Deutsche Keramische
Gesellschaft e. V.

KERAMIK

CERAMICS 2022

07.–09.03.2022
Virtuelle Konferenz
Virtual Conference

Partner Country: Slovakia
Partnerland: Slowakei

Abstracts

97. DKG-Jahrestagung
97th DKG Annual Meeting

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Grußwort der Programmkommission

Liebe Mitglieder der DKG und Freunde der Keramik,

hiermit laden wir Sie herzlich zur KERAMIK 2022, der 97. Jahrestagung der Deutschen Keramischen Gesellschaft (DKG), ein. Die Veranstaltung, die allen Keramikern und Keramikinteressierten offensteht, planen wir - wie bereits 2021 - rein virtuell.

„Virtueller Gastgeber der KERAMIK 2022" ist das Karlsruher Institut für Technologie (KIT).

Mit 10.000 Beschaeftigten und 24.000 Studenten erbringt das KIT als eine von elf „Exzellenzuniversitaeten" in Deutschland herausragende Leistungen sowohl in der Grundlagenforschung als auch Anwendung in den Natur-, Ingenieur-, Wirtschafts- sowie Geistes- und Sozialwissenschaften. Als die „Forschungsuniversitaet in der Helmholtz-Gemeinschaft" bereitet das KIT damit seine Studenten durch ein forschungsorientiertes, universitaeres Studium auf verantwortungsvolle Aufgaben in Gesellschaft, Wirtschaft und Wissenschaft vor.

Wir freuen uns, insbesondere die Slowakische Republik als Konferenzpartner der KERAMIK 2022 gewonnen zu haben und begruessen damit auch gleichzeitig alle unsere Kollegen aus den europäischen und nichteuropaeischen Nachbarlaendern zur Tagung.

Viele weitere Informationen zur KERAMIK 2022 und die Moeglichkeit sich online für eine Teilnahme zu registrieren, Beitraege einzureichen etc. finden Sie auf dieser Anmeldeplattform.

Bis bald
Im Namen des Programmausschusses

Greeting by the Program Committee

Dear members of DKG and friends of ceramics,

We hereby cordially invite you to KERAMIK 2022, the 97th Annual Conference of the German Ceramic Society (DKG). This event, which is open to all ceramists and those interested in ceramics, is scheduled entirely virtual - as we did in 2021.

„Virtual host of KERAMIK 2022" is the Karlsruhe Institute of Technology (KIT).

With 10,000 employees and 24,000 students, KIT is one of eleven „Universities of Excellence" in Germany and provides outstanding achievements in both basic research and application in science, engineering, economics and the humanities and social sciences. As the „Research University within the Helmholtz Association", KIT thus prepares its students for responsible positions in society, industry and science through research-oriented, university-based studies.

We are particularly looking forward to having won the Slovak Republic as a conference partner of KERAMIK 2022 and thus also welcome all our colleagues from the European and non-European neighbouring countries to the conference.

You will find further information about CERAMICS 2022 and the options to register online for participation, to submit contributions etc. on this registration platform.

See you soon
On behalf of the programme committee

Prof. Dr. Michael Hoffmann
Vorsitzender der Programmausschuss
Chairman of the Programme Committee

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Inhaltsverzeichnis
Table of Contents

Lectures

46 Wear characteristics of a (Hf-Ta-Zr-Nb-Ti)C high-entropy carbide
Prof. DUSZA, Jan

47 Synthesis of La_2NiO_4+? ceramic particles by molten-flux method
ESCOBAR CANO, Giamper

48 Understanding and engineering dislocations in oxides at room temperature
Dr FANG, Xufei

49 Sintering of functional lead-free ceramics by Cool-SPS
FAURE, Lauriane

50 Hard, tough, electrically conductive ceramics based on boron carbide
Dr. FENG, Bing

51 Porous alumina ceramics with multimodal pore size distributions
Dr. FEY, Tobias

52 Vorstellung des TFA 6.1 "Charakterisierung poröser Keramiken"
Dr. FEY, Tobias

53 Novel coating technologies for multilayer investment casting molds based on calcium zirconate for titanium alloys
Dr. FREITAG, Lisa

54 Dislocation-tuned electrical properties of oxide ceramics
Dr. FRÖMLING, Till

55 Dielectric breakdown strength investigation with a novel indentation method
Dr FULANOVIC, Lovro

56 Novel approaches in additive manufacturing of glass-based structures
Prof. Dr GALUSEK, Dušan

57 Advanced technologies for the manufacture of future piezoceramic components
Dr. GEBHARDT, Sylvia

58 Outstanding lead-free high-temperature multilayer ceramic capacitors based on $Na_{0.5}Bi_{0.5}TiO_3$
GEHRINGER, Maximilian

59 Advanced method for determination of ceramic lifetime and method-based fault diagnosis for sustainable filtration applications
Dr. GOEBBERT, Christian

60 LLZO free-standing ceramics and inks: Scalable electrolyte processing for solid-state batteries
Dr. GÖRNE, Arno

61 Conductivity, microstructure and mechanical properties of tape-cast LATP with LiF and SiO_2 additives
GROSS, Jürgen

62 Small-scale mechanical characterization of $LiTaO_3$ and $LiNbO_3$ single crystals for SAW filters
Dr. GRUBER, Manuel

63 Ceramic filter discs for ultra-/ and nano-filtration applications
Dr. GÜNTHER, Christiane

64 Silicon carbide - graphene composites with high thermal and electrical conductivity
Dr HANZEL, Ondrej

65 Surface bioactive silicon nitride modification by oxyacetylene flame treatment
Dr HIČÁK, Michal

66 Advanced materials for energy conversion
Dr. HINTERSTEIN, Manuel

67 $Li_7La_3Zr_2O_{12}$ solid electrolyte sintered by the ultrafast high-temperature method
IHRIG, Martin

68 Hydroxyapatite scaffolds printed by fused filament fabrication
Prof. JANEK, Marián

69 A low-cost approach for oxide-FRCMC with tailored structure
Dr. JANSSEN, Rolf

70 Cool-SPS : examining underlying mechanisms, sintering oxides
Dr JOSSE, Michael

71 Thermal storage systems made of mPCM and C/C-SiC for e-mobility
KESSEL, Fiona

72 Production of ZnO nanoparticles by pulsation reactor and investigation of the dispersing character of the produced particles
Prof. Dr. KHALIL, Tarek

73 Development, production and tribological characterization of ceramic coated bearings for media lubricated pumps
Prof. Dr. KILLINGER, Andreas

74 Ceramic sensors for materials characterization
Dr. KITA, Jaroslaw

75 Prediction of crack propagation in re-entrant honeycomb ceramics by polarimetry and digital image correlation
KÖLLNER, David

76 Novel single phase ($Ti_{0.2}W_{0.2}Ta_{0.2}Mo_{0.2}V_{0.2}$)$C_{0.8}$ high entropy carbide using reactive spark plasma sintering
Dr KOMBAMUTHU, Vasanthakumar

77 The influence of graphene platelets on mechanical properties of titanium diboride ceramic composites
Dr KOVALCIKOVA, Alexandra

78 Field assisted sintering of refractory composite materials Nb-/Ta-Al_2O_3 and the influence on material properties
KRAFT, Bastian

79 Commercialization of ceramic oxygen separation membranes
Dr. KRIEGEL, Ralf

80 Microstructure and thermoelectric properties of electrospun Ca_3Co_4 xO_{9+}? ceramics from nanofiber mats
KRUPPA, Katharina

81 The influence of gallium-containing mesoporous glass nanoparticles on pre-osteoblasts differentiation
KURTULDU, Fatih

82 Material development for SOC: Quo vadis?
Dr. KUSNEZOFF, Mihails

83 Surface modified ceramic materials for water treatment
KUTSCHIN, Thomas

84 The influence of the C-fiber orientation on friction and wear mechanisms of C/SiC brake pads
Dr LANGHOF, Nico

85 Silicon carbide ceramics with high thermal, electrical conductivity and other exceptional properties
assoc. prof. LENČÉŠ, Zoltán

86 Powder aerosol deposition of lead halide perovskites for optoelectronic applications
LEUPOLD, Nico

87 A novel evaluation method for prepreg processability of oxide fiber composites (OFC)
LINDNER, Felix

88 FE simulation of co-sintering process of metal-ceramic laminates
LIU, Chao

89 Transfer layer evolution and influence of environment on friction in hipims W-C:H coatings
Dr LOFAJ, Frantisek

90 Development of Babin plots for geometry optimization of transverse TEG
Dr. LÖHNERT, Romy

91 Stainless steel/ceramic composites for applications with molten aluminum alloys
MALCZYK, Piotr

92 Efficient photon-harvesting technologies for water splitting reactions
Prof. Dr. MATHUR, Sanjay

93 Recycling possibilities for solid oxide cells
Prof. Dr. MENZLER, Norbert

94 Influence of surface treatment on the strength of a dental zirconia for implants
Dr MIELLER, Bjoern

95 Superparamagnetic Fe@SiCN ceramic fibers for functional applications
Dr MOTZ, Guenter

96 Tape casting and optimization of the slurry composition of a SiCer-compatible LTCC tape
MOTZKUS, Clemens

97 Synthesis and characterization of SiO_2-CaO based mesoporous bioactive glass nanocarriers for delivery of therapeutic biomolecules
NAWAZ, Qaisar

98 Accelerated microstructure design using the high performance simulation framework Pace3D for phase-field models
Prof. Dr. NESTLER, Britta

99 Ceramic R&D in Japan –future prospects–
Prof. Dr OHJI, Tatsuki

100 The future of power generation starts today - with highly efficient solid oxide fuel cell systems
Dr OHNMACHT, Markus

101 Non-destructive monitoring of internal structure during the production of high dense 3D printed alumina parts
Dr ORLOVSKÁ, Martina

102 Usage of sintering aids for coarse pottery clays at low firing temperatures: an introduction
PESEK, Patrick

103 Solution processed $BiFeO_3$ thin-films for photovoltaic applications
PETRICK, Simon

104 Customized ceramic granules for laser powder bed fusion of complex alumina components
PFEIFFER, Stefan

105 Impact of electric fields on the microstructural evolution of strontium titanate
PREUSKER, Jan

Posters

Hans-Walter Hennicke Lecture Competiton

Plenarsprecher
Plenary Speakers

Prof. Dr. Britta Nestler is a full professor of the Department of Mechanical Engineering at Karlsruhe Institute of Technology (KIT). She holds a double position as a director of both, the Institute of Applied Materials at KIT as well as of the Institute of Digital Materials Science at Karlsruhe University of Applied Sciences HsKA.

