramic capillary membranes with tailored pore sizes and functionalisation for virus retention

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In this talk a versatile and effective virus filtration system based on zirconia is presented, which features high membrane throughput rates and high virus retention capacities at the same time.
Tubular ceramic capillary membrane filters were processed via an extrusion process based on yttria stabilized zirconia (YSZ) powders with particle sizes of 30 nm, 40 nm or 90 nm resulting in open porosities of around 50 %. By varying the YSZ particle size, average membrane pore sizes ranging from 24 nm to 146 nm could be tailored. As model viruses MS2 (diameter = 25 nm, isoelectric point (IEP) = 3.9) and PhiX174 (diameter = 26 nm, IEP = 6.2) were used. Since with virus filtration based on the size exclusion principle the obtained throughput rates were demonstrably low, hydrophilic and hydrophobic surface functionalisation strategies were investigated for capillaries with pore sizes significantly above the sizes of the virus particles. Here, we report that Virus retention increases most strongly for capillaries functionalized with C8-chains (n-octyltriethoxysilane, OTS), showing a log reduction value (LRV) of ~9 for both viruses with throughput rates of up to ~400 L/(m^2h) even under varying feed conditions. Accordingly, such hydrophobic ceramic membranes are a versatile alternative to conventional polymeric membranes for virus retention applications.

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Non-arrhenius grain growth in $SrTiO_3$ and related materials: The impact of space charge on grain boundary migration

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Grain growth in ceramics is a surprisingly complex problem and involves many physical concepts on lengths scales from continuum to atomistics. This talk contributes to our understanding by focusing on one specific aspect that usually does not gain much attention: Solute drag, i.e. the impact of space charge on grain growth. This is motivated by the grain growth transition in strontium titanate, where grain growth rates decrease by orders of magnitude from 1350°C to 1425°C. This transition is coupled to bimodal microstructure evolution and is caused by the co-existence of two different grain boundary types. A careful TEM analysis of the grain boundary structure did not reveal any changes with the grain growth transition. However, there are chemical changes of the grain boundary providing evidence that the space charge layer including the cationic stoichiometry is different for the two GB types. As space charge can be tuned by the defect chemistry, various additional investigations gave more evidence for the existence of solute drag, e.g. simple annealing experiments with different $p(O_2)$ and doping. The application of electric fields allows implying defect-chemical gradients and investigating the microstructural response. Overall, the existence of solute drag agrees well with phase field simulations as presented in the first talk of this series. These findings shed new light on our understanding of grain growth in functional ceramics. As solute drag results in a nonlinear GB mobility, grain growth is no longer continuous. This phenomenon offers potential for tweaking transport properties during microstructure evolution and for functional applications.

Novel functionalities in atomically controlled oxide heterostructures by pulsed laser deposition

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Complex oxides of the transition metals are of great importance and interest both from a fundamental science as well as a technological point of view. With regards to fundamental science, complex oxides show an interplay between strong electron correlations, band behaviour, as well as rich repertoire of ordering phenomena in the spin and orbital sectors making them an enduring focus of theoretical and experimental investigation. Recent progress in epitaxy-based thin film growth approaches has added a dimension to complex oxide research, providing opportunities to improve experimental control over the properties of the system. We now possess a number of tools that can be used to design novel functionalities in complex oxides. We have demonstrated recently that strong oxygen octahedral coupling at interfaces transfers the octahedral rotation from one oxide into the other at the interface region. As a result, we possess control of magnetic and electronic properties by atomic scale design of the oxygen octahedral rotation. In this contribution I will highlight recent work on magnetic anisotropy in manganites, as well as the control of the metal-to-insulator transition temperature in nickelate heterostructures and superlattices. I will furthermore highlight some recent new insights in the "physics" of pulsed laser deposition of complex oxides, as well as the large-scale growth of epitaxial oxides on wafers up to 200 mm in diameter.

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Dislocations in ceramics: A new tool for anisotropic functionality

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The disturbed bonding state at a dislocation in ceramics can lead to a strong electrical charge of the dislocation core and an attendant space charge around it. In conjunction with the strongly localized charge and strain, dislocations can be used as a tool to tune electronic, ionic, and thermal conductivity as well as surface reactivity. Akin to flux pinning in superconductors, dislocations are also expected to provide an interaction with domain walls and, therefore, a possible hardening mechanism.

As a dislocation structure displays high temperature stability compared to chemical doping and anisotropic properties due to their one-dimensional nature, further design opportunities may arise. Mechanical deformation, best assisted with high temperature, is one versatile tool to introduce dislocation density far in excess of that existent after conventional sintering. Mechanical reliability can be simultaneously enhanced, as a plastic zone and enhanced fracture toughness has been quantified.

Key to harvesting all these opportunities is the ability to tailor dislocation structures and to characterize them. Starting with select literature examples outlining prior work on dislocation-tuned functionality, we will provide tools to implement and characterize dislocation structures as well as first examples for tuned and enhanced functionality.

Future trends in additive manufacturing of ceramics: from smart feedstocks to real-time 3D printing and further full exploitation of the numerical chain

ROSSIGNOL, Fabrice

We will give here an overview of the major trends in additive manufacturing of ceramics while focusing on microfabrication as an illustration. Two major application fields will be covered through examples of recent developments performed at the Institute of Research for Ceramics, in Limoges, France: microelectronics and biosensors. In addition, the future issues linked to an even better exploitation of the numerical chain will be presented. More specifically, the new hype/hope of smart feedstocks, real-time 3D printing and Artificial Intelligence (machine learning, deep learning, etc.) will be discussed in an attempt to anticipate how far these new horizons could affect the industrial sector in the mid term.

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Damage mechanism classification of ceramic composites by acoustic emission and machine learning

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Ceramic matrix composites (CMCs) show enhanced fracture toughness due to several crack deflection mechanisms. During mechanical loading, the following damage mechanisms are expected to happen before the final failure of the composite: matrix cracking, fiber debonding, fiber breakage and fiber pullout. Hence, the understanding of these mechanisms is crucial for the further development of CMCs. In this work, the damage development during loading is evaluated using acoustic emission (AE) monitoring. At first, specific mechanical tests were used to induce each damage mechanism separately. AE signals recorded during these tests were related to their respective mechanism types and used to obtain a training dataset for a supervised machine learning model. This approach is advantageous since each damage mechanism could be analyzed individually. The model was then used to classify AE signals measured during tensile tests of oxide-based CMCs. The results show that the model can be successfully applied to identify and quantify the AE signals during loading of composites, as well as to provide information about the amount of energy related to each damage mechanism.

The path to the smallest artificial ceramic structure

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Ceramic stereolithography is so far limited by the ceramic particle fraction in the photocurable resin, which leads to scattering and lack of transparency. This hinders the micro- and nanofabrication of ceramic parts by utilizing the two-photon-polymerizaion (2PP) as for that highly transparent photoresists are crucial for the printing success. So far ceramic structures can only be obtained processing silicon based preceramic polymers, and by that missing the possibility to generate e.g. technical ceramics with desirable mechanical properties. In this work a new approach is presented to overcome the limits of heterogenous ceramic slurries by creating a water-based resin with low viscosity and high transparency while keeping a high ceramic content of 50wt% for satisfying sintering results. This resin preparation, 2PP printing and post-printing processes are stated to form a microstructure from alumina toughened zirconia (ATZ) with a 2PP-typical resolution and accuracy.

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Recent developments in ceramic materials for the processing of titanium and nickel based alloys

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Titanium and nickel based alloys play an important role in highly demanding applications such as in aerospace industries, power plants, offshore technologies and bio-medicine. However, titanium and to a lesser extent also nickel based alloys are highly corrosive in contact with ceramics at high temperature. This severe reaction tendency impairs their melting, casting and recycling. In this contribution we will present recent developments in refractory ceramic materials and their respective processing for titanium and nickel based alloys. We will start with an overview on high-temperature perovskite ceramics, their processing, microstructure, properties and performance. Especially the investigation of interface reactions with titanium alloys at high temperature by using diffusion couple experiments as well as the improvement of their thermal shock performance by designing their microstructure will be a focus of our talk. Secondly, we evaluate developments of refractory investment casting molds for nickel-based alloys, whereas we will also discuss modern shaping techniques such as additive manufacturing and novel characterization methods for molds and cores.

Pores in cellular ceramics

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Besides the formation of geometrically defined cells, in the manufacturing processes of cellular ceramics pores are formed – desired or undesired. Those pores range on all scales, and they perceptibly influence the materials properties. For applications of cellular ceramics it is often necessary to know the pore properties such as pore size distribution, orientation, open or closed. In the first part of this contribution well-established processes for the manufacturing of cellular ceramics with respect to the pore formation mechanisms will be discussed. The second part deals with the most important methods for porosity characterization – micro computertomography, Mercury intrusion, the buoyancy method, pycnometry, gas adsorption – and strengths and weaknesses of these methods and results will be exemplified with ceramic foams.

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Additive manufacturing (AM) of ceramic-based functionally graded materials (FGM) by multi material jetting (CerAM MMJ)

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Material Jetting (CerAM MMJ) could be established for AM of dense alumina, zirconia as well as silicon nitride components. But also non-ceramic materials can be processed, which could also be demonstrated for components made of technical glasses and hard metals. Because of the selective deposition of the molten particle-filled thermoplastic suspensions CerAM MMJ is also suitable for AM of multi-material components. In our study, we investigated the AM of ceramic-based Functionally Graded Materials (FGM) by CerAM MMJ, like zirconia components with a varying microstructure, multi-colored zirconia components as well as Si_3N_4 -based components, which combine an electrically conductive phase and a non-conductive phase. Through the further development of the CerAM MMJ device, significant improvements could be achieved with regard to reproducibility and resolution of the manufacturing process. Within the presentation the above mentioned investigations and their results are summarized. In addition, the results of the mechanical characterization of test specimens made of different materials are presented. For zirconia bending strengths up to 1275 MPa could be measured.