The direction of research is on microstructure simulation in materials science. She has developed material models and a simulation framework Pace3D to describe phase transformation and microstructure formation processes on the micrometer length scales and incorporating multiple physics such as mass and heat transfer, thermo-chemo-mechanics, fluid flow and electro chemistry. The aim of the research is to achieve an accelerated material design by the support of high-performance computations and by establishing reliable digital twins. Recently she is involved in the development of data science methods as integral part of the novel research data infrastructure Kadi4Mat.

Britta Nestler got the first call for professorship in 2001 after her PhD in Materials Science at RWTH Aachen in 2000. She studied both, physics and mathematics and finished her diploma 1996 (Physics) and 1999 (Mathematics). Her extraordinary research success in microstructure simulations was honored with a number of prizes and awards, among them Richard-von-Mises prize 2002 of the GAMM society, the Materials Science and Technology Prize 2005 of the Federation of European Materials Societies (FEMS), the research prize 2007 of the state Baden-Württemberg, the Gottfried-Wilhelm Leibniz prize of the German Research Foundation (DFG) in 2017 and recently in 2019 with the Order of Merit of the Federal Republic of Germany "Courage for the Future: Overcoming Borders" awarded by the Federal President.

***Frau Prof. Dr. Britta Nestler** ist Universitätsprofessorin an der Fakultät für Maschinenbau des Karlsruher Instituts für Technologie (KIT). Sie hat eine Doppelfunktion als Direktorin sowohl des Instituts für Angewandte Materialien am KIT als auch des Instituts für Digitale Materialwissenschaft der Hochschule Karlsruhe HsKA inne.*

Die Forschungsrichtung liegt auf der Mikrostruktursimulation in den Materialwissenschaften. In ihren Forschungsarbeiten hat sie Materialmodelle und ein ein Simulationsframework Pace3D entwickelt, um Phasenumwandlungs- und Mikrostrukturbildungsprozesse auf der Mikrometer-Längenskala unter Berücksichtigung multiphysikalischer Vorgänge wie Massen- und Wärmeübertragung, Thermo-Chemo-Mechanik, Fluidströmung und Elektrochemie beschreiben. Durch die Unterstützung von Materialsimulationen auf Hochleistungsrechnern und die Erstellung digitaler Zwillinge ist das Ziel der Forschung ein beschleunigtes Materialdesign. Kürzlich beschäftigt sie sich zusätzlich mit der Entwicklung von Data Science Methoden als integraler Bestandteil der neuartigen Forschungsdateninfrastruktur Kadi4Mat.

Britta Nestler erhielt 2001 den ersten Ruf auf eine Professur nach ihrer Promotion in Materialwissenschaften an der RWTH Aachen im Jahr 2000. Sie studierte sowohl Physik als auch Mathematik und schloss ihr Diplom 1996 (Physik) und 1999 (Mathematik) ab. Ihr außergewöhnlicher Forschungserfolg in der Mikrostruktursimulation wurde mit einer Reihe von Preisen und Auszeichnungen gewürdigt, darunter der Richard-von-Mises-Preis 2002 der GAMM-Gesellschaft, der Materials Science and Technology Prize 2005 der Federation of European Materials Societies (FEMS), der Forschungspreis 2007 des Landes Baden-Württemberg, Gottfried-Wilhelm Leibniz-Preis der Deutschen Forschungsgemeinschaft (DFG) 2017 und zuletzt 2019 mit dem Bundesverdienstkreuz „Mut zur Zukunft: Grenzen überwinden" vom Bundespräsidenten verliehen.

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Dr. Tatsuki Ohji is a Fellow of National Institute of Advanced Industrial Science and Technology (AIST), Japan, and President 2019-2020 of The American Ceramic Society. BS and MS in mechanical engineering from Nagoya Institute of Technology and Ph. D. in inorganic materials engineering from Tokyo Institute of Technology, he has authored or coauthored more than 360 peer-reviewed papers and 20 book chapters, edited more than 40 books and conference volumes, and chaired or co-chaired more than 40 international conferences and symposia. His research interests include mechanical property characterization of ceramics, ceramic composites and porous materials, microstructural design of ceramic materials for better performance, structural control of meso/ macro porous ceramics, and green/additive manufacturing of ceramic components.

Fellow of The American Ceramic Society, the American Association for the Advancement of Science (AAAS), ASM International, and the Ceramic Society of Japan, Honorary Fellow of the European Ceramic Society and Academician of the World Academy of Ceramics, he has received numerous awards including W. David Kingery Award, John Jeppson Award, Samuel Geijsbeek PACRIM International Award, and ECD Bridge Building Award all from The American Ceramic Society, IIM Lectureship Award from ASM International, Honour Medal of Aurel Stodola from the Slovak Academy of Science, Lee Hsun Award from Chinese Academy of Sciences, Academic Achievement Award from the Ceramic Society of Japan, and Distinguished Research Achievement Award from the Japan Society of Powder and Powder Metallurgy.

He is a Governor of Acta Materialia Inc and an Editor of "Journal of the American Ceramic Society" and "Ceramics International" in addition to an editorial board member of many international journals.

***Dr. Tatsuki Ohji** ist Mitglied des National Institute of Advanced Industrial Science and Technology (AIST), Japan und war von 2019 – 2020 Präsident der American Ceramic Society.*

Er erwarb seinen Bachelor und Master in Maschinenbau am Nagoya Institute of Technology und promovierte in anorganischer Materialtechnik am Tokyo Institute of Technology, er hat mehr als 360 Gutachten und 20 Buchkapitel verfasst oder mitverfasst, mehr als 40 Bücher und Konferenzbände herausgegeben und mehr als 40 internationale Konferenzen und Symposien geleitet oder mitverantwortlich durchgeführt. Seine Forschungsinteressen umfassen die Charakterisierung mechanischer Eigenschaften von Keramik, keramischen Verbundwerkstoffen und porösen Materialien sowie das mikrostrukturelle Design keramischer Materialien zur Verbesserung von meso- / makroporösen Keramiken und der additiven Fertigung von keramischen Komponenten.

Als Mitglied der American Ceramic Society, der American Association for the Advancement of Science (AAAS), ASM International und der Ceramic Society of Japan, ehrenamtliches Mitglied der European Ceramic Society und Akademiemitglied der World Academy of Ceramics, erhielt er zahlreiche Auszeichnungen von der Amcerian Ceramic Socienty, darunter den W. David Kingery Award, den John Jeppson Award, den Samuel Geijsbeek PACRIM International Award und den ECD Bridge Building Award; den IIM Lectureship Award von ASM International, die Ehrenmedaille von Aurel Stodola von der Slowakischen Akademie der Wissenschaften, den Lee Hsun Award von der Chinesischen Akademie der Wissenschaften, Academic Achievement Award von der Ceramic Society of Japan und Distinguished Research Achievement Award der Japan Society of Powder and Powder Metallurgy.

Er ist Governor von Acta Materialia Inc und Mitglied des Editorial board des „Journal of the American Ceramic Society" und „Ceramics International" sowie Redaktionsmitglied vieler weiterer, internationaler Zeitschriften:

Prof. Gregory S. Rohrer received his Bachelors degree in Physics from Franklin and Marshall College, his Doctoral degree in Materials Science and Engineering from the University of Pennsylvania, and joined the faculty at Carnegie Mellon in 1990, where he is now the W.W. Mullins Professor of Materials Science and Engineering.

From 2005 to 2021, he was the Head of the Department. Prof. Rohrer has authored or co-authored more than 310 publications. His technical contributions have been in the area of surfaces and grain boundaries, where he discovered mechanisms for charge separation in oxide photocatalysts, grain boundary texture within polycrystalline materials, and grain boundary energy changes associated with complexion transitions. He has also developed methods for three-dimensional microstructure analysis and grain boundary property measurement.

Prof. Rohrer is a fellow of the American Ceramic Society and his research has been recognized by numerous awards, among which are the Richard M. Fulrath Award, the Robert B. Sosman Award, and the W. David Kingery Award, all of the American Ceramic Society. In 2011, he served as chair of the University Materials Council, from 2016 to 2019 he was a member of the Board of Directors of the American Ceramic Society. He has been an editor of Acta Materialia Inc. ince 2016.

***Prof. Gregory S. Rohrer** machte seinen Bachelor-Abschluss in Physik am Franklin and Marshall College, er promovierte in Materialwissenschaften und Ingenieurwissenschaften an der University of Pennsylvania und trat 1990 der Fakultät der Carnegie Mellon bei, wo er heute W.W. Mullins Professor für Materialwissenschaften und -technik ist.*

Von 2005 bis 2021 war er Leiter der Abteilung. Prof. Rohrer hat mehr als 310 Publikationen autorisiert oder mitverfasst. Seine wissenschaftlichen Beiträge liegen in den Bereichen der Forschung zu Oberflächen und Korngrenzen, wo er unter anderem die Mechanismen zu den Oberflächen und Korngrenzen im Bereich der Oxid- Photokatalysatoren, in polykristallinen Materialien etc. untersuchte. Unabhängig davon hat Herr Prof. Rohrerneue Methoden für die dreidimensionale Mikrostrukturanalyse und die Messung von Korngrenzen-Eigenschaften entwickelt.

Prof. Rohrer ist Mitglied der American Ceramic Society und seine Forschung wurde durch zahlreiche Auszeichnungen von der American Ceramic Society gewürdigt, darunter der Richard M. Fulrath Award, der Robert B. Sosman Award und der W. David Kingery Award. Im Jahr 2011 war er Vorsitzender des University Materials Council, von 2016 bis 2019 war er Vorstandsmitglied der American Ceramic Society. Er ist seit 2016 Editor von Acta Materialia Inc.

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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.

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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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Dr. Karl Berroth graduated 1978 in Mineralogy and Crystallography at University of Freiburg and obtained his PhD from Max-Planck-Institute of Solid State Research / University of Stuttgart. He worked there as scientific associate, before he took position as laboratory head at Institute for Material technology of Rosenthal Glas & Porzellan AG, Selb in 1981. Additionally he worked in R & D of„Werksgruppe 4, Ingenieurkeramik" of Rosenthal Technik AG and became head of R&D there in 1983. In "WG 4" material and production technology for Aluminium titanate, Silicon carbide and silicon nitride high tech materials were developed and components thereof produced and marketed. 1985 Dr. Berroth changed to SCHUNK Ingenieurkeramik GmbH -in the course of formation at this time- as head of R & D, technology and quality management. There, aside of a small production line for oxide and silicate based ceramics, R & D and production line for reaction bonded silicon carbide was initiated and build up for large and complex shaped components -also in high volume- made of SiSiC. After 7 years at SCHUNK Ingenieurkeramik GmbH in leading positions in 1992 he responded to a call as head of a newly founded chair for ceramics at EMPA; Swiss Federal Laboratory for Materials Science and Technology, Switzerland, for building up a ceramic R & D group with up to 30 staff. He also got lecturer at Swiss Federal Institute of Technology in Zurich. On January 1st 2000 he overtook former FCT Technologie GmbH, -renamed it to FCT Ingenieurkeramik GmbH- as Geneneral Manager and shareholder. Starting with 6 coworkers a company for research and development of silicon nitride and silicon carbide ceramics as well as for production and marketing of components for industrial applications thereof was grown up. Through to a broad portfolio of tailored materials and production technologies and the introduction of new products into many applications FCT Ingenieurkeramik GmbH reached rapid grow and international reputation. Finally, in 2019 he sold the business, -meanwhile grown to 150 staff and worldwide customers- within a process of follower. Aside of this Dr. Berroth also was lecturer at ETH Zurich, engineering school of Burgdorf, University of Bayreuth, University of Schmalkalden, speaker at many national and international conferences, seminars, workshops, and author of numerous publications and patents.

***Dr. Karl Berroth**, diplomierte 1978 in Mineralogie und Kristallographie an der Universität Freiburg und promovierte 1980 auf dem Gebiet der Festkörperchemie am Max-Planck-Institut für Festkörperforschung und Universität Stuttgart. Danach war er dort als wissenschaftlicher Mitarbeiter beschäftigt. 1981 trat er als Laborleiter ins Institut für Werkstofftechnik der Rosenthal Glas & Porzellan AG, Selb ein und arbeitete zudem an F+E arbeiten der „Werksgruppe 4, Ingenieurkeramik" der Rosenthal Technik AG mit. Er wurde dort 1983 Entwicklungsleiter. In WG 4 wurde Werkstoff- und Fertigungstechnik für Aluminiumtitanat, Siliziumkarbid und Siliziumnitrid entwickelt und Bauteile aus diesen Werkstoffen hergestellt und vermarktet. 1985 wechselte Dr. Berroth dann als Leiter F+E, Technik und Qualtätssicherung zur damals in Gründung befindlichen SCHUNK Ingenieurkeramik GmbH, Düsseldorf. Hier wurde neben der Fertigung von oxid- und silikatischen Keramikwerkstoffen die Fertigung von reaktionsgebundenem siliziuminfiltrierten Siliziumkarbid aufgebaut. Nach 7 Jahren Aufbau der SCHUNK Ingenieurkeramik GmbH in leitenden Positionen folgte er 1992 einem Ruf an die EMPA; Eidgenössische Materialprüfungs- und forschungsanstalt, Dübendorf, Schweiz, um dort eine Forschungsabteilung für Keramik -mit bis zu 30 Mitarbeitenden- aufzubauen und einen Lehrauftrag an der ETH Zürich anzunehmen. Zum 01.01. 2000 übernahm er als Gesellschafter-Geschäftsführer die damalige FCT Technologie GmbH, dann umbenannt in FCT Ingenieurkeramik GmbH. Beginnend mit 6 Mitarbeitern wurde daraus eine Firma für die Entwicklung von Siliziumnitrid- und Siliziumkarbidkeramiken sowie für die Herstellung und Vermarktung von Bauteilen für industrielle Anwendungen daraus. Durch das Angebot einer breiten Werkstoffpalette und geeigneter Fertigungsverfahren und -einrichtungen und die Einführung der Werkstoffe in unterschiedlichste Anwendungen erzielte die Firma hohes Wachstum und internationales Renommee. 2019 gab er die Firma, -zwischenzeitlich auf über 150 Mitarbeitende und weltweiter Kundschaft angewachsen- im Rahmen einer Nachfolgerregelung ab. Daneben wirkte Dr. Berroth auch als Dozent an der ETH Zürich, der Ingenieurschule Burgdorf, der Uni Bayreuth, der Hochschule Schmalkalden sowie als Referent bei unterschiedlichsten Seminaren, Workshops, Konferenzen sowie als Autor von zahlrechen Publikationen und Patenten im In- und Ausland.*

Prof. Dr. Ralf Diedel received his diploma in the subject of mineralogy with the main focus economic geology at the RWTH Aachen (Rheinisch-Westfälische Technische Hochschule Aachen) in 1984. In 1986 he got his PhD with thesis „Metallogenesis of the Kupferschiefer in the Lower Rhine Biay". Following to an assistant period at this department chair he changed to the newly founded Forschungsinstitut für Anorganische Werkstoffe Glas/Keramik GmbH (FGK) to Höhr-Grenzhausen in 1989 as technical-scientific director and took over the management from 1993 until 2016. Within this time period he expanded the FGK with regard to subject areas, staff and structural projects and established FGK as an application oriented ceramic institut with national and european cooperation partners.

In Höhr-Grenzhausen Ralf Diedel intensified cooperation between all ceramic teaching and research institutions, which merged to Bildungs- und Forschungszentrum Keramik e. V. BFZK. His engagement for the preservation of the WesterwaldCampus of the university Koblenz led to his appointment as Honorary Professor in 2011. At the Koblenz University he hold a lectureship in „quality assurance". His expertise in the fields of mineral raw materials and quality assurance flowed in numerous expertises for project sponsors, banks and insurances.

In his honorary activities for the DKG he was member of the board for 10 years and director of the technical committee „Rohstoffe" (raw materials) for 19 years and meanwhile the technical committee „Qualität" (quality). Additionally he undertook the task of cash auditor for several terms. Beyond his functions within the DKG he has been appointed in several scientific-technical advisory boards until today.

Prof. Dr. Ralf Diedel diplomierte 1984 im Fach Mineralogie mit dem Schwerpunkt Lagerstättenkunde an der Rheinisch-Westfälischen Technischen Hochschule Aachen und wurde dort 1986 mit seiner Dissertation über die „Metallogenese des Kupferschiefers in der Niederrheinischen Bucht" promoviert. Nach einer sich anschließenden Assistentenzeit am selben Lehrstuhl wechselte er 1989 als Technisch-Wissenschaftlicher Leiter in das neu gegründete Forschungsinstitut für Anorganische Werkstoffe Glas/Keramik GmbH (FGK) nach Höhr-Grenzhausen, dessen Geschäftsführung er 1993 übernahm und bis zu seinem Austritt 2016 innehatte. In diesem Zeitraum erweiterte er das FGK inhaltlich, personell und baulich und etablierte es als anwendungsorientiertes Keramikinstitut mit nationalen und europäischen Kooperationspartnern.

In Höhr-Grenzhausen intensivierte Ralf Diedel die Zusammenarbeit zwischen allen keramischen Lehr- und Forschungseinrichtungen, die sich zum Bildungs- und Forschungszentrum Keramik e.V. BFZK zusammenschlossen. Sein Engagement für den Erhalt des WesterwaldCampus der Hochschule Koblenz, wo er seit 2002 einen Lehrauftrag im Fach Qualitätssicherung innehat, führte 2011 zu seiner Ernennung als Honorarprofessor. Seine Expertise auf dem Feld der mineralischen Rohstoffe und der Qualitätssicherung war in zahlreichen Gutachten für Projektträger, Banken und Versicherungen gefragt.

In seiner ehrenamtlichen Tätigkeit für die DKG war er 10 Jahre lang Vorstandsmitglied, leitete 19 Jahre lang den Fachausschuss Rohstoffe, zwischenzeitlich den Fachausschuss Qualität und übernahm für mehrere Amtszeiten die Aufgabe des Kassenprüfers. Außerhalb der DKG wurde Ralf Diedel bis heute in zahlreiche wissenschaftlich-technische Beiräte berufen.

DKG-Ehrennadel
DKG-Ehrennadel

Eine DKG-Ehrennadel kann vom Vorstand als Zeichen außergewöhnliches Engagement für die Gesellschaft oder für die KERAMIK und bei einer langjähriger Mitgliedschaft an persönliche DKG-Mitglieder und Nichtmitglieder verliehen werden.

Die DKG-Ehrennadeln werden in drei Stufen verliehen:

- DKG Ehrenadel in SILBER
- DKG Ehrennadel in GOLD
- DKG Ehrenadel in GOLD (emailliert)

Dr. Torsten Rabe studied process engineering and inorganic non-metallic materials. He received his diploma from the Bauhaus University in Weimar in 1980 and was awarded a PhD in 1983 with a dissertation on the development of alumina ceramics for microelectronic applications. Since 1991, Dr. Rabe has worked at the Federal Institute for Materials Research and Testing in Berlin (BAM). Since 2012 he has been head of the department „Technical Ceramics“ at BAM. The main focus of his research activities are:

- Materials (LTCC, HTCC...) and technologies (hot embossing, zero shrinkage sintering...) for innovative ceramic multilayer devices in the fields of sensors, biotechnology, microsystem, communication and energy technologies,
- Development and evaluation of process accompanying test methods for the manufacturing of advanced ceramics (powder, slurry and green body characterization),
- Application-oriented development of structural ceramic materials (nitrides, zirconium oxide, composites).
- Scientific output includes 120 publications (50 of which as first author) in journals, books and conference proceedings.

In his voluntary activities, Dr. Rabe was head of the DKG Expert Committee „Material and Process Diagnostics“ and of the DKG/DGM Working Group „Process-Accompanying Test Methods“ until 2021, as well as a member of the DKG/DGM Coordination Committee „Advanced Ceramics“.

***Dr. Torsten Rabe** studierte Verfahrenstechnik und Anorganisch-Nichtmetallische Werkstoffe. Er erhielt an der Bauhaus Universität in Weimar 1980 sein Diplom und wurde 1983 mit einer Dissertation zur Entwicklung von Aluminiumoxid-Keramik für mikroelektronische Anwendungen zum Dr.-Ing. promoviert. Seit 1991 arbeitet Dr. Rabe an der Bundesanstalt für Materialforschung und -prüfung in Berlin (BAM). Seit 2012 leitet er den Fachbereich „Technische Keramik“ an der BAM. Schwerpunkte seiner Forschungstätigkeit sind:*

- *Werkstoffe (LTCC, HTCC…) und Technologien (Heißprägen, zero shrinkage Sintern…) für innovative keramische Multilayerbauteile in Sensorik, Biotechnologie, Mikrosystem-, Kommunikations- und Energietechnik,*
- *Entwicklung und Evaluierung von prozessbegleitende Prüfverfahren für die Fertigung von Hochleistungskeramiken (Pulver-, Schlicker- und Grünkörpercharakterisierung),*
- *Applikationsorientierte Entwicklung von strukturkeramischen Werkstoffen (Nitride, Zirkonoxid, Komposite)*
- *Der wissenschaftliche Output umfasst 120 Publikationen (davon 50 als Erstautor) in Zeitschriften, Büchern und Tagungsbänden.*

Dr. Rabe war in seiner ehrenamtlichen Tätigkeit bis 2021 Leiter des DKG Fachausschusses “Material- und Prozessdiagnostik” und des DKG/DGM Arbeitskreises „Prozessbegleitende Prüfverfahren“ sowie Mitglied im DKG/DGM Koordinierungsausschuss “Hochleistungskeramik”.