Strategies for improving the strength of 3D-printed alumina ceramics

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The Lithography-based Ceramic Manufacturing (LCM) technology has been established as novel technique for fabricating structural and functional ceramics. Much effort has been devoted to analysing the influence of the printing direction, surface finish, as well as sintering conditions on the strength distribution of 3D-printed alumina ceramics. In this work we explore a novel strategy to further enhance the strength of 3D-printed ceramics, based on a multi-material approach. The layer-by-layer capability of the LCM techniques is employed to combine alumina and alumina-zirconia ceramics in a multilayer architecture, introducing compressive residual stresses in the surface alumina layers. Biaxial bending is performed both on the 3D printed multi-material and monolithic alumina parts. Results are analysed in the framework of Weibull statistics. A characteristic biaxial strength higher than 1 GPa is measured on the multilayers, compared to 650 MPa in monolithic alumina, the difference corresponding to the magnitude of compressive residual stresses due to the thermal mismatch between material regions during cooling from sintering. This is the first report of employing additive manufacturing to tailor the strength of alumina ceramics, taking advantage of the layer-by-layer printing process. Designing complex-shaped ceramic architectures with residual stresses through additive manufacturing opens a new path for fabrication of technical ceramics with tailored mechanical properties.

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Multi-wire sawing of ceramic materials

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Ceramic substrates are used for a wide range of applications in electronics and sensor technology. Conventional substrates for power electronics are effectively manufactured via tape casting. Especially substrates made of non-oxide materials, which partly have larger sintered skins, or if very low substrate thicknesses or changing substrate thicknesses are needed for applications, the production by multiwire sawing can be a favorable alternative. The technology allows high flexibility in substrate thicknesses > 100 - 2000 µm with high surface quality, flatness, and strength. The possibilities of the technology are demonstrated using translucent Al_2O_3 materials and highly thermally conductive Si_3N_4 materials based on low-cost raw materials (> 90 W/mK). Typical roughness values (R_a) achieved for Al_2O_3 materials are between 0.2 - 0.4 µm and for Si_3N_4 materials between 0.3 - 0.5 µm. Due to the low surface damage during multi-wire sawing, high biaxial substrate strengths are reached (translucent Al_2O_3 materials > 1000 MPa and Si_3N_4 materials > 850 MPa).

Ceramic mold inserts for injection molding

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The injection molding technology is established widely for processing of plastic materials enabling a resource and time saving manufacturing of complex shaped parts. But this technology will become unprofitable if the product design changes frequently and the lot size decrease. The R&D project pursues the development of a novel conception of cost efficient molding tools for injection molding of small series up to 10,000 parts based on thin walled, precise and wear resistant mold inserts made of ceramics or ceramic-like composites. Based on a conception for the development and characterization of test parts consisting of three levels (basic design investigation - investigation of shape complexity - manufacturing of demonstrators) mold inserts made of alumina, ZTA, SiSiC and composites with polysiloxane matrix were produced by liquid ceramic manufacturing (LCM), ceramic slip casting, binder jetting and molding from a prototype. These mold inserts joint with a supporting rear structure could be mounted into a mold base together with other tooling components like ejector pins. Investigations of injection molding with thermoplastics (e.g. fiber reinforced materials, melt temperatures up to 320°C, injection pressures up to 1200 bar), thermoset composites (tool temperature up to 200°C) and ceramic feedstocks yielded series productions with up to 1000 parts. The R&D project is funded by the Free State of Thuringia with means of the European Union as part of the European Funds of Regional Development.

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Digital pore analysis of oxide ceramics - a comparison of optical and scanning electronic microscopes

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Porosity is an important material parameter for characterizing ceramic materials. Pores that remain after sintering are often the starting point of material failure. That is why a qualitative analysis of the pore distribution is important in the development of functional ceramics. In addition to the number of pores, their morphology and size distribution play a special role in the evaluation of mechanical and electrical properties as a function of porosity. For their analysis currently infiltration and imaging techniques are used. The use of high-resolution polarization microscopes makes it possible to analyse an open and closed porosity more efficient then using conventional methods. The use of focus stacking, coupled with computer-aided image segmentation, enables automated analysis of large sample surfaces. An additional advantage of this method is that it eliminates the need for etching methods for surface processing. To verify the method, samples were examined and compared according to the presented method and DIN EN ISO 13383. In the final comparison between optical and scanning electronic microscopy, the possibilities and limitations of the new method are demonstrated.

New concept for an innovative high-temperature resistant thermal insulation

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In addition to the development of sustainable and regenerative energy production methods, the reduction of waste heat released into the environment is one of the greatest potentials for saving primary energy sources. Particularly at temperatures above 600 ° C, high quality insulation materials are necessary since the heat losses do not take place via convection but primarily via thermal radiation. A novel concept for an innovative lightweight and high-temperature resistant ceramic-based thermal insulation is presented in this contribution. An alumina-based vacuum composite plate was realized which combine excellent insulation with a dense and strong housing. Experiments show that the detected heat conduction was very low, especially below the detection limit (0,02 mm^2/s) of the measuring device. Using this new insulation concept for various industrial applications have the potential to save installation space, mass and expensive materials. For this purpose, a possible strategy for the economic production new ceramic-based vacuum insulations by adapting the deep drawing process for ceramic green tapes is presented. Using this manufacturing technology allows the realization of large-area thin-walled ceramics, that can be used as a dense and high-temperature-stable shell structure – a necessity for an insulation product as described above. The presentation will summarise both – the concept of insulation and the current state of developments in deep drawing ceramic green tapes.

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Lithography-based ceramic manufacturing for printing of non-oxide ceramics

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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 far less mature.
This contribution mainly focuses on the advances concerning AM of aluminum nitride and silicon nitride-based ceramics. By using the so-called Lithography-based Ceramic Manufacturing (LCM) process it was possible to print and sinter silicon nitride components with the same thermal and mechanical properties as from parts made by conventional manufacturing (isostatic pressing). With a relative density of 99.8%, a hardness of 1500 and a biaxial bending strength of 760 MPa the tested composition is exactly at eye-level with its conventionally processed analog (hardness of 1500 and biaxial bending strength of 770 MPa) or aluminum nitride components with a thermal conductivity >160 W/m.K. These material properties in combination with the high precision of the LCM process allow the production of highly complex components that have not been feasible before and that are fully functional. Besides the printing process and the properties of the ceramic material, this presentation will also give an outlook on the status of lithographic printing of other non-oxides such as silicon carbide and cermets. Another focus will be on first case studies using 3D printed nitride ceramic parts to demonstrate the capability of this new manufacturing approach but will also highlight current boundaries and limitations of this technology.

Microstructure-based simulation of material properties for computational ceramics engineering

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While the industrial design of new metallic or polymer-based materials is nowadays broadly utilizing a variety of, often commercially available, computational tools, the development of ceramics is still mostly based on traditional recipes and empirical variation of material composition. However, microstructure-based simulation of ceramic material properties can be a very valuable tool to support selection of raw materials and thermal processing parameters for a tailored, application-specific material performance. In this work, we report on recent progress in the simulation of ceramic micro- or nanostructures, respectively. Two aspects will be discussed: the first one is simulation of the electrical properties of powder-based ceramics containing at least two phases. Based on representative volume elements (RVE) with realistic microstructures created stochastically by an in-house structure generator, the percolation behavior of the minority phase is analyzed. Finite element simulations utilizing these RVE yield conductivity / resistivity of the composite in dependence on microstructure, which can be used for material design. The second aspect is a newly developed integrated microstructure model on sintering which utilizes the Monte Carlo simulation approach. This model includes the most relevant phenomena of solid state sintering, i.e. stochastic atomic diffusion processes, local minimization of interface energy and rearrangement of entire particles. Besides fundamental insights, the model has also practical use: it can generate even more realistic RVE for property simulation, and it can assist in parametrizing the viscous properties of materials for macroscopic sinter simulations on component scale.

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Enhancement of the electromechanical response and fracture resistance of a multilayer piezoelectric resonator using residual stresses

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The contribution deals with a modelling and optimization of a multilayer, ceramic-based, piezoelectric resonator (energy harvester), which is commonly used as a converter of ambient vibrations onto an electrical energy usable e.g. for powering of low-power electronics as autonomous sensors etc. The proposed, multilayer concept of the energy harvester takes an advantage of presence of suitably designed residual stresses inside particular layers in order to enhance its fracture resistance and simultaneously its electrical performance. A crack arrest, by means of the compressive residual stresses (in the outer "non-piezo" layer), is utilized to this end. Primarily, the extended classical laminate theory (taking into account also the piezoelectric characteristics of functional layers) is used to define various designs of particular layers with various levels of residual stresses inside. The weight function method is subsequently employed to select a design, which is the most resistant to propagation of preexisting surface cracks upon the harvester operation. Selected laminate configurations are verified by means of FE simulations. The new proposed design of the energy harvester shows a significant improvement of the fracture resistance and simultaneously ability to protect the functional piezo-ceramic layers from cracking in comparison with traditional designs containing no or very low residual stresses.

Characterization method for electromechanical and structural correlation in piezoceramics under high-power drive.