Lectures

Additive manufacturing of infrared heating elements by means of multi material fused filament fabrication (CerAM FFF)

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Fused Filament Fabrication (FFF) is known as a material extrusion process that enables manufacturers to produce parts by using a thermoplastic filament, which is melted in a heated nozzle and then formed layer by layer to obtain the desired complex part. One advantage of this method is the simultaneous processing of several materials that can be integrated in a single part. By using highly particle-filled filaments, it is possible to manufacture ceramics or metals in an additively manner. It opens up new possibilities for selectively combining mechanical and physical properties in a single component such as electrical conductivity/electrical insulation, dense/porous, brittle/ductile or even different colors. Within this contribution, a process for CerAMfacturing of multi material ceramic components – which can be heated up to 1400°C by applying an electrical current – for the contactless welding of plastic components through infrared heating was developed. For the aforementioned application, two materials had been developed containing Si_3N_4 and $MoSi_2$ in different ratios. In the electrically conducting material, the conductive $MoSi_2$ phase is percolating whereas Si_3N_4 dominates in the electrical insulating one. In contrast to the state-of-the-art technologies, utilizing quartz glass or metal foil emitters, this new technology offers a much greater geometrical freedom and manufacturing flexibility thanks to Additive Manufacturing. Besides applications related to contactless welding, the technology and process could also enable the manufacturing of components for turbine or aerospace applications. The presentation shows the process development and component testing of infrared heaters manufactured by CerAM FFF.

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Influence of copper and zinc in borosilicate bioactive glasses for bone tissue regeneration

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Bioactive glasses (BGs) are interesting materials for bone tissue engineering due to their osteoinductive and osteoconductive properties which allow them to bond to hard tissue and stimulate bone formation. The release of leaching products during the dissolution process of BGs not only plays a therapeutic role in the cell's osteogenic activation but also on properties such as antibacterial behavior and angiogenesis. Copper has been incorporated in BG due to the stimulating effects of copper ions on angiogenesis, antibacterial properties and osteogenic differentiation of mesenchymal stem cells. Similarly, zinc ions have been shown anti-inflammatory effects and stimulation of bone formation. The present study evaluates the effect of the incorporation of different concentrations of copper and zinc ions into the composition of the borosilicate 0106-B1 BG in terms of thermal properties, bioactive behaviour and antibacterial effect. In vitro bioactivity assessment was performed in simulated body fluid and characterized by means of SEM, FTIR and XRD. Additionally, antibacterial properties were evaluated against S. aureus and E. coli bacteria. The results show the potential bioactive behavior of the Zn and Cu-doped bioactive glasses for application in bone tissue regeneration.

Influence of sintering temperature on the cytotoxicity of the 3D printed HAP scaffolds.

BA A, Ubos; VASKOVA, Ida; HAJDUCHOVA, Zora; VETESKA, Peter; FERANC, Jozef; JANEK, Marian

Hydroxyapatite (HAP) as a main inorganic based constituent of bones, enamel and dentin possesses high biocompatibility and osteoconductivity. Pure HAP thanks to its properties is widely used for medical applications in forms of organic-HAP (e.g. as a biomimetic filler) or inorganic-HAP composites and/or sintered products. In this study, the hydroxyapatite scaffolds prepared by 3D printing were sintered at various temperatures. Their cytotoxic/antiproliferative activity was investigated by the agar-diffusion test using mice fibroblast cell line NIH-3T3 according to the ISO Norm Nr. 10993-5:2009. In vitro testing for cytotoxicity is a first indication for new materials toward ensuring their biocompatibility. Results showed that the sintering at temperatures 1200 °C and 1500 °C led to a slightly higher number of cells compared to the negative controls, thus these samples did not show a cytotoxic effect on the cells in this test. The testing however showed that sintering at 1300 °C and 1400 °C stimulated the development of cell culture, as the cell numbers in these samples greatly exceeded the cell numbers in the negative controls.

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Novel MgO and BaO doped zirconia compounds prepared by solid state synthesis

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In this study, two different compositions based on MgO, BaO and ZrO_2 were produced by means of solid state synthesis, characterised and evaluated with regard to their applicability as refractory materials. The aim was to find a processing route to produce compounds with high relative densities and low open porosity. The oxide composition of both compounds MBZ~0.1 and ZTB is based on two theories. MBZ 0.1 denotes the theoretical formation of an A-cation substituted perovskite of the formula $Mg_{0.1}Ba_{0.9}ZrO_3$. ZTB ('zirconia-toughened barium zirconate') is theoretically forming a two-phase composite of MgO-stabilised c-ZrO_2 and $BaZrO_3$. Different process parameters such as mixing the powders in dry or wet state, dwell time at final sintering temperature and conducting the synthesis in a one or two-stage thermal treatment process were investigated. The novel perovskite-like structures having bulk densities of more than 90 % of the true density and apparent porosity levels of <5 vol.%, and therefore providing a good starting point for the technological production of coarse-grained refractories. From profound investigations on the phase analysis and distribution as well as thermal stability, the ZTB compound was considered as more suitable to be further investigated as refractory material.

Development and adaptation of boron- and zirconium diboride-containing carbon fiber preforms for the fabrication of UHTCMC ceramics via reactive melt infiltration

BAIER, Luis; FRIESS, Martin; JEMMALI, Raouf; KLOPSCH, Linda

Ultra High Temperature Ceramic Matrix Composites (UHTCMCs) describe a group of fiber-reinforced ceramics suitable for consistent use at temperatures above 2000°C. Especially in the aerospace sector, UTHCMCs are steadily gaining importance, as materials are in demand at sharp edges (inlets), which are confronted with increasing temperatures due to increasingly complex trajectories, with simultaneous structural stability. The German Aerospace Center (DLR) is developing this class of materials via Reactive Melt Infiltration (RMI), which creates a basis that, compared to conventional sintering processes, can provide different fiber architectures in a load-bearing manner and, thanks to fast process times, is an economical approach. The production of the preform used for reactive melt infiltration is already one of the essential process steps, since the basis for important material properties of the final ceramics, such as the matrix composition, is already determined here. The infiltration quality of a boron and zirconium diboride-based slurry into a pitch-based carbon fabric is investigated. For this purpose, different fiber and fabric types were infiltrated with a water-based slurry using a foulard and via VARI and adjusted with reference to process parameters. Homogeneity of particle distribution and infiltration properties especially within the fiber bundle were evaluated by CT as well as SEM and EDX. Afterwards, the preforms were pyrolyzed and infiltrated with Zr2Cu via RMI. Final SEM and EDX investigations provide information on the microstructural properties, such as zirconium diboride formation and distribution in the matrix and differences of the ceramics based on the different approaches.

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Engineering ceramics and their composites: Tailoring material variables for tribological applications in extreme environment

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This presentation will provide an overview of the experimental results from our groups, where we will demonstrate the microstructure-toughness-wear correlation in some engineering ceramics of technological relevance. Specific examples of the published studies from the presenter's research group will be discussed to demonstrate how thermo-erosive environment influences the wear properties of ultrahigh temperature ceramics (UHTC) as well as high speed sliding in extreme cryogenic environment can impact the tribological properties of structural ceramics. The results of extensive finite element analysis (FEA) with high quality structural elements will be presented to illustrate the spatial temperature and stress distribution in the worn region. Finally, we will present how the material characteristics define their properties and tribological response as well as how to design ceramic-based materials for specific tribological application.

Performance of asymmetric oxygen transport membranes: guidance from modeling

WILKNER, Kai; MUECKE, Robert; BAUMANN, Stefan; MEULENBERG, Wilhelm A; GUILLON, Olivier

Oxygen transport membranes (OTM) can be used in the fields of oxy-combustion, the separation of pure oxygen or in membrane reactors for the synthesis of chemical energy carriers or commodity chemicals. The advantages of membranes are their higher energy efficiency compared to conventional processes as well as their modularity. Advanced membranes are designed in an asymmetric way, i.e. a thin dense membrane layer on a porous support providing sufficient mechanical stability. This structure, however, leads to a very complex combination of several transport mechanisms.

For a differentiated consideration of 12 individual material, microstructural, and operational parameters a 1D transport model is applied to asymmetric OTM. The model includes surface exchange, ionic and electronic transport as well as binary diffusion, Knudsen diffusion and viscous flux inside the support pores enabling the definition of a support limitation factor. A systematic sensitivity analysis revealed that for materials with very high ambipolar conductivity the transport is limited by the porous support in particular the pore tortuosity while for materials with low ambipolar conductivity the transport is limited by the dense membrane. The model identifies the most performance-sensitive parameters of the entire set of 12. Therefore, it is well suited guiding experimentalists in the targeted development of high performance gas separation membranes for any kind of application.

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Thermal radiation as origin of light emission during flash sintering of ionic conductors

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Flash sintering is a sinter method which uses external electrical fields to achieve full densification of ceramics at lower furnace temperatures and in a short time, compared to conventional sintering. The reason for the rapid densification in flash sintering is still under debate and a variety of possible fundamental mechanisms have been proposed. For instance, the formation of an avalanche of Frenkel defects, is mentioned as a speculative mechanism, causing such rapid densification. One evidence for the formation of Frenkel defects during flash sintering of ionic conductors, like 3YSZ, is the emission of light, which is not caused by thermal radiation. Electron-hole recombination, related to the formation of Frenkel defects, is proposed as an explanation for the detected spectra. In our research the combination of theoretical and experimental results about optical spectroscopy showed that no photoemission or electroluminescence, caused by the proposed electron-hole recombination, can be detected. If all influences of the experimental setup are considered in the interpretation of the detected spectra, it can be proven, that the emitted light during flash sintering of 3YSZ, is caused by Joule heating and the following thermal radiation. The results of the conducted investigations disprove one of the few evidences for the formation of an avalanche of Frenkel defects during flash sintering. Based on these results, and results from other investigations, it is assumable, that the formation of an avalanche of Frenkel defects is not the reason for the rapid densification of ionic conductors in flash sintering.

Gathered experience on fracture toughness testing of dental ceramics

BELLI, Renan; LOHBAUER, Ulrich

Within the modern mechanistic framework used to understand the fracture phenomenon in solids, the fracture toughness has established itself as the most important parameter for predicting critical crack sizes and/or stress magnitudes for safe/unsafe service. It is also the basis for understanding the relationships between subcritical crack growth and material resistance (R-curve effect), susceptibility to environmental effects and degradation processes. It is a material parameter of high utility for material development and structural design, if it were not for the sensitivity of mechanical measurements to a wide spectrum of variables that can rapidly affect result accuracy and precision. Much effort has been dedicated to standardize procedures, validate geometrical configurations and sort out influencing factors in order to improve testing methodologies.

Here we intend to share our experience accumulated over many years of validating testing procedures, optimizing practical protocols, minimizing sources of error and comparing methodologies. We focus on a wide variety of material classes, such as glasses, glass-ceramics and zirconias, with occasional incursions in technical ceramics such silicon nitride. We will discuss aspects that might help viewers to identify issues in testing protocols, select appropriate methods in view of circumstances related to application and material aspects. We address interpretation of results in the context of R-curve materials or K_{Ic}-materials.

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Understanding fracture in textured layered ceramics: from macro to micro scale

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Embedding textured alumina layers has been established as a novel design strategy to enhance the fracture resistance in ceramic laminates. It has been 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 MPam$^{1/2}$) 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 (e.g. under bending or thermal shock loading), 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. In this work, the benefit of textured microstructure against contact loading will be demonstrated and compared to equiaxed microstructures. The potential of using embedded textured layers to mitigate Herztian contact damage will also be addressed. Recent findings on the fracture resistance of grain boundaries between textured grains will shed light on understanding the fracture process of textured layered ceramics, which should be explored in light of novel processing techniques such as additive manufacturing.

M-O-M' interaction-controlled oxygen evolution/reduction reaction activity of double perovskite oxide bifunctional catalysts

BHARDWAJ, Aman; AYTUNA, Ziyaad; FISCHER, Thomas; MATHUR, Sanjay

Metal-air batteries are one of the prominent energy storage devices of the present need, and the bifunctional oxygen evolution-reduction (OER/ORR) reaction electrocatalyst is their core-technology component. The non-precious metal-based perovskite oxides and derivatives are prototype materials for these applications and offer high terrestrial availability and high stability. Their electronic geometry and structure are often considered key factors in controlling the catalytic activity. Herein, we demonstrate the impact of M-O-M' interactions in double perovskite oxide electrocatalysts over its bifunctional electrochemical activity towards the oxygen evolution and reduction reactions.
The materials synthesized through sol-gel chemistry were characterized for the physicochemical by various diffraction and spectroscopic techniques and further evaluated for electrochemical characteristics. A vibronic superexchange interaction resulting in a predominant M^{3+}-O-M'^{3+} interaction was found to positively impact the electrocatalytic activity by enhancing the current densities and lowering the Tafel slope. Our finding presents a promising pathway for developing advanced precious metal-free catalysts and substantiating their suitability as the bifunctional oxygen electrode for metal-air batteries.

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Improved semiconducting properties of Ba_2CuWO_6 for photovoltaics

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Thin film solar cells made of metal halide perovskites are attracting a lot of attention since power conversion efficiencies have increased rapidly over the past two decades. While prospective cheap and easy processing could be advantageous, problems such as toxicity and low stability will be difficult to overcome.

These drawbacks require new material combinations suitable for efficient, nontoxic and stable absorbers for thin film solar cells. Most of these properties can be found in the class of inorganic perovskites with formula ABO_3. However, they are mainly inefficient as absorber in solar cells due to their wide band gap and low charge carrier mobility. In this contribution, results from the investigation on the influence of substituting the B-site by more than two different cations on the properties of the ABO_3-perovskites will be presented. Therefore, important optical and electrical characteristics were analysed considering the synthesis conditions.

$Ba_2CuMo_xW_{1-x}O_6$ ($0 \leq x \leq 0.3$) was synthesized by solid state reaction and thin films were grown by pulsed laser deposition. Fundamental material properties were characterized by Rietveld refinement of diffraction data, UV-VIS-NIR spectroscopy, as well as SEM and EDX.

Commercial LTCC for thin-film deposition

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Low-temperature co-fired ceramics (LTCC) are used to fabricate multilayer circuits which are robust in harsh environments. Thick-film technology is well established for the metallization of circuit boards and microsystems. For specific sensor applications, the combination of LTCC and thin-film technology is advantageous to reach higher structure resolutions. Due to the high roughness of as-fired LTCC surfaces compared with silicon-wafers, the deposition of low-defect- films with narrowly specified properties is challenging. There is spare literature about thin films on commercial LTCC comparing different material systems or sintering techniques. For developing thin film sensors on multilayer circuits it is crucial to identify thin-film-compatible commercial LTCC material as well as the crucial surface properties. In this work we evaluate the thin-film capability of different LTCC surfaces.

The as-fired surfaces of free-sintered, constrained-sintered (sacrificial tape), and pressure-assisted sintered commercial LTCCs (DP951, CT708, CT800), as well as respective polished surfaces, were analyzed by tactile and optical roughness measurements and scanning electron microscopy. The thin-film capability of the LTCC surfaces was assessed by sheet resistance and temperature coefficient of resistance (TCR) of deposited Ni thin-film layers. Contrary to the expectations, no correlation between roughness and thin-film capability was found. Ni thin films on constrained sintered DP951 show the lowest sheet resistance and highest TCR within the experimental framework of the as-fired surfaces. The influence of surface morphology on the film properties is discussed.

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Self-generated Ba(Ce,Fe,Y)O_3?? composites for application in air electrodes of protonic ceramic fuel cells

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Protonic ceramic fuel cells (PCFCs) are based on proton-conducting oxides, offering the advantages of sufficiently fast ionic transport at relatively low temperatures (300-600°C) and significantly smaller activation energies than oxide ion conductors. With respect to materials development, improvement of proton uptake capacity and conductivity of potential PCFC air electrode materials is an important issue, in order to expand the active region for oxygen exchange beyond the gas/electrolyte/cathode triple phase boundary.

In our recent study, self-generated Ba(Ce,Fe,Y)$O_{3-?}$ composites were obtained by one-pot synthesis. Fundamental material properties of the composites were studied with regard to the application in air electrodes for PCFCs. X-ray diffraction and scanning electron microscopy showed that the materials consist of (i) an Fe-rich and (ii) a Ce-rich perovskite phase, in proportions depending on the precursor composition (Ce-Fe ratio). Elemental mapping with scanning transmission electron microscopy indicated that Y substitution influences the mutual solubility of Ce^{4+} and $Fe^{3+}/^{4+}$ in the Fe-rich and Ce-rich phase, respectively. Thermogravimetry showed that the proton uptake capacity of the composites is lower than expected, which could be ascribed to the limited incorporation of Y in the Ce-rich phase. Based on these results, strategies to overcome this effect by substitution with ions with similar radii, but different basicity, were developed.

Zirconia in dentistry : state of the art and current trends

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The use of zirconia in the dental field is booming for more than two decades. In this presentation, we will review the current use of zirconia as a dental ceramic and give some perspectives both for restorations (crowns and bridges) and implants. Zirconia can be found in a large variety of compositions, which gives different features from highly translucent, possibly shaded grades, towards tough and crack resistant modalities for demanding specifications. Zirconia can also be doped with several alternative elements in addition to the commonly used yttria, which may increase the portfolio of properties, as it is the case for example of Ceria-doped zirconia that present some ductility before failure thanks to Transformation-Induced Plasticity. Current developpements aim at improving the trade-off between strength, aging resistance and optical properties of zirconia dental restorations. These properties are often mutually exclusive, but we show that strategies such as grain boundary engineering or down-scaling the grain size to less than 100 nm are very powfertool tools to obtain dominant solutions in this trade-off. Strategies are also pursued to improve the tolerance of zirconia to defects and their ductility by improving their ability to transform under stress without compromising long-term stability. This may open the door to safer ceramic implants. Last, the progresses of additive manufacturing can lead to zirconia-based materials with complex shapes and architectures, but progresses should me conducted in terms of defects generated during the process and the possibility to make multi-materials and gradients.

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Bioactivity analysis of radiopaque glass 45S5 with progressive additions of Bi_2O_3: a dissolution study under static conditions

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The bioactive glass 45S5 (BG) allows a rapid ion exchange when it is exposed to a body fluid. Thus, biological processes, as bone mineral formation, are favored when BG is used in musculoskeletal implants. The integration of BG into hard tissue in in-vivo models may be evaluated through x-ray radiographs with enhanced image contrast to improve accurate minimal invasive diagnostics. Previously published works studied the influence of Bi2O3 on the radiopacity of BG. However, information on the effect of progressive additions of Bi2O3 up to 15 wt. % on the glass obtained by melt-quenching is missing and the role of Bi2O3 in the BG bioactivity needs to be further studied. This contribution shows the apatite formation ability and dissolution analysis of BG with progressive additions of Bi2O3 after 3, 7, 14, and 21 days of static immersion in simulated body fluid (SBF). The glass microstructure, and ion concentration in SBF after immersion were investigated through x-ray diffraction (XRD), scanning electron microscopy (SEM), inductively coupled plasma optical emission spectroscopy (ICP-OES), and ICP mass spectrometry (ICP-MS). The XRD and SEM results showed that higher amounts of Bi_2O_3 slowed the formation of apatite-like structures on the BG after immersion in SBF, as documented by the characteristics XRD patterns and morphology of precipitates. ICP analysis confirmed that the Si release was faster at a lower concentration of Bi_2O_3. On the contrary, the P intake rate from the SBF to the BG was inversely proportional to Bi_2O_3 content. The addition of the Bi_2O_3 reduces the apatite rate formation of the BG, increasing the glass stability and decreasing its dissolution rate. Nonetheless, BG with Bi_2O_3 still shows the ability to form apatite after 7 days.