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The electromechanical response of piezoceramics consists of multiple intrinsic and extrinsic contributions, e.g. lattice strain or domain wall motion, which determine the materials' macroscopic properties and their usability in electronic devices. While these contributions are well investigated in off-resonance conditions, comparably little is known about them in resonance drive. A better understanding of their behavior could help to prevent thermal depolarization and improve the performance of various high-power devices, such as ultrasonic motors, cutting/welding devices, therapeutic ultrasound, and voltage transformers.

This work aimed to get a direct insight into the structure-property relationship in piezoceramics during the high-power drive. First, a methodology was developed to determine the electromechanical parameters in the vicinity of the resonance frequency at comparatively-high AC electric fields. The characterization of materials under these harsh conditions is challenging due to high currents, high frequencies, and overheating. In a second stage, the setup was combined with synchrotron high-energy X-ray diffraction with stroboscopic data acquisition and a flat-panel detector, in order to monitor ***in situ*** the fast structural changes of the samples in the high-power drive. A correlation between the change of the electromechanical parameters, especially the decrease of the mechanical quality factor, at high vibration velocities and increased domain wall vibration was experimentally demonstrated for soft and hard $Pb(Zr,Ti)O_3$ piezoceramics. Moreover, the contribution of domain wall motion to the overall strain response was evaluated and compared to macroscopic measurements as well as FEM simulations.

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Fabrication of higher thermal stability doped β-tricalcium phosphate bioceramics by robocasting

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β-tricalcium phosphate (β-TCP) is one of the most attractive biomaterials for bone repair since it shows an excellent biological compatibility, osteoconductivity, and resorbability. It can be used to produce bone implants serving as temporary supports for bone regeneration.However, there are still some issues for β-TCP porous scaffolds fabrication. Indeed, β-TCP cannot be used as scaffolds in large bone defects or in load-bearing areas due to its weak mechanical properties related to insufficient densification level. Indeed, the sintering temperature is limited because of the occurrence of a phase transition β to α-TCP at 1150°C with a large lattice expansion causing microcracks and reducing shrinkage during sintering. The thermal stability can be increased by the incorporation of dopants inside the β-TCP lattice. Indeed, such dopants like cations can replace the calcium inside the structure and stabilize the β phase as well as improve the biological properties. Thus, cationic substitution can allow reaching higher relative density value.

In this work, doped β-TCP powders are synthetized by coprecipitation of $Ca(NO_3)_2$ and $(NH_4)_2HPO_4$ solutions in presence of different cations in order to prevent the phase transformation and increase the sintering temperature. Owing to the β-phase stabilization, new rapid sintering techniques like microwave sintering can be successfully applied with a limited grain growth. The sintered samples are compared in terms of microstructural and mechanical properties. In addition to the sintering and thermal stability study, 3D-printable suspensions are prepared from optimized doped powder to manufacture porous scaffolds by robocasting.

Mechanical and physical characterization of Al_2O_3-C foam filters produced by distinct processing routes

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The influence of the production route and filter dimensions on the mechanical properties of carbon-bonded alumina foam filters was investigated. Features like cold crushing strength and fracture behavior were analyzed and used to compare them. Microfocus computer tomography µCT was applied to characterize the geometry of the filters, providing a deep correlation with their fracture behavior. Furthermore, computer-generated filter geometries were used as reference. The results indicated that the centrifugation process is better suited for the production of carbon-bonded alumina filters, and is even more effective when the foam dimensions are increased in the range of those analyzed in this research. Finite element simulations showed the influence of the relative density and strut tapering on the cold crushing strength of filters, providing a correlation between filter structure and its failure mechanism. The attained results provided further insights towards the production of inclusions-free metal parts.

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Combination of PIP and LSI processes for SiC/SiC ceramic matrix composites

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Silicon carbide fiber-reinforced silicon carbide matrix composites (SiC/SiC CMCs) are promising candidates for components in the hot gas section of jet engines, as they exhibit high temperature resistance and low density compared to their metal alloy counterparts. Three common manufacturing routes are chemical vapor infiltration, reactive melt infiltration (RMI) and polymer infiltration and pyrolysis (PIP). This work describes a new approach of a combined PIP and RMI process. A combination of the processes seems attractive: the remaining porosity after PIP process can be closed by subsequent siliconization, resulting in a dense material. SiC/SiC CMCs were manufactured by PIP process using Hi-Nicalon Type S fibers. Generally, the processing of SiC/SiC, produced solely by PIP route, is rather time-consuming and the composites show a certain residual porosity. In order to obtain a dense matrix and to reduce the processing time, an additional RMI with silicon alloy is carried out after a reduced number of PIP cycles. To protect the fibers during the siliconization, a CVD fiber coating was applied. Microstructure was examined via µCT, SEM and EDS. Bending strength was determined to 433 MPa; strain to failure was 0.60%. The overall processing time was reduced by 55% compared to standard PIP route. The hybrid material contained 70% less unreacted carbon than material produced by LSI process alone.

Corroded refractory microstructures and the linked change of mechanical behaviour

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The estimation of refractory behaviour during process temperature,standard test like compression under load or hot modulus of rupture are performed. However, these methods determine specific temperatures and can only evaluate the refractory material itself or by using post mortem samples. Therefore, measurement systems like the High Temperature Resonance Frequency Damping Analysis (HT-RFDA) emulate the change of the E-Modulus in wide temperature ranges, allowing to monitor elastic behaviour of tested samples. To address the impact of corrosive materials like slags, this measurement system was enhanced and aligned to adjusted samples to enable in-situ measurements during the corrosion process. Due to the improved system monitoring of the elastic properties during infiltration, phase transition, crack propagation and solidification could be achieved. Furthermore, the impact additions like ZrO2 to the refractory and improvement of corrosion behaviour can be evaluated. In this study the change of elastic properties due to the impact of the corrosion of a blast furnace slag on a high alumina castables was investigated. The applied methods include a first characterization of the material with open porosity, density, in-Situ HT-RFDA measurements, examination of phase formation mechanisms by SEM and XRD as well as a study of the slag behaviour.

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Interdependence of piezoelectric coefficient and film thickness in $LiTaO_3$ cantilevers

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Electromechanical energy demands on homogenous thick films of piezoceramics with sufficiently large piezoelectric constant and reproducible performance. Single-phase $LiTaO_3$ films deposited by sol-gel processing have been fabricated as cantilevers to investigate the interdependence of dielectric and piezoelectric properties as a function of film thickness. Phase pure $LiTaO_3$ films with varying thickness in the range of 2.07-4.37 µm on stainless steel substrates were obtained after calcination of samples at 650°C. The relative permittivity of optimized spin-coated films peaked at 479.73 (1 kHz), whereas the piezoelectric coefficient (d_{33} mode) determined by piezoresponse force microscopy was in the range of 21-24 pm/V. A figure of merit (FOM) up to 3.29 (10–18 m^2/V^2) was determined for cantilever devices, which were able to generate a peak-to-peak voltage of 0.046-0.15 V using a 1 MΩ resistor as an impedance load at a fixed acceleration of 1.5 m/s^2. While the power density was in the range of ~4-20 × 10–9 W/m^3, which increased with the increasing film thickness. The leakage current density decreased in the range of 4 × 10–5-6 × 10–7 A/m^2 in the same direction. An optimal energy conversion efficiency was obtained for a thickness of ~3 µm. These devices were tested up to a temperature of 150°C for voltage generation. $LiTaO_3$ cantilevers are very promising for vibrational energy harvester (VEH) applications especially due to their cost effectiveness, small size, stability at higher temperatures, and repeatable properties, which makes them suitable for MEMS devices for industrial applications.

Multiscale study of the mechanical and thermomechanical behavior of C/C composites – an everlasting source of open questions

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The mechanical and thermomechanical behavior of C/C composites, although seemingly well known since decades, poses some fundamental questions that are still open. Here are some of them:
i) Why is the elastic modulus of pyrocarbon matrices only 10% of the graphene value? We build hints to the answer on the basis of molecular dynamics simulations and on orientation distributions of the anisotropy axes in polycrystalline graphite.
ii) How to measure the heat expansion of pyrolytic carbons? We propose an experimental determination method and show some results.
iii) What is the role of internal interfaces in the non-linear behavior of C/C composites? We present a modeling approach supported by push-out tests to address this issue.
iv) By what material parameters is the heat expansion of C/C composites primarily controlled on the macroscale? We show that answering this question is linked to the previous issue of the non-linear mechanical response.

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Evaluation of cold sintering feasibility for preparation of solid-state battery electrolyte

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Cold sintering is an emerging processing technique, which is promising for realization of dense ceramic materials for a broad range of applications. It is of special importance for applications where use of high temperatures leads to formation of undesirable secondary phases, like in case of solid state battery materials. The present work deals with investigation of the cold sintering process of the solid state Na- and Li-conducting electrolytes. As the Na-conducting electrolytes, the IKTS proprietary $Na_2O–Y_2O_3$-SiO_2-glass ceramics as well as commercially available NASICON ceramics were investigated. In case of Li-conducting electrolyte, the focus was on $Li_{1,3}Al_{0,3}Ti_{1,7}(PO_4)_3$ (LATP) material prepared at IKTS. The type and concentration of the sinter additives as well as the sintering pressure and temperature were systematically varied during sintering experiments. The microstructure, phase composition and density of the resulting materials were investigated using SEM, XRD and standardized Archimedes method, respectively.

The feasibility of significant densification (residual porosity <10% and corresponding density of >90% of the bulk value) under cold sintering conditions was shown for both types of ionic conductors. Most successful cold sintering experiments with the batterie materials required temperatures of 130-140 °C and pressures of about 600 MPa applied for 1-2 hours depending on the material type and sintering additive used. In addition, the resulting material shows no or only a small amount of undesirable secondary phase formation compared to conventional sintering. The electrochemical and further structural characterization of the obtained materials is the subject of ongoing experiments.