A review on thermoplastic material extrusion-based additive manufacturing methods in the ceramic industry

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For the shaping of thermoplastic ceramic feedstocks, e.g. a feedstock consists of thermoplastic additives and ceramic powders, warm pressing, extrusion, injection molding and even dip-coating has been explored in the past. In comparison to dry pressing, the green density can be set more precisely and reproducibly, therefore geometrical size deviation after sintering is significantly lower. Another advantage is the higher green strength of thermoplastic shaping ceramic parts. Besides, thermoplastic-based ceramic feedstocks can be easily recycled and reused. However, the main disadvantage is the relatively long debinding times, compared to aqueous ceramic binder systems and ready to press powder. In addition, limited wall thicknesses can be achieved, due to diffusion limitation during the debinding processes. In the last years, the shaping of thermoplastic ceramic feedstocks was extended to material extrusion-based additive manufacturing (MEX-AM) method, successfully, but a lot of acronyms have been used in the past for the same processing method. In this talk, I will present an overview on different printers and extruder types, namely Bauden, direct-drive and screw-extruders for the shaping of ceramics.
In the beginning of the talk, I will look on the advantages and disadvantage of the different printer and extruder types for the ceramic processing. Later, material requirements and examples of 3D printed ceramic structures for commercial applications will be part of the talk.

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All-oxide photovoltaics for flexible devices by costeffective deposition methodologies

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Current photovoltaic technologies (PV) have led to important performance improvement and price reduction but they are still subjected to fundamental limitations such as a maximum power conversion efficiency, complex engineered multilayer architectures, materials instability and high energy payback time.

Ferroelectric oxide perovskites show an unconventional bulk PV mechanism which could result in power conversion efficiencies beyond the traditional solar cells. Here we will present our progress on the understanding of how cation composition in perovskite oxides and perform interface engineering modifies the optical and electronic properties in the ferroelectric BiFeO3 films combining x-ray diffraction, photoresponse, x-ray absorption analysis and compared to first-principles calculations.

Another relevant aspect in epitaxial ferroelectrics for PV is the difficulty to be fabricated in flexible and lightweight modules as these substrates do not meet the requirements for epitaxial growth and cannot stand high temperature treatments. We have developed a facile chemical route based on the use of Sr3Al2O6 sacrificial layer to detach oxide thin films of various compositions from the growing substrate and enable their transfer to flexible substrates. Therefore, from this work it is envisaged many new opportunities to prepare artificial oxide heterostructures and devices offering a whole new dimension for photovoltaics and beyond.

Reactivity of Nasicon type solid electrolyte during fast sintering process and versus high voltage spinel positive electrode material in temperature

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Nowadays, disruptive battery technology is necessary to improve energy and power densities of the batteries. Here we will focus on all solid-state batteries with solid inorganic electrolytes instead of conventional liquid ones. The challenges are multiple: choice of solid electrolyte (SE), management of solid-solid interfaces between electrodes and SE, reactivity at the interfaces, lithium ion conduction, but also the process to build these new batteries. The aim of the study is to understand the reactivity of $Li_{1.3}Al_{0.3}Ti_{1.7}(PO_4)_3$ (LATP) as SE during the sintering process especially by Spark Plasma Sintering (SPS), and versus high voltage positive electrode material. By SPS, depending on the sintering conditions, we highlight a change in colour from white to dark blue. Characterizations by X-Ray Diffraction, Scanning Electron Microscopy and ^{7}Li, ^{27}Al and ^{31}P Nuclear Magnetic Resonance have revealed a partial reduction of titanium with the presence of Ti^{3+}, but also changes into crystallite size depending on the sintering temperature and heating time. In parallel, we studied also the reactivity between LATP and the positive electrode material $LiNi_{0.5}Mn_{1.5}O_4$ (LNMO) in temperature. This study highlights difficulties encountered during the sintering of the SE and allows proposing degradation reaction of LATP upon SPS. Moreover, the reactivity between LATP and LNMO at a temperature as low as 500°C makes difficult the assembly of the all-solid-state stacking. These challenges will be discussed in detail, before proposing alternatives to stabilize these solid-solid interfaces in lithium metal batteries. The authors acknowledge Nano One Materials Corporation for providing the LNMO material used for the project.

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Deformation and fracture of high-entropy carbide grains at the micro-scale

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Deformation and fracture behaviour of a (Hf-Ta-Zr-Nb)C high-entropy carbide grains of different orientations were investigated by nanoindentation, micropillar compression and micro-cantilever bending tests. The sample was prepared by spark plasma sintering and the crystallographic orientation of grains was determined by electron backscatter diffraction (EBSD). Micropillars and micro-cantilevers were milled out from grains of specific orientations, based on the EBSD maps, using a focused ion beam (FIB) technique. Nanoindentation tests exhibited a significantly enhanced hardness (36.1±1.6 GPa,) compared to the hardest monocarbide (HfC, 31.5±1.3 GPa) and the binary (Hf-Ta)C (32.9±1.8 GPa). Micropillar compression test of {001} oriented grains revealed that (Hf-Ta-Zr-Nb)C had a significantly enhanced yield and failure strength compared to the corresponding base monocarbides, while maintaining a similar ductility to the least brittle monocarbide (TaC) during the operation of {110}<1-10> slip systems. It was found that the deformation behaviour of cantilevers depends mostly on their defect structure (i.e. pores), while the grain orientation ({001} and {101}) has only a slight influence on that. About half of the microcantilevers exhibited plastic behaviour before failure and fractured at their fixed end ('defect free' beams). The other 'defected' beams which were broken further from the fixed end, exhibited a brittle linear fracture with visible granular shape pores on the SEM images. The real fracture strength of all micro-cantilevers was calculated to 12.59 ± 3.2 GPa. According to the analysis of EBSD and SEM images, the fracture surface was found to be the (001) type plane for both types of micro-cantilevers.

Frequency dependence on the sintering and microstructural evolution of gadolinium doped ceria during AC electric field sintering

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The similarities and differences between DC and AC flash sintering are not well investigated so far. Here, AC electric field assisted sintering of gadolinium doped ceria (GDC) ceramics was carried out with amplitudes varying from 10 V/cm to 40 V/cm and a wide spectrum of frequencies ranging from 0.1 to 1000 Hz in logarithmic intervals and compared to DC field conditions. The onset temperature for densification with DC and AC (various frequencies) electric field are bolstered by the asymmetricity of the electro-chemical reduction of ceria during the sintering process. The back and forth diffusion of defects during polarity reversal may contribute to the net reduction in the mean free path of the defect propagation resulting in varying incubation time for different frequencies. Microstructural analysis of the GDC ceramics sintered under different frequencies indicates towards the sintering behavior which may be different for ceramics sintered under DC, AC (low frequency, <10 Hz) and AC (high frequency, >100 Hz).

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Manufacturing, characterization and testing of novel temperature-resistant 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 new upper stage propulsion systems were performed at the DLR Institute of Space Propulsion in Lampoldshausen including a novel uncooled C/C-SiC nozzle extension. The green body was produced by wet filament winding. Following the preform was ceramized to the C/C-SiC state via liquid silicon infiltration. Since delaminations are a major concern of wound CMCs structures, the multi angle fiber architecture, which was used here, mitigates this risk. Computer tomography scans, which were carried out at all stages of the processing, show the changes of the component through each step. Furthermore, the quality of the material formed was determined by microstructure analysis using SEM. The structural integrity and thermal stability were tested by hot gas firing tests under laminar and separated flow conditions. The test proved that the ceramic nozzle shows the advantages of the high mass specific characteristic values and a sufficient high temperature resistance under extreme conditions.

Additive manufacturing of oxygen transport membranes - 3D-OTM

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Ceramic oxygen-permeable membranes are capable of efficiently separating oxygen from air. The separated oxygen can be fed directly to chemical reactions (membrane reactors). This combines the separation and reaction processes that are usually carried out separately (process intensification). In the context of the energy transition, such technologies offer very good prospects, as they can support the electrification of the chemical industry in addition to increasing efficiency.

The very stable perovskite $SrTiO_3$, which, however, is not O_2-permeable and therefore has to be functionalized by means of dopants, was the selected material in this work. By partially substituting Ti with 25-35% Fe ($SrTi_{1-x}Fe_xO_{3-\delta}$, STF), a good permeation rate could be achieved along with excellent stability. A solid-state reaction route for powder synthesis as well as reactive sintering of the membrane material was studied.

Additive manufacturing techniques were used to produce a membrane component in tubular geometry as a first step. The developed test specimen were quantitatively evaluated on the basis of their performance with regard to the separation of pure oxygen from air. It was shown that the performance is consistent to classically manufactured discs. Based on these positive results, complex shaped reactors can be produced using the benefits of additive manufacturing.

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Application of a novel ceramic material in a prototype tilting pad thrust bearing for harsh service environments

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Product-lubricated pumping applications require extremely robust bearings, especially in the case of sand-loaded lubrication fluids. For high power/high speed pump applications, tilting pad bearings are the preferred bearing solution, due to their superior rotordynamic characteristics. For product-lubricated applications, materials with good corrosion and abrasion resistance characteristics have to be used. The lubricant for the bearing in these applications is often a water based fluid (e.g. sea water) with low viscosity and with particle contamination (e.g. sand). Existing tilting pad product lubricated bearings (PLB) solutions have the issue that catastrophic failures tend to occur under severe conditions or that they are limited in available size (e.g. polycrystalline diamond (PCD) bearings). A tilting pad thrust bearing and a thrust collar made of a novel ceramic material with improved tribological properties was designed, manufactured and tested for use in such applications. This new ceramic bearing offers several advantages over existing PLB materials.

- Lower coefficient of friction and reduced start-up torque
- Excellent dry-running capabilities
- Increased reliability and robustness
- Increased wear and abrasion resistance
- No size limitation due to manufacturing constraints such as for PCD bearings

The novel ceramic material is a Diamond-SiC composite, and the material has been tested extensively as per its tribological and wear/erosion characteristics before its use and application in a prototype thrust bearing and thrust collar.