Transparent ceramic mirco-optical components - fabrication, characterization and application

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The development and manufacturing procedure of transparent microoptical ceramic parts and their characterisation is presented. Cubic zirconium oxide from Tosoh company and a specially developed stepby-step process for the preparation of ceramic compositions for lowpressure injection moulding is utilised. In addition, the wax-based starting material can successfully be used for squeegee forming of small optical parts in the range of 4 square millimeters. The rheological properties of the casting compounds were characterised and optimised for the forming process. Subsequently, a suitable thermal process consisting of debinding, sintering and hot isostatic post-compaction was developed. A suitable procedure for surface polishing of the microstructured surface is demonstrated on an example of a lens array with 400 individual lenses on an area of 64 square millimeters. Finally, the microstructural and surface properties as well as the optical imaging properties of the components are shown. The individual development steps and their results are presented.

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Numerical simulation of flash sintering of oxide ceramics

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As a promising sintering technique, flash sintering utilizes high electric fields to produce rapid densification at low furnace temperatures. The onset of flash sintering is signaled by a non-linear increase in conductivity, which induces a power spike that may cause hot spot formation. By using a controlled current ramp from the beginning the sudden power spike can be avoided. Furthermore, the grain size and porosity can be tuned by controlling the current rate during flash sintering. This study aims at understanding the densification mechanisms during flash sintering of 10 mol.% gadolinium-doped ceria (GDC10) and 8 mol.%Y_2O_3 doped ZrO_2 (8YSZ) by finite element (FE) simulation. Both conventional and current-rate controlled flash sintering have been studied. A numerical strategy has been developed to predict the thermal profile, onset temperature, and densification during flash sintering. Temperature and density dependent material properties that were defined in the simulation models were determined based on the experimental data. The simulation results derive an inverse relationship between the flash-onset temperature and the applied electric field, which corresponds to the early study on the role of Debye temperature in the onset of flash sintering. For the densification of the samples, a good agreement between simulation results and experimental observation was also achieved.

Exsolution of embedded nanoparticles in defect engineered $SrTi_{0.9}Nb_{0.05}Ni_{0.05}O_{3-}$ perovskite oxide thin films

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Metal exsolution is an elegant method for the in-situ synthesis of ceramic-based composite electrode materials with unique nanostructures for applications in solid oxide cells (SOCs). Based on the partial substitution of B-site cations by catalytically active elements and subsequent exsolution from the perovskite lattice under reducing conditions (SOCs fuel electrode), metallic nanoparticles can be prepared at the oxide surface with high coverage.

For the systematic study of the mechanistic processes governing exsolution, epitaxial $SrTi_{0.9}Nb_{0.05}Ni_{0.05}O_{3-}$ (STNNi) thin films fabricated by pulsed laser deposition (PLD) are employed as model systems. To invetiagte the interplay of nickel exsolution behaviour and lattice defects the defect structure of the perovksite host lattice is controlled and systematically varied based on the laser fluence applied for thin film growth.

The exsolution behavior is studied under reduction in H_2/Ar gas atmosphere, yielding homogenously distributed nanostructures on the surface of the thin films. Based on microscopic investigations spontaneous phase separation and the presence of dopant-rich features in the as-synthesized thin film material is evident. As we discuss the formation of such embedded Ni-rich nanostructures considerably influences the overall exsolution behaviour. Our results furthermore show that the introduction of non-stoichiometry to the precursor material has a detrimental effect on the exsolution dynamics of embedded nanostructures, which is correlated to the distortion of the perovskite host lattice.

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Influence of slurry preparation parameters on the microstructure and performance of the electrodes of lithium-ion batteries

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An optimal dispersion of the slurry plays a significant role in the electrode manufacturing process of lithium-ion batteries. Only an optimal comminution of the agglomerates of the starting powders and the short-term coagulates with the binder guarantees a high electrode layer quality and electrochemical performance. These include good distribution of the electrically conductive carbon black particles (CB) around the active material particles in order to achieve sufficient electrical conductivity within the electrode structure. Additionally, a well dispersed slurry ensures an effective use of the whole active material content since no large agglomerates have to be removed. This reduces as well the post-processing and scrap rates of electrodes Furthermore a high homogeneity of the entire layer can be achieved, whereby cracks can be avoided and better compression conditions during calendaring are ensured. This work shows results from mixing and coating experiments of typical electrode materials like NCM, LFP, LTO, Graphite and LMNO slurries and discusses the main parameters influencing their dispersion state. These are particle size and particle shape of the components, type of dispersing devices, dispersing parameters (energy input, dispersing time, mixing steps sequence, dry pre-mixing) and colloidal stability to avoid a recoagulation process. In addition to the listed parameters, the aspect of upscaling from the laboratory to the pilot plant process plays an important role in this presentation. In the end, the relationship between the resulting particle size distribution of the slurry and both the microstructure of the electrode layer and the electrode performance is presented.

Alginate-based gelcasting of spaghetti filters for metal melt filtration

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Ceramic foam filters are frequently applied in the foundry industry in order to reduce flow turbulence and the amount of non-metallic inclusions in the cast melts. Thereby, the corresponding filter material (e.g. Al_2O_3, Al_2O_3-C, ZrO_2, SiC) is tailored to meet the requirements during the casting of the underlying metal (e.g. aluminum, steel or cast iron). Besides the material, the microstructure and geometry of the filters are crucial regarding their mechanical and fluid dynamic properties. The common precursors for ceramic filters are polyurethane foams which result in a characteristic filter pore structure and hollow cavities within the sintered or pyrolyzed filter struts. Despite being state of the art, these inevitable limitations can lead to challenges during special casting processes with long casting duration and severe thermal, chemical and mechanical conditions (e.g. continuous casting of steel).

The present work presents an alternative processing route to manufacture cellular materials with solid strut cross-section and adjustable periodic filter pore structure by additive manufacturing. The slurry-based process exploits the gelation capability of minor alginate additions in contact with calcium-rich solutions. With the aid of a computer-assisted portal robot system, the nozzle is moved in a periodic pattern to create three-dimensional lattice structures. After investigation and optimization of the process parameters, carbon-bonded filter structures were successfully scaled up and tested in contact with molten steel.

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Towards high-performance full-ceramic friction pairings with low wear rate

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Ceramic matrix composites (CMC) are a known material for high-performance friction applications. Typically, frictional couples with CMC consist of C/SiC rotors and organic brake pads (LowMet). This work investigated the fabrication and characterisation of short-fibre reinforced CMC brake pads. The brake pads were manufactured via the Liquid Silicon Infiltration (LSI) process. Different amounts of high temperature resistant inorganic friction modifiers (5, 10, 15 vol.-% of petrol coke and/or SiC-particles) were homogenously incorporated into the brake pads. Additionally, brake pads containing these friction modifiers and $FeSi_x$-phases were manufactured through a modified LSI-process using a FeSi alloy. The brake pads (900 mm^2) were tested on a full-scale inertia dynamometer. The corresponding brake rotor was a commercial C/C-SiC disc (Ø 420 mm) with a SiC-rich friction layer. Start-stop-brakings were conducted from 20, 10 and 5 m/s with braking pressures of 5 MPa and 3 MPa. Automotive application related braking energies were simulated by setting the moment of inertia of the system to 96 kgm^2. The chemical and morphological properties of the friction surface of brake pads was investigated using SEM, EDX and XRD analysis. Through the incorporation of frictional additives and $FeSi_x$-phases, the build-up of a 3^{rd}-body-layer is observed for full-ceramic friction pairings and the wear of CMC pads can be lowered by up to 85% compared to state of the art LowMet pads. Furthermore, the braking time with CMC pads is reduced by 26 to 50% compared to LowMet materials. It is shown, that high temperature resistant inorganic additives play a key-role in tailoring the tribological properties of full-ceramic friction pairings.

Innovative ceramic anode material based on $TiNb_2O_7$/carbon nanocomposites for lithium-ion batteries

WILHELM, Michael; MATHUR, Sanjay

It is essential to develop energy storage devices with exhibit besides good cycle stability a high energy and power density to increase performance. Conventional Li-Ion technologies using graphite as anode material and are due to the formation of a passivating solid-electrolyte interphase (SEI) at a low working potential (~0.1 V vs. Li/Li^+) limited in their performance. $TiNb_2O_7$ (TNO) is reported as a promiscuous alternative anode material for lithium-ion batteries (LiBs). $TiNb_2O_7$ nanocomposites were in-situ synthesized through a simple and scalable sol-gel route. Incorporating $TiNB_2O_7$into carbon nanocomposites increases the electronic conductivity and enhances Li^+-ion diffusion coefficient. Electrochemical measurements exemplified high reversible capacities at different C rates. The combined advantages of the carbon nanocomposites and the crystalline phase were compared to the pristine TNO phase and exhibited benefits in high-rate Li-ion capability. This study demonstrates the usage of $TiNb_2O_7$/carbon nanocomposites as a sustainable and promising alternative anode material for LiBs.

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Rhenium nitride thin films via magnetic field-assisted CVD from volatile rhenium precursors

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Rhenium nitride is a promising candidate as superhard conductor. Different rhenium-to-nitrogen ratios and several phases result in challenging synthetic strategies for phase pure rhenium nitride. Novel heteroleptic rhenium(I) compounds, [fac-Re(I)$(CO)_3$(L)] (e.g., L= tfb-dmpda, (N,N-(4,4,4-trifluorobut-1-en-3-on)-dimethyl propylene diamine)), containing preformed Re–N bonds act as efficient precursors for selective growth of polycrystalline rhenium nitride (ReN) films by vapor phase deposition. This is the first known access to synthesize phase pure rhenium nitride by single source precursor approach without the need of additional gases. Interdependence of materials strength and thin film orientation is strongly influenced by external magnetic fields. Deposition of ReN films in presence of an external magnetic field showed an orientation effect with preferred growth of crystallites along 100 direction.