Bioactivity, biocompatibility and antibacterial activity of polymer-derived biosilicate-c composite foams

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Carbon and its polymorphs have received great attention in bone-tissue engineering applications. In addition, due to their absorption properties, carbon materials, for instance graphene oxide, have been used in cancer photothermal therapy/ photodynamic therapy. It was observed that polymer-derived Biosilicate-C composite foams can rapid heat up to 70°C after under IR. The observed heating temperature constitutes importance on bacteria disinfection since common bacteria are disinfected upon heating above 55 °C. When fired in inert atm, silicones (i.e. H44, Silres MK), transform into amorphous silicon oxycarbide featuring both Si-O and Si-C bonds along with free carbon nanosheets. In addition, firing silicones in air yields pure SiO_2. By mixing silicones with suitable fillers bio-ceramics with a well-defined crystalline phase such as Biosilicate glass-ceramics can be prepared. In this study, Biosilicate - C composites were fabricated in the form of highly porous foams by the polymer-derived ceramic route, and their biological response was analysed. Two different commercial silicone polymers (a poly-methyl-siloxane, MK, and a polymethyl-phenyl-silsesquioxane, H44) were used as a source of silica, and mixed with the fillers, which served as the sources of Na_2O, CaO, and P_2O_5. The samples were heat treated either in air or in N_2 to obtain products resembling Biosilicate glass-ceramic embedded in a silico-phosphate glass matrix with and without free carbon. All fabricated samples exhibited in-vitro bioactivity when immersed in SBF as well as antibacterial activity against S. aureus and E. coli. Direct contact cell viability test indicated a lack of cytotoxicity of both samples with and without carbon after WST-8 assay

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Wear characteristics of a (Hf-Ta-Zr-Nb-Ti)C high-entropy carbide

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Single-phase, high purity rock salt structured (Hf-Ta-Zr-Nb-Ti)C high- entropy carbide with a high relative density of 99.4 % was prepared by ball milling and a two-step Spark Plasma Sintering process, The chemical composition of grains was homogeneous and their size ranges from 3 μm to 25 μm. Tribology measurements were carried out using a ball-on-flat technique, dry sliding in air, with SiC tribological partner.The deformation, fracture and wear damage were studied by scanning electron microscopy and confocal electron microscopy. Focused Ion Beam technique was used for cutting a wedge into the material to study the cross-section under the wear surface of the investigated material. The dominant wear mechanism was mechanical wear with limited grain pull-out and fracture, and with a localized and thin tribo-layer formation. The coefficient of friction was similar for all loads after 300 m, approximately 0.41. With increasing load, the wear rate exhibited an increasing tendency from 2.5 10^{-6} mm^3/Nm at 5 N to 9.0 10^{-6} mm^3/Nm at 50 N.

Synthesis of La_2NiO_4+? ceramic particles by molten-flux method

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Due to its high chemical stability in CO_2 atmosphere and its anisotropic crystal structure, the Ruddlesden-Popper phase La_2NiO_{4+}***?*** has attracted considerable attention in the field of oxygen-transporting membranes. To prepare ceramic La_2NiO_{4+}***?*** membranes with an asymmetric microstructure, the templated grain growth method can be used. For this, large anisotropic plate-like La_2NiO_{4+}***?*** template particles are required. Since the morphology of the templates plays an important role in the preparation of textured membranes, they can be obtained via a molten-flux synthesis. This method enables the production of powders with anisotropic particles at lower temperatures. Here, we report on the synthesis of plate-like La_2NiO_{4+}***?*** particles from NaOH flux. For this purpose, a mixture of La_2CO_5 and NiO from a sol-gel process were used as reactants, which were then mixed with NaOH and water in crucibles and heated to 400 °C for 6-18 hours. The chemical reactions during the synthesis were investigated by thermogravimetric analysis. Powder X-ray diffraction patterns indicate a K2NiF4-type tetragonal structure with I4/mmm space group for the product powder. Scanning electron microscopy showed plate-like La2NiO4+? particles with lateral sizes of 1-30 µm and varying aspect ratios while energy-dispersive X-ray spectroscopy revealed a homogeneous distribution of lanthanum and nickel. The influence of processing parameters on size and morphology of the La_2NiO_{4+}***?*** particles was systematically studied.

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Understanding and engineering dislocations in oxides at room temperature

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Dislocations are line defects and major carriers of plastic deformation in crystalline materials. So far the understanding of dislocations in ceramics is less developed contrasting the advanced studies in metallic materials. In recent years, in light of the increasing interest in dislocation-tuned functional properties of oxides, the dislocation-based mechanical behavior, for instance, dislocation-mediated plasticity and dislocation-based toughening have also become highly relevant. Due to the brittle nature of oxides, it remains a great challenge to introduce dislocations with controlled structures without forming cracks, particularly at room temperature. Understanding the dislocation-based mechanics in oxides plays a critical role in tackling such a challenge as well as assessing the mechanical and functional reliability of future dislocation-based devices. In this talk, we focus on room-temperature dislocation mechanics in oxides and present a roadmap to introduce dislocations and understand the dislocation behavior (dislocation nucleation, multiplication, and motion), and ultimately to tailor the dislocation-mediated plasticity in a model oxide, SrTiO3, by combining nanoindentation, micro-pillar compression, bulk indentation tests across the length scales. Several useful concepts and approaches for experimental design such as cyclic indentation tests, surface dislocation engineering, and thermal treatment will be demonstrated to tailor the dislocation plasticity and, more strikingly, the hardness and fracture toughness of the crystal. The proofs-of-concept on $SrTiO_3$ will be further validated on other oxides to showcase the general applicability of the findings.

Sintering of functional lead-free ceramics by Cool-SPS

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Nowadays, materials industry has to face many challenges to lower its environmental footprint and looks to reduce the sintering time and temperature for the production of ceramics, as well as the use of controversial chemical compounds such as lead in functional materials. Many new sintering methods are being developed to sinter ceramics at low temperature such as Cold Sintering or reactive hydrothermal liquid-phase densification etc… However, for materials such as oxides, a post treatment is frequently required to obtain a phase pure and fully densified ceramic. It has been proven previously that Cool-SPS can be used to obtain highly densified ceramics of thermodynamically fragile materials at low temperature and high pressure, with an adapted sintering strategy taking advantage of the many degree of freedom of Cool-SPS for successful densification. In the present work, we explore the Cool-SPS processing of lead-free functional perovskites. Several ceramics from this large family were rapidly and successfully densified by Cool-SPS, with final relative densities ranging from 80% to 95%. Experimental parameters, such as degassing and displacement, were studied in order to investigate and optimize the sintering path. The evolution of these parameters illustrates steps like chemical reaction and densification and are likely linked to the relative density of the final ceramic. Moreover, the effect of the Cool-SPS conditions on the microstructures and dielectric properties of the final materials was investigated. In the end, we will demonstrate that Cool-SPS allows for the elaboration of dense ceramics of lead-free functional perovskites, paving the way towards more sustainable materials and processes.

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Hard, tough, electrically conductive ceramics based on boron carbide

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Boron carbide (B_4C) is an advanced non-oxide ceramic and possesses a set of outstanding properties such as high melting point, high mechanical strength, high thermal stability, extreme hardness and high neutron absorption cross section. These outstanding properties make B_4C for various industrial applications attractive, including refractory devices, wear resistance enhancement, hard material polishing and reactor control in nuclear technology. In particular, the combination of its extraordinary high stiffness (E = 450 GPa) and low specific weight (2.52 g/cm^3) produces an outstanding stiffness/weight ratio compared to metals and other ceramic materials like Al_2O_3, SiC and Si_3N_4. Consequently, boron carbide is an ideal ceramic material for minimizing the weight of structural elements in lightweight applications. Nevertheless, one obstacle for its wide application is the low fracture toughness K_{IC}, typically in the range of 2 - 3 MPa?m. A strategy increasing K_{IC} of boron carbide is to produce composites by addition of suitable secondary phases to B_4C matrix.
In this work, B_4C-TiB_2 composites were fabricated via reactive pressureless sintering of B_4C and TiC powder mixtures. The effect of in-situ formed TiB_2 phase on sintering behavior, mechanical properties and electrical conductivity of boron carbide were studied. It is shown that dense B_4C-TiB_2 composite with high fracture toughness up to 4.7 MPa?m and in combination with high hardness could be achieved. In addition, the electrical conductivity of the materials can be modified from between semiconductive and metallic behavior by the TiB_2 content. B_4C-TiB_2 composites offer opportunities for new applications, like lightweight constructive component, ballistic protection and high temperature electrode with the improved properties.

Porous alumina ceramics with multimodal pore size distributions

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Pore networks with multimodal pore size distributions combining advantages from iso? tropic and anisotropic shaped pores of different sizes are highly attractive to optimize the physical properties of porous ceramics. Multimodal porous Al_2O_3 ceramics were manufactured using pyrolyzed cellulose fibers (l = 150 ?m, d = 8 ?m) and two types of isotropic phenolic resin spheres (d = 30 and 300 ?m) as sacrificial templates. The sacrificial templates were homogeneously distrib?uted in the Al_2O_3 matrix, compacted by uniaxial pressing and extracted by a burnout and sintering process up to 1700 °C in air. The amount of sacrificial templates was varied up to a volume content of 67 Vol% to form pore networks with porosities of 0-60 Vol%. The mechanical and thermal prop?erties were measured by 4?point?bending and laser flash analysis (LFA). Based on ?CT?measurements, the representative volume?of?interest (VOI) of the samples digital twin was determined for further analysis. The interconnectivity, tortuosity, permeability, the local and global stress distribution as well as strut and cell size distribution were evaluated on the digital twin's VOI. Based on the experimental and simulation results, the samples pore network can be tailored by changing the fiber to sphere ratio and the overall sacrificial template volume.

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Vorstellung des TFA 6.1 "Charakterisierung poröser Keramiken"

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Der Teilfachausschuss 6.1 "Charakterisierung poröser Keramiken" wurde 2016 gegründet, um den wachsenden Ansprüchen und Anforderungen seitens der Industrie hinsichtlich der Herstellung, Charakterisierung sowie Simulation poröser Keramiken zu entsprechen. Neben dem Austausch zwischen Industrie und Hochschulen steht auch die gemeinsame Forschung im Vordergrund. Im Rahmen eines gemeinsamen Ringversuches der Ausschussteilnehmer wurden definierte Proben mit unterschiedlichen Verfahren hinsichtlich mechanischer und thermischer Eigenschaften sowie der Mikrostruktur untersucht. Die Ergebnisse des Ringversuches werden auszugsweise vorgestellt.

Novel coating technologies for multilayer investment casting molds based on calcium zirconate for titanium alloys

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Investment casting enables to produce near-net shaped complex metal components required for advanced applications. Especially the casting of titanium alloys is challenging due to the reducing melt and the high melting point leading to undesired alpha case formation. Recently, corrosion resistant shell molds consisting of calcium zirconate and a silica-free binder were generated by dip-coating according to the lost-wax process. Conventional dip-coating, however, can adversely affect the surface quality. Thus, spraying and centrifugation were applied as alternative coating technologies for the first layer, being in contact with the melt during casting. Alginate gelation was used additionally to enhance green strength by ion exchange. The homogeneity of the alginate-based spray coating was revealed by scanning electron microscopy and computed tomography. Spraying was favored for the production of multilayer shell molds since it provided the highest flexibility for the required complex shapes. Functionally graded molds had an improved surface smoothness with pores in the lower µm range. Cracking of the shell molds was prevented by an intermediate layer between the high-shrinkage spray coating and the lower-shrinkage dip-coating layers as well as alternating dip-coating layers. The microstructure exhibited a bridging zone and homogeneously distributed coarse grain across the diameter. By casting Ti6Al4V, defect-free components without alpha case layer were obtained.