Oxide short fibre reinforced composites - mechanical properties and automation approach

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Oxide fibre composites (OFC) combine lightweight constructionpotential, excellent thermo-mechanical behaviour and high corrosionresistance compared to metallic materials. In comparison to continuouspreforms such as fabrics, the use of short fibre bundles enables betterdrapability around narrow radii and thus the realization of complexcomponents by near-net shaping or forming. In addition, they lead to asignificant cost reduction of up to 60% compared to long-fibrereinforced composites, depending on the filament count of the fabricand the roving, even though the mechanical properties, with the fibresoriented in loading direction, decrease. For a better understanding ofthe fibre spraying process developed at the Department of CeramicMaterials Engineering and the generated short-fibre reinforcedcomposites, the mechanical properties, with regard to fibre orientationand length, were investigated in a basic research by a hand lay-up. Inthe fibre spraying process, continuous oxide fibre bundles are choppedby a cutting unit to a predefined length immediately before beingejected into a slurry spray, which carries them towards a mould. Theslurry infiltrates the fibre bundles in-flight and the infiltrated fibrebundles are piled up on the mould layer by layer, together with excessslurry. The thereby fabricated preimpregnated fibre preforms(prepregs) are afterwards conditioned, stacked and laminated ifnecessary, consolidated and finally sintered. The resulting compositesexhibit a randomly oriented fibre bundle architecture which results inin-plane isotropic mechanical properties. A concept for the scale-up toa robot based fibre spraying system will be presented.

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Innovative porous refractory raw materials for more sustainable steel ladle linings

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Refractories for steel ladles use dense high purity raw materials, for example white fused alumina (WFA) or tabular alumina (TA) grains. Their porosity is often below 5 Vol. % which minimizes penetration of slag into those aggregates. Nevertheless, the overall porosity of the formulated refractory products are often in the range of 10 to 20 Vol. %. This is due to the relatively higher matrix porosity that surrounds the alumina aggregates. Consequently, slag penetration and corrosion starts in the matrix and becomes the performance limiting factor. This paper investigates to which extend the dense refractory aggregates could be replaced by grains with an elevated porosity without deteriorating the overall service life. The advantages would be: the density of the ladle lining could be reduced which could result in a better heat containment and a lower material requirement, or even a capacity increase if the crane is the limiting factor. This could improve the sustainability and reduce at the same time the cost per ton of steel under the condition that service life is kept at least constant and that it allows a save ladle operation. The addition of porous aggregates could potentially improve the thermal shock resistance and then even increase the service life at least for refractory products that typically deteriorate faster due to thermal shocks than corrosion. These aspects will be investigated using a newly designed porous multi-component refractory aggregate in comparison to dense high purity alumina aggregates.

The influence of particle properties on the behavior of ceramic-reinforced photo-curable resins for stereolithography

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The intensity of scattered light in a ceramic-reinforced resin is a function of the dimensions, the concentration and the optical properties of the ceramic particles in relation to that of the polymeric matrix. This contribution is a study of the influence collective affecting the curing behavior and the scattering of light in photo-curable resins. The particle size distribution was measured using a laser diffraction particle size analyser based on the Mie theory, and the scattering of light was quantified with a UV/VIS spectrometer equipped with an integrating sphere accessory. It was possible to separate scattered light from absorbed and transmitted light, and small changes in the particle size and concentration induced noticeable changes in the quantum of scattered light, with smaller particles leading to large scattering of low-wavelength light. The developed resins are intended for use in stereolithography additive manufacturing, which adds challenging mechanical and temporal requirements on the curing behavior of the resin. Thus, a photo-rheometer was employed to establish a correlation between polymerization rate and conversion levels versus particle size distribution and concentration.

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Removal of viruses from water using granular copper oxide based ceramic filters

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Access to reliable and safe drinking water sources remains of high concern with global impact, being further amplified by climate change and man-made pollution. Poor water quality directly affects many lives and threatens public health carrying pathogens and pollutants. Common ceramic filters are able to reliably remove large-sized microorganisms such as protozoa and bacteria, but they remain ineffective for removal of viruses. The small size of viruses (i.e. ~25 nm) prevents their efficient removal. In this study, we report the development of functional alumina granules, functionalized with copper oxide nanoparticles during spray-drying. This newly developed granular materials have a highly interconnected pore structure, providing an excellent permeability as required for efficient water purification. The presence of copper oxide nanoparticles led to a high antiviral performance, causing complete virus removal or inactivation. The efficiency has been observed in contaminated water samples with a virus concentrations up to 10^4 PFU/mL.

Silicon nitride – silicon carbide composites as radiation detector plates

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Silicon nitride (Si_3N_4) and silicon carbide (SiC) ceramics show excellent mechanical properties and are used in a wide range of industrial applications. The addition of SiC into a Si_3N_4 matrix results in composites with an improved long-term stability at high temperatures and wear resistance in comparison to monolithic Si_3N_4. Also, Si_3N_4-SiC composites can show semi-insulating behavior. There are only a few materials which reach this semi-insulating region. Most of those materials are either plastics or glasses with their specific disadvantages. Semi-insulating Si_3N_4-SiC composites possess the ideal property spectra for detector plates of resistive plate chamber (RPC) detectors, not only because they meet electrical requirements but also due to their resistance against radiation. RPCs made of Si_3N_4-SiC plates reach best performance concerning time resolution and efficiency for electron fluxes of up to 10^5 $s^{-1}cm^{-2}$. Besides testing equipment for research there is a large potential market for radiation detectors in the fields of radiotherapy, process monitoring or in the nuclear energy sector. Si_3N_4-SiC composites with SiC fractions near the percolation threshold were sintered by gas pressure sintering. The electrical conductivity was determined. The results show that SiC amount and processing condition must be properly adjusted to obtain composites with a semi-insulating electrical resistivity of 10^9 to 10^{10} Ohm cm.

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Additive manufacturing of electrical conductive Si_3N_4-$MoSi_2$ components for heating applications using ceramic Fused-Filament-Fabrication (CerAM FFF)

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Ceramic Fused-Filament-Fabrication (CerAM FFF) is a thermoplastic additive manufacturing method in which an endless thermoplastic filament is used as a semi-finished product which is melted and deposited under a heated nozzle. Since this is a direct working additive manufacturing method were the material is deposited and solidified selectively, multiple materials can be additively manufactured simultaneously. For a successful co-processing the different powders must be adjusted regarding the shrinkage behavior during sintering as well as in terms of thermal expansion to achieve defect free parts.

Using the two component FFF process with electrical conductive ceramic materials, resistance heaters can be produced that could be heated to > 1000 °C when electrical power is applied. The second non-conductive material can be used as a covering oxidation barrier or as substrate and insulation material between the electrical conducting paths. Such components can adopt both structural and functional properties, which can be used, for example, as self-igniting injectors for gas turbines acting as a mixer, a flow tube and an igniter at the same time.

The presentation shows recent results using the powder system Si_3N_4/$MoSi_2$/SiC in an electrically conductive and non-conductive mixture giving an insight in the process development, material properties and heating experiments of additively manufactured and sintered test geometries.

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Preparation and characterization of $La_{0.8}Ca_{0.2}Fe_{0.8}Co_{0.2}O_{3-\delta}$ as a new air electrode material for solid oxide cells

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Perovskites from the series $(La,Sr)(Co,Fe)O_{3-\delta}$ are mixed ionic and electronic conductors that are suitable for a broad variety of possible applications, with cathodes for solid oxide fuel cells (SOFCs), anodes for solid oxide electrolyser cells (SOECs), electrochemical gas sensors and catalysts being the most promising fields of application. However, due to long-term stability issues, current research efforts are directed towards development of alternative Sr-free materials. Recently, high electronic- and good ionic conductivities, as well as exceptionally fast oxygen exchange kinetics were demonstrated for $La_{0.8}Ca_{0.2}FeO_{3-\delta}$ (LCF82). In the present work, substitution of this material with cobalt is investigated with the aim to further improve sintering activity and electronic conductivity. Therefore, $La_{0.8}Ca_{0.2}Fe_{0.8}Co_{0.2}O_{3-\delta}$ (LCFC8282) was synthesized using a sol-gel method. XRD confirmed that the material is single phase. Thermal expansion coefficients (TEC) were determined as a function of oxygen partial pressure ($1\times10^{-3} \leq pO_2/bar \leq 1$) and temperature ($600 \leq T/°C \leq 1000$). In comparison of LCF82 with LCFC8282, the TEC of the Co-substituted material is slightly higher with values of $19\text{-}23\times10^{-6}\ K^{-1}$. The electronic conductivity of LCFC8282 is in the range of $128 \leq S\ cm^{-1} \leq 145$ at $600 \leq T/°C \leq 800$ and pO_2=0.1 bar and thus higher than that of LCF82 (~110 $S\ cm^{-1}$). First results on the oxygen exchange kinetics of LCF8282 show high activity towards oxygen reduction.

Development of Laser Speckle Photometry for Inline Defect Inspection in Ceramics

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Advanced ceramic components are frequently used in industrial applications. 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. Despite optimized production technologies, defects still occur in the manufacture of ceramics due to the complexity of the processes. Therefore, the characterization of ceramic products is particularly important in the field of high-performance ceramics to guarantee a good quality. Time-resolved Laser Speckle Photometry (LSP) is an inline optical method based on the evaluation of the temporal change of speckle patterns. In this method, an optically rough surface is illuminated by a laser beam creating the speckle patterns. A short thermal excitation leads to the deformation of the surface, which generates time-varied speckles. These are recorded by a digital camera for the further evaluations. A developed concept for quality monitoring of ceramic materials based on the LSP will be presented in current paper. The LSP implementation will be carried out using the example of dense and porous Al_2O_3 as well as cordierite ceramics. In addition, a robot-guided measurement principle for the detection of various defects will be shown. The current detectability of LSP in unsintered and sintered ceramics is 70 µm. Keywords: Laser Speckle Photometry (LSP), optic, non-destructive testing, defect detection, inline inspection, ceramics

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Determination of the strength of thin ceramic materials

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Fracture strength and failure probability are highly important characteristics to warrant the reliability of ceramic materials with respect to application-relevant conditions. Therefore the accurate determination of the material's strength and the statistical analysis of the experimental results are crucial. For thin specimens with thicknesses of 50-200 µm, which are typical of advanced functional ceramics, an assessment of the strength is particularly challenging, since established, widely used testing methods, such as the Ring-on-Ring testing (ROR), require macroscopic specimens with a thickness of typically 1 mm or more. The ball-on-3-ball test (B3B), which is a novel small-scale biaxial testing method, appears suitable for the characterization of materials of limited thickness. However, the testing of such thin specimens is associated with small displacements and low loads, typically in the range of microns and milli-Newtons, respectively, which requires new experimental solutions.

In this research work, we designed a miniaturized B3B rig to be used in a micro-indentation system. The applicability of the miniaturized B3B setup will be demonstrated and discussed for different ceramic materials, including Al_2O_3 and LATP.

Production oriented optimization of ceramic parts for 3D printing: sinter compensation and design optimization

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Historically, product development in the ceramic industry has been a rather experimental procedure involving an empirically informed iterative process of prototyping. This is both expensive and time consuming and thus methods of reliably predicting the required shape of parts are in high demand. However, the availability of such methods remains low, largely due to the complex and highly variable material properties of ceramics and the complicated processes that lead to deformation during sintering. The new CeraTop technology recently developed by sico-solutions provides the means to optimize topology and sintering for ceramics. Our new process technology makes it possible to predict how a 3D-printed green body must be manufactured so that the sintered end product has the desired shape while exhibiting the best possible mechanical properties. Using gradient-based optimization algorithms we determine a geometry which retains all desired properties, i.e. mechanical strength, while reducing mass and risk of breakage by considering the materials Weibull statistics. On the resulting part, the sintering process is then simulated and a shape optimization is applied to calculate the geometry that needs to be printed to achieve the desired shape after sintering. Combined, these methods provide a comprehensive and cost-effective improvement to the design process of ceramic parts. In addition, the shape of the green compact, which must be printed before the firing process so that the required component shape is achieved after the firing process, is determined. Our technology is not restricted only to additive manufacturing, it is also usable for all common ceramic shape forming processes.

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Limits of computer tomography aided characterization of different types of porous ceramic materials

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Ceramics with open porosity are attractive materials in many fields of applications covering medicine, catalysis, and filtration. Manifold technologies to produce porous ceramics are available, e.g. foaming and replica processes, resulting in various microstructures. Development and manufacturing of new materials is accelerating, while crucial characterization is becoming increasingly difficult and conventional measurements lack the desired speed. Computed tomography (CT) offers the possibility to three-dimensionally characterize entire samples with minimal sample preparation, while its main advantage is that it is non-destructive. Still, the assessment of quantitative results from CT measurements is not trivial. The poster presents CT characterizations of newly developed as well as commercially available openly porous ceramic samples. Properties such as porosity, permeability or pore characteristics were measured conventionally and compared to results calculated from CT-measurements using the commercial software VG StudioMax. The determined differences between measured and calculated values are presented and application areas as well as limits of the CT characterization are evaluated.

Erosion behavior of Y_2O_3 in fluorine based etching plasmas: Orientation dependency and reaction layer formation

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Yttrium oxide (Y_2O_3) is one of the thermodynamically most stable materials in contact with inductively coupled fluorine plasmas, which are extensively used in semiconductor manufacturing processes. This makes it a very promising material for the next generation of plasma facing components. Nevertheless, the fundamental chemo-physical erosion mechanisms are still not completely understood. In the present study we describe a novel orientation dependent mechanism occurring during plasma exposure, which leads to the formation of a plateau-like topography. We developed a straightforward method for re-localization of microstructural features which couples electron microscopy techniques (SEM, EBSD) with atomic force microscopy (AFM). This enables the characterization of specific sample areas before and after plasma exposure and allows a direct correlation between material properties and erosion behavior. Furthermore, transmission electron microscopy (TEM) and secondary ion mass spectroscopy (SIMS) are used to investigate microstructural changes and chemical gradients near to the surface depending on the applied bias voltage ranging from 50 – 300V during plasma exposure. This combined investigation of plasma induced surface morphology and near-surface properties enables to reveal new fundamental insights of the erosion behavior of polycrystalline yttria in CF_4 based etching plasmas.

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Study of microstructural evolution of a sub-micron Hydroxyapatite powder sintered by SPS, microwave sintering and conventional sintering

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For recent years, new sintering techniques were developed such as microwave sintering and spark plasma sintering. Both these new techniques can be used to sinter ceramics at a faster rate with reduced dwell times compared to conventional sintering. Some studies showed that the grain growth phenomenon could also be reduced. The hypothesis backing this study is that this behaviour difference might be explained by different mechanisms occurring at the first step of sintering.

The material chosen for this study is a sub-micrometer sized pure hydroxyapatite.A two step sintering process was then applied. The first step involved either conventional heat treatment, microwave or spark plasma techniques to achieve partially densified (~70% theoretical density) ceramics for microstructural analysis. Then in the second step the samples were fully densified by conventional sintering (1300°C – 2h – 5°C/min) to achieve >95% theoretical density, for all three of the chosen routes used in the first step. Characterizations include SEM observation, mercury porosimetry, specific surface area measurement and thermal conductivity by Laser Flash measurement. Differences were observed after the first step of sintering: the microstructures are finer for microwave and spark-plasma sintered samples with larger necks between particles compared to conventionally sintered ones. Correspondingly, the thermal conductivity measurements reveal higher values for these samples implying promotion of neck formation. The finer microstructure for microwave and spark-plasma pre-sintered samples is maintained after the second sintering step leading to denser ceramics compared to those obtained by uniquely conventional sintering.

Optimization and thermal management of 3D printed ceramic flow reactors

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Additive manufacturing enables fast, cost efficient and high quality products with little or no waste, as the flexibility in production and complex design options gives the opportunity to form 3D printed structures that can revolutionize many sectors. Many areas from refinery to pharmaceuticals are dependent on the use of a catalyst to achieve increased efficiency. However, conventional reactors for heterogeneous catalysis suffer from poor heat and mass transfer and high pressure drops. 3-way catalytic converters existing in almost all vehicles are one of the most frequent example in modern life. One of the problems associated with catalytic converters is the low efficiency at cold start and another is the formation of hotspots when the catalyst is operational that raises a safety concern.
3D printing offers a sophisticated solution to flow malfunctions and high pressures drops by precisely controlling the catalyst distribution within complex flow channel designs to regulate the temperature gradient over the reactor.

In this study, 3D-printed (3DP) ceramic reactors were evaluated for mass-flow characteristics, optimal catalyst distribution and thermal management of the catalyst with a model reaction. Methods for active Au catalyst layer deposition were explored in the Yttria stabilized ZrO_2 (YSZ) 3DP parts. Debinding and sintering temperature were modified to control the porosity and Au adsorption on 3DP YSZ reactor. Channel dimensions and flow patterns are varied to control mass-flow while catalyst distribution is controlled to regulate heat generation over the structure. Finally, the conversion of benzyl alcohol oxidation reaction over Au and AuPd catalyst and catalyst gradients were tested in our novel devices.

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Synthesis of modified cathode materials for ceramic all-solid-state lithium batteries

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All-solid-state lithium batteries (ASB) are promising candidates for futures energy storage systems. They can solve current issue associated with liquid organic electrolytes and promise higher energy densities and intrinsic safety. A fully inorganic, garnet based ASB featuring only a Li metal anode, Ta-substituted LLZ ($Li_{6.6}La_3Zr_{1.6}Ta_{0.4}O_{12}$; LLZ:Ta) as solid electrolyte (SE) and a composite cathode of LLZ:Ta + $LiCoO_2$, was recently demonstrated. This kind of ASB delivers high areal capacities of up to 1.63 mAh/cm^2. In order to increase the capacity of those bulk-type ASBs, high capacity Ni-rich $Li[Ni_xCo_yMn_{1-x-y}]O_2$ (NCM) is considered as an active material. However, current NCM based cathode materials are optimized for application in liquid-based cells. In solid-state cells, the reactivity as well as the resulting decreased thermal stability of active material and SE is an issue during co-sintering at elevated temperatures which are needed for the fabrication of a dense composite cathode. A common strategy to optimize cathode materials is doping of the host structure with elements like Al, B, Fe, Mg, Sn, W and Zr. We present the synthesis of B-doped $Li[Ni_{0.8}Co_{0.1}Mn_{0.1}]O_2$ (NCM811), by a hydroxide co-precipitation route, as a possible active material for integration into bulk-type, fully inorganic ASBs based on ceramic materials. Additionally B-doped NCM811 is used as a model compound to verify the presence of boron in B-doped, Ni-rich NCM as well as to analyse its impact on the microstructure and electrochemical properties. Besides X-ray diffraction and Rietveld refinement, we used ion beam analysis (nuclear reaction analysis (NRA) and particle-induced gamma emission (PIGE)) to experimentally prove that boron is incorporated into the NCM lattice.

Controlling the lithium proton exchange of LLZO to enable reproducible processing and performance optimization

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Ceramic solid state-electrolytes attract significant attention due to their intrinsic safety and, in the case of the garnet type $Li_{6.45}Al_{0.05}La_3Zr_{1.6}Ta_{0.4}O_{12}$(LLZO), the possibility to use Li-metal anodes to provide high energy densities. However, one of the major obstacles hindering their wide-spread application is the translation from laboratory to industrial scale. Even though the plausibility of manufacturing via wet processing routes has been shown, the impact of the sensitivity of LLZO to air and protic solvents due to Li^+/H^+-exchange is not fully understood yet. An uncontrolled alteration of the powder surface results in poor reproducibility and electrochemical performance. To close this knowledge gap, the influence of the Li^+/H^+-exchange taking place at various steps in the manufacturing process was systematically investigated. For the first time, this allowed a mechanistic understanding of its impact on the processability and on the resulting electrochemical performance of a free-standing LLZO separator. The importance of a close control of the pre-treatment and storage conditions of LLZO, as well as contact time with the solvent could be extracted. As a result, we were able to optimize the processing of thin, dense, free standing LLZO separators and significantly improve the total Li-ion conductivity to 3.90 10^{-4} Scm^{-1} and the critical current density to over 300mAcm^{-2} without making structural changes to separator or the starting material. These findings do not only enable a deeper understanding and control over the manufacturing process, but also show potential for further improvement of cell concepts already existing in literature.

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Electronic transport at ferroelectric domain walls under alternating voltages

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Ferroelectric domain walls are natural interfaces separating volumes with different orientation of the spontaneous polarization in ferroelectric materials. The domain wall's unusual electronic transport properties make them highly attractive as nanoelectronics components, e.g., as switches, rectifiers or tunable capacitors. While the transport behavior under direct currents is well understood, little is known about their response to alternating currents (AC). Here, we explore the AC characteristics of charged ferroelectric walls in $ErMnO_3$ in the adiabatic regime (kHz-MHz) using a combination of atomic force microscopy and macroscopic dielectric spectroscopy. We find a distinct electronic response for walls in different charged state. For the charged domain walls, we demonstrate rectifying behavior that can be controlled by varying the amplitude of the AC input signal. Our results reveal novel opportunities for the application of charged domain walls as active components in AC circuitry.

Temperature-dependent high-power electromechanical properties of $Na_{1/2}Bi_{1/2}TiO_3$-$BaTiO_3$-based piezoelectric composites.

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Next generation high-power devices, like ultrasonic motors or transducers, are expected to enable a higher vibration velocity, v, and output power. This requires the use of ferroelectric ceramics with high quality factors, Q, and excellent electromechanical properties over a large v range. Moreover, it is necessary that the properties are stable at elevated temperatures in order to operate in the required ranges and to withstand self-heating effects. Large Q values are typically obtained by implementing ferroelectric hardening mechanisms. However, state-of-the-art hard ferroelectrics based on $Pb(Zr,Ti)O_3$ (PZT) suffer from strong Q degradation with increasing v or temperature.
This work investigates the high-power properties of composites consisting of a relaxor-ferroelectric (1-x)$(Na_{1/2}Bi_{1/2})TiO_3$-x$BaTiO_3$ (NBT-BT) matrix phase and ZnO inclusions, and compares them to Zn-doped NBT-BT and commercial PZT.
The potential of the composite approach as an alternative hardening concept was elaborated recently, especially in terms of enhanced Q and depolarization temperature. Here, we focus on the electromechanical properties of the composites under high-power drive, which were determined using a pulse-drive method with burst excitation. It is shown that the composites exhibit excellent v stability and negligible property change in a broad v range, whereas PZT shows severe degradation. In addition, the enhanced thermal stability is established from temperature-dependent measurements up to the composites' depolarization temperature. The composite hardening approach is thus demonstrated with enhanced high-power stability in comparison with classical acceptor-doped hardening effects.

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Determination and measurement of infiltration and capillary suction behavior of casted carbon-bonded silicon carbide refractory materials

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This study addresses the development and measurement of the infiltration and suction behavior of carbon-bonded silicon carbide (SiC-C) refractory castables and the influence of the pore size distribution at high and low temperatures. It has been found to be one of the major physical factor for infiltration in comparison to open porosity. Considering the infiltration behavior of slags with spreading wetting characteristics on refractory materials, the suction behavior becomes an important role. Moreover, the pore radius is significantly determining for the suction. In order to evaluate the influence of the pore radius on the infiltration behavior, the pore size distribution has been investigated, modifying the grain size distribution. An experimental setup based on the determination principle of the diffusion coefficient of plaster moulds was designed and tested with different liquids with different viscosities and good wettability on carbon bonded samples. Different level of suction behavior with similar tendency could be observed as a function of the viscosity. Provided that a slag wet the refractory material such a room temperature test could forecast the progress of the infiltration behavior at high temperatures. The results lead to the conclusion that the infiltration is not only and not priory controlled by the open porosity, but more by the pore size distribution. Corrosion tests of the casted carbon-bonded SiC samples with the slag showed a good correlation between the median pore radius and the infiltration depth. The trends of the corrosion test results are in a good agreement with the capillary suction measurements.

Ceramic-based thermoelectric generator via spray-coating and laser structuring

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Thermoelectric energy conversion has become an important topic in energy research for utilization of wasted heat. Thermoelectric generators (TEGs) are capable of direct conversion of thermal energy into electrical energy without moving parts. They usually consist of alternating n-type and p-type thermoelectric materials connected via metal contacts. Next to optimizing material's properties, there is an increasing interest in alternative additive and subtractive scalable manufacturing methods for TEGs. In this context, processing and sintering of thermoelectric ceramic materials is an important topic. A manufacturing process for a ceramic-based TEG, which consists of spray-coating of the thermoelectric materials and subsequent laser structuring to control the resulting design, is presented. With this combination of additive and subtractive manufacturing, the resulting layer thickness as well as the structuring and the final design of the layers can be precisely controlled. As a flexible substrate, which can be adapted to different needs, a low-temperature co-fired ceramic (LTCC) was used. The thermoelectrically active materials are $Ca_3Co_4O_9$ (front side) and Ag (back side). Subsequent sintering ensures the thermoelectric properties of the porous ceramic layer and leads to a rigid TEG. The universal production method can be further extended to different kinds of thermoelectric materials and generator designs.

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Mechanical properties of $BaCe_{0.65}Zr_{0.2}Y_{0.15}O_{3-\delta}$ proton-conducting material determined using different nanoindentation methods

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Proton-conducting membranes have great potential for applications in proton conducting membrane reactors for the production of commodity chemicals or synthetic fuels as well as for use in solid oxide fuel cells. However, to ensure the long-term structural stability under operation relevant conditions, the mechanical properties of the membrane materials need to be characterized. $BaCe_{0.65}Zr_{0.2}Y_{0.15}O_{3-}\delta$ is of particular interest due to its proven functional properties. In this research work, the mechanical properties of $BaCe_{0.65}Zr_{0.2}Y_{0.15}O_{3-}\delta$ were determined on different length scales using different methods including impulse excitation, indentation testing, and micro-pillar splitting. A detailed microstructural analysis of pillars revealed that irregular results are caused by pores causing crack deflection and complex crack patterns.

Hans-Walter Hennicke
Lecture Competiton

Development of a novel method for the production of CNT/ Al_2O_3-composites with homogenous CNT-distribution

BECHTELER, Christian

The presented work describes the development of a novel method for CNT/ Al_2O_3-composite production with a homogenous CNT-distribution. The investigated research contains detection of appropriate raw materials, suspension development, granule production, as well as the improvement of debinding and sintering and characterisation. Two different Al_2O_3-powders and a 4 wt.% CNT-suspension including 1.5 wt.% additives and 94.5 wt.% distilled water were used. Composites were produced by molecular mixing process. After freeze drying the granules were pressed in an uniaxial press for pressureless sintering. Beside that, the produced granules were used directly for hot pressing. Debinding temperatures for the investigated CNT/Al_2O_3-powders are very different because of an occurring catalytic effect. For pressureless sintering as well as hot pressing optimal sinter parameters were figured out. Relative densities of the pressureless sintered rings are between 49 and 83 % depending on the CNT-content. Hot pressing enabled nearly full densification of composites with low CNT-contents. At a CNT-content of 1,5 wt.% a relative density of 98 % was achieved. The density of the CNT/alumina-composite depends strongly on the CNT-content, alumina powder and sintering parameters. Also hardness, fracture toughness as well as electrical conductivity were characterised. The outcome of the research is a production method for CNT/alumina-composites with homogenous CNT-distribution. It enables the production of dense ceramics with an increased fracture toughness and electrical conductivity.

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Light scattering and optical properties of transparent ceramics

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Optical heterogeneities in ceramics, in particular pores, cause light scattering, leading to lower transparency or even full opacity. Therefore, the elimination of residual porosity is one of the main challenges in the preparation of transparent ceramics. Prediction of the influence of pores on the in-line transmittance of transparent ceramics is helpful for optimizing the preparation of transparent ceramics. Moreover, transmittance spectra calculated on the basis of light scattering predictions can be compared with results of spectrophotometric measurements. The rigorous solution for light scattering by spheres via Mie theory has one major disadvantage when it comes to practical applications – it requires numerical calculations. For this reason, approximations to Mie theory are often used as handy analytical tools, although the judicious choice of the adequate approximation is sometimes not an easy task either. Furthermore, until our recent publication, authors in the field of ceramics did not consider the polydispersity of pores (or other optical heterogeneities) while investigating light scattering. The aim of this contribution is therefore to explain methods of light scattering predictions and to demonstrate the light scattering effects on the in-line transmittance of transparent ceramics, including those with polydisperse pore size distributions.

Novel approach for the fabrication of mullite CMC

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Oxide fiber composites (OFC) exhibit promising properties for high-temperature structural applications in corrosive atmospheres, e.g. in gas turbines. They combine the advantages of ceramics, such as high-temperature stability and corrosion resistance, with ductile fracture behavior. In order to achieve those properties, the transfer of cracks from the matrix into the fibers has to be prevented. This can be attained either by a weak interphase around the fibers or a porous and therefore weak matrix. Thus, the fibers can still carry external load, while the matrix has already failed. The objective of this work was to develop a mullite-mullite composite, using the latter of the two principles of reinforcement. For the fabrication of those composites, a colloidal processing route (prepreg route) was developed to build up a mullite matrix around the mullitic ceramic fibers (3M Nextel™720). Since the slurry composition affects the processability of the prepregs and the microstructure of the sintered matrix, it has a high impact on the properties of the final composite. For good processability, the slurry requires a certain viscosity level within the process window as well as certain time-dependent rheological behavior. The influence of various slurry-components on these properties was investigated by rheological measurements. Tailoring the processing properties by adjusting the slurry composition alone, lead to an improvement of the 3-point-bending strength of up to 50%.

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Influence of preparation parameters on the properties and sintering behavior of oxide ceramic slurries

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An important component of Oxide Ceramic Matrix Composites (Ox-CMC) is the matrix. Typically, ceramic slurries are used during fabrication of Ox-CMC which later form the matrix. The preparation method and time have significant effect on the properties of the slurry and the final matrix. In this thesis, the influence on properties and sintering behavior of slurries in relation to the preparation parameters are studied. For this purpose, the suspensions were prepared with two technologies (planetary and co-ball mill). Furthermore, grinding materials (Al_2O_3 und ZrO_2 beads), additives (glycerin) and preparation times were varied. The resulting properties like particle size distribution (PSD), rheology, solids content, microstructure and shrinkage were analyzed. Compared to the co-ball mill, the planetary mill reached a defined particle size distribution after only one third of the time. Further, the tests showed that with increasing milling time the slurries tended towards an increasing viscosity and distinctive thixotropy although the particle size distribution remained almost unchanged. This leads to problems during the fabrication of wound Ox-CMCs. Overall, this effect can be reduced by replacing 10 to 20 wt% of water by glycerin. At the same time with identical solids content, more glycerin content lead to lower viscosity, increased weight loss but lower shrinkage of the samples. The results of this thesis can be used to optimize winding processes and the CMC production.

Influence of optical radiation on nanocrystalline functional ceramic coatings for energy conversion fabricated via powder aerosol deposition

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The Powder Aerosol Deposition Method (PAD) is becoming increasingly important as an energetically and economically preferable coating method compared to classic ceramic technologies such as tape casting. The spray process produces dense ceramic films in the thickness range of tens of µm at room temperature with a high deposition rate and a high coating quality directly from of the ceramic powder. Due to the process-related lattice deformation, ceramic coatings often exhibit reduced functional properties (like a reduced conductivity or a lower insulation behavior) in the as-deposited state. Thermal post-treatment at temperatures well below the sintering temperature can significantly reduce the lattice deformation and the stress within the films and restore the functional film properties close to bulk values. In this work, we present the post-treatment of PAD films using three different high-power LEDs with different wavelength in the visible range as an alternative to time-consuming furnace or energy-intensive laser processes. Thermoelectric $CuFe_{0.98}Sn_{0.02}O_2$ films serve as an example. The influence of LED radiation on the conductivity of the PAD films was investigated at different LED powers and working distances between LEDs and the films. In addition, the exposure time was varied and the effect of the LED operation modes on the film was analyzed. The exposed samples are compared with purely thermally treated samples. We can show that the LED-based post-treatment not only restores the bulk-like film properties, but also significantly reduces the required processing time compared to the oven process. The space-saving and potentially portable alternative film post-treatment developed here also offers a number of advantages over known methods such as the furnace or laser treatment due to its uniform surface treatment.

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Graded porous ceramics by injection moulding

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Graded porous ceramics are the holy grail in applications as biocompatible implants, thermal barrier coatings, foams for molten metal filtration or thermal shock resistant structures. The challenge is to adjust the graded porosity regarding its hierarchy and an acceptable production time. We achieve this goal by using sacrificial pore templates for continuous adjustment of the porosity in a ceramic injection moulding compound. Homogenous porous alumina feedstock with phenolic resin spheres as pore formers were prepared. Different levels of graded porosity have been achieved by stacking three ceramic green bodies to a layered structure. Microstructural investigations utilizing SEM showed dense and defect-free interfaces with homogenously distributed pores, independent of stacking sequence or degree of porosity. The mechanical properties revealed a comparable Young's modulus (238-261 GPa) to reference samples with an identical amount of homogenously distributed pores. Under compressive load, fracture frequently occurred in the exterior layer proving a mechanically stable connection between the individual porous layers. The presented technique facilitates an easily adjustable gradation of porosity, which can be applied to fabricate complex structures.

Modeling of the cooling behavior of thermoelectric multilayers

STARGARDT, Patrick

Multilayered designs are an attractive approach towards cost-effective manufacturing of thermoelectric generators. Therefore, efforts are being made to co-fire two promising thermoelectric oxides, namely calcium cobaltite and calcium manganate. In this study, ceramic tapes, multilayer technology, and pressure-assisted sintering (PAS) were used. A major challenge for the PAS of low-sintered calcium manganate was cracking during cooling. A relationship between the properties of the release tape used during PAS and the cracking behavior was experimentally observed. To understand the origin of failure, reaction layers in the multilayer were analyzed and thermal stresses during cooling were estimated by finite element (FE) simulations. Thermal expansion, elastic properties, and biaxial strength of the thermoelectric oxides and selected reaction layers were determined on separately prepared bulk samples. The analysis showed that the reaction layers were not the cause for cracking of calcium manganate. Using the FE model, thermal stresses in different manganate multilayer designs with varying properties of the release tape were studied. The FEM study indicated, and a validation experiment proved that the thickness of the release tape has the main effect on thermal stresses during cooling in separately sintered calcium manganate.

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The ball-on-three-balls- and the ring-on ring-test: A comparison of biaxial strength testing methods

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The strength of ceramic products that are available as (thin) discs or plates can be measured using biaxial strength tests. In the standardized Ring-on-Ring test, specimens are supported by a ring and loaded coaxially with a smaller ring. Using compliant interlayers, an even load distribution under the loading ring is ensured and the influence of friction is minimized. The maximum tensile stress at failure is determined from the maximum load and the test geometry using analytically derived relations. For the Ball-on-Three-Balls test, specimens are positioned on three balls which are free to roll and loaded by a fourth ball. Fitted analytical expressions of Finite-Element Analysis (FEA) results are utilized to evaluate the maximum tensile stress of the specimen and are available for many specimen size variations. For this work, these tests were conducted and modified on alumina discs. The modifications involve the use of varying intermediate layers and multiple specimen geometries. Their results are compared based on Weibull theory. It is shown how the utilized interlayers or their absence influence the measured strength values. The obtained results are extensively rationalized with FEA to evaluate the effects of deviations from ideal loading conditions. More specifically, the influence of friction between sample and fixture and the effects of an inhomogeneous load distribution on the maximum stress were investigated. To support these findings, fractography was conducted.

The influence of laser processing on the flexural strength of aluminum oxide (Al_2O_3)

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For technical applications, ceramics are used due to their high strength and hardness, corrosion resistance and lower density compared to metals. However, ceramics are inherently brittle and the scatter of fracture strengths may make them unreliable. Laser-processing of edges and surfaces changes the microstructure and the near surface stress state of ceramic components. As such, laser-processing can therefore directly influence the flexural strength of a ceramic material. In this work, the flexural strength of alumina (Degussit AL23), was determined by the four-point bending test and the Ball-on-three-Balls (B3B) bending test after various forms of laser-cutting and laser-structuring. The strength data of the experiments were calculated from the measured fracture force, and then evaluated based on a Weibull distribution. Since the effective volume of each sample in a B3B test cannot be obtained analytically, numerical calculations using the Finite Element Method were carried out to estimate the effective volume and the normalized fracture strength. The findings revealed a visible increase in normalized fracture strength for specimens with a laser-structured surface. Possible reasons are compressive residual stresses caused by the laser-structuring process at the surface of the components. To prove this, residual stresses were measured via X-Ray Diffraction Method. The results showed that the hypothesized compressive residual stresses exist, being a direct cause for the notably higher values in normalized fracture strength of the material. These strength values are beyond typical values for Al_2O_3 ceramics with polished surfaces. A noticeable increase in the Weibull Modulus of the laser-structured samples was also found.

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Switching the fracture toughness of single-crystal ZnS using light illumination

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Enormous change of the dislocation-mediated plasticity has been found in semiconductors that exhibit photoplastic effect. Herein, we report that the light illumination also dramatically affects the fracture toughness of ZnS. The crack tip toughness K_{I0} on (001) single-crystal ZnS, measured by near-tip crack opening displacement method, is found to have increased by ~45% in complete darkness compared to that in UV light. The increase of fracture toughness is attributed to the drastic increase of the dislocation mobility in darkness, as explained by the crack tip dislocation shielding model. Our finding suggests a promising route to tuning the fracture toughness of photoplastic semiconductors by controlling the light illumination.