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Dislocation-tuned electrical properties of oxide ceramics

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The defect chemistry of ceramics is usually modified by controlling point defects and interfaces. Dislocations as one-dimensional line defects have so far been mostly disregarded as defects for modification of functional properties but are finding increasing attention recently. Based on the model materials TiO_2, $SrTiO_3$, and yttria-stabilized zirconia, we show that mechanically introduced dislocations can enhance the electronic or ionic conductivity. This illustrates the opportunity to tune ceramics beyond what can be achieved by chemical doping, which is hampered by the solution limit of the dopants. The effect of dislocations on the functional properties of ceramics depends on a convolution of dislocation character, core properties, possibly existing space charges, and mesoscopic structure. The control over the mesoscopic dislocation structure and its behavior at elevated temperatures is complex but a key step in using dislocations as one-dimensional dopants. This opens the door for multiple modification possibilities, such as anisotropic conductivity change in semiconductors and solid electrolytes.

Dielectric breakdown strength investigation with a novel indentation method

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Dielectric breakdown strength (DBS) is defined as the maximum voltage divided by the sample thickness that an investigated insulator can withstand. The fundamentals have been already defined in the last century; however, the mechanisms of dielectric breakdown are still not completely understood. From empirical studies, it is known that material microstructure, particularly its imperfections, profoundly influences the DBS. Large pores, secondary phases, electrode precipitation, etc., can decrease the DBS. Additionally, the measurement setup along with sample geometry can affect the DBS similarly. In particular, the concentration of the electric field and mechanical stresses at the edge of the electrode can reduce the DBS.

In order to avoid such a concentration of stresses and control the microstructure, a new sample preparation method for the investigation of DBS is proposed. In our approach, the upper electrode is a concave indentation generated in the green sample state, which is the viscoplastic regime of a tape-casted sample. With such geometry, the concentration of the high electric fields at the electrode edges is avoided, while the position of the breakdown spot can be foreseen, and the microstructure underneath is known. The reliability of the method was verified on $BaTiO_3$, a prototype materialy used for ceramic capacitors. The results of the DBS investigation show that in most cases, the breakdown occurres in the center of the concave indentation. Moreover, the variations of the thickness and voltage ramp have a significant influence to the DB, as proposed in the literature. In the case of $BaTiO_3$, the DBS differed by up to 60 % and 30 %, with the change of thickness and voltage ramp, respectively.

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Novel approaches in additive manufacturing of glass-based structures

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Although recycling is considered a default method of glass waste management, various types of glasses are still landfilled. Flame synthesis can convert such glass into microspheres (solid, hollow or porous), which are used as fillers in inks in additive manufacturing techniques. Due to the spherical shape the influence of microspheres on the viscosity of ink is minimal and high solid loadings are achieved. Translucent, porous scaffolds were prepared by viscous flow sintering of green preforms fabricated by direct ink writing (DIW) and masked stereolithography of the microspheres with suitable organic additives. Microspheres with åkermanite stoichiometry were used in AM of porous highly crystalline scaffolds for biomedical applications. The rapid crystallization of the system limited viscous flow sintering, resulting in components with complex porosity and promising mechanical properties. Alternatively, engineered photocurable silicone-based binders mixed with glass powders were used in digital light processing of reticulated glass-ceramic scaffolds for tissue engineering applications. The silicone component converted into silica upon firing stabilized the 3D-printed shapes during sintering and prevented softened glass from excessive viscous flow. Silicone resins, filled with phosphates and other oxide fillers, yielded upon firing in air a product resembling Biosilicate® glass-ceramics, one of the most promising systems for tissue engineering applications. The process required no preliminary synthesis of parent glass, and the polymer route enabled the application of direct ink writing (DIW) of silicone-based mixtures for the manufacturing of reticulated scaffolds at room temperature.

Advanced technologies for the manufacture of future piezoceramic components

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The trend towards complex structures or highly miniaturized devices demands for new technology solutions to manufacture piezoceramic components. Piezoceramic thick films with typical thickness of 20-150 µm fulfil microsystems requirements for low profile and compact devices. Using screen-printing technology, net-shaped structures can easily be applied together with isolation and electrode layers. Miniaturized and highly compact devices are possible, which are needed as sensors for process monitoring in context with industry 4.0 applications, actuators for optical control and ultrasonic transducers for non-destructive evaluation.

Ultrasonic imaging plays an important role in medical and material diagnostics. The demand for more precise control requires higher resolution, which can only be obtained using high-frequency ultrasonic transducers. The soft mold process developed at Fraunhofer IKTS enables the fabrication of fine-scale 1-3 piezocomposites via slip casting. Based on this technology, ultrasonic transducers with operating frequencies up to 40 MHz can be manufactured.

Piezoceramic fibers allow for the development of customized sensors, actuators, and ultrasonic transducers. Using a phase inversion process, piezoceramic fibers with diameters of 100 to 800 µm can be produced on large scale. They can be integrated into polymer matrices to build piezofiber composites. While piezocomposites with aligned fiber arrangement are advantageous for actuators and ultrasonic transducers, sensors and energy harvesters also accept random arrangement.
The presentation will give an overview covering design aspects, technology, and applications of piezoceramic components beyond conventional powder pressed parts.

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Outstanding lead-free high-temperature multilayer ceramic capacitors based on $Na_{0.5}Bi_{0.5}TiO_3$

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The capacitor as passive component is utilized in a vast variety of applications like aerospace, electric mobility, power transfer in renewable energy technologies, and even portable smart devices. These red-hot topics seek for persistent miniaturization at constant or improved performance as well as paramount reliability. Additionally, capacitors must endure harsh temperature conditions ranging from -50 to more than 300 °C by maintaining stable dielectric properties. Advanced factory-made capacitors can be operated only to temperatures up to 150 °C. Lead-free $Na_{0.5}Bi_{0.5}TiO_3$ (NBT)- and $BaTiO_3$ (BT)-based solid-solutions are promising materials fulfilling the demanding requirements for high-temperature capacitors in latter applications. The complex defect chemistry hampered the research for appropriate capacitor materials. However, we developed the excellent material system NBT-BT-CZ-BA which massively extends the temperature range of stable dielectric properties from -68 °C to 391 °C. In this study the aforementioned system was used to successfully fabricate multilayer ceramic capacitors (MLCC) with 70Ag/30Pd inner electrodes by tape casting and co-firing processes. Each capacitor consists of five dielectric layers with an individual sheet thickness of 45 µm. The MLCCs boast with enormous temperature-independent permittivity, low dielectric loss, high energy storage density and dielectric breakdown strength. Furthermore, the study revealed challenging interactions between used materials during tape casting and MLCC fabrication. The excellent results convincingly demonstrate that NBT-based MLCCs are game-changing and viable candidates for high-temperature multilayer ceramic capacitors.

Advanced method for determination of ceramic lifetime and method-based fault diagnosis for sustainable filtration applications

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

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LLZO free-standing ceramics and inks: Scalable electrolyte processing for solid-state batteries

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The substitution of liquid electrolytes with solid ionic conductors is a promising strategy for a new generation of safer batteries with higher energy densities. In terms of developing components for All Solid-State Batteries, plenty of research focused on optimization of solid electrolyte properties. Garnet materials, especially doped $Li_7La_3Zr_2O_{12}$ (LLZO), are considered a very promising class of solid electrolytes due to their high lithium-ion conductivity in the range of 10^{-4} S/cm and their compatibility with metallic lithium as anode. Recently, the combination of inorganic electrolytes with conducting polymers to form hybrid materials has also come into focus. Scientific research mainly addresses material properties and characterization, but less manufacturing methods and cell assembly. Fraunhofer IKTS is investigating different scalable manufacturing processes for All Solid-State Batteries in several projects. We have developed a process chain for synthesizing our own LLZO and produce freestanding LLZO separator sheets with high ionic conductivity, and we have started the processing of LLZO inks for rapid printing of electrolytes or as conductive additives. This talk will give an overview about the material's synthesis, slurry preparation, tape casting, lamination, and sintering, as well as ink development and printing. Some of the challenges encountered during the development will be included, too.

Conductivity, microstructure and mechanical properties of tape-cast LATP with LiF and SiO_2 additives

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LATP sheets with LiF and SiO_2 addition prepared by tape casting as electrolytes for solid-state batteries were characterized regarding conductivity, microstructure and mechanical properties aiming towards an optimized composition. The use of additives permitted a lowering of the sintering temperature. Rietveld analyses of the samples with additives revealed a phase mixture of NaSICON modifications crystallizing with rhombohedral and orthorhombic symmetry as a superstructure with space group Pbca. It seems that LiF acts as a sintering additive but also as a mineralizer for the superstructure of LATP. As general trend, higher LiF to SiO_2 ratios led to lower porosities and higher values of elastic modulus and hardness determined by indentation testing, but the presence of the orthorhombic LATP leads to a decrease of the ionic conductivity. Micro-pillar testing was used to assess the crack growth behavior revealing weak grain boundaries.

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Small-scale mechanical characterization of $LiTaO_3$ and $LiNbO_3$ single crystals for SAW filters

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The requirements on new materials used in mobile communications are driven by the demand for higher data transfer rates. Single crystalline piezoelectric materials such as $LiTaO_3$ and $LiNbO_3$ have thereby qualified as substrates for precise and efficient frequency filters and are consequently employed in the newest 5G mobile communication network standards. Crystal growth in specific orientations are usually pursued to ensure optimized functional properties. The question rises whether such orientations may withstand thermo-mechanical loading during qualification and/or service.

In this work, biaxial strength measurements along with in-situ SEM fracture toughness experiments were performed on miniaturized specimens to investigate the mechanical performance of $LiTaO_3$ and $LiNbO_3$ materials. Atomistic modelling calculations supported the experimental results and provided recommendations for crystal orientations with enhanced mechanical properties. In addition, nanoindentation experiments and preliminary in-situ TEM tests showed onset of plastic deformation for specific loading scenarios. These findings at the micro-scale may be utilized in the fabrication of $LiTaO_3$ and $LiNbO_3$ materials of particular orientations with optimized structural and functional properties.

Ceramic filter discs for ultra-/ and nano-filtration applications

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Inopor is currently the only supplier who can deliver ceramic nanofiltration membranes with a low cut-off up to 200 Dalton. These are successfully used in a broad range of applications for example in the food and beverage filtration (wine, beer, fruit juice), water and wastewater treatment or for other different issues of the chemical and biotechnology industry. The team of Rauschert/Inopor and Fraunhofer IKTS is participating in different development projects. Results of two projects and potential applications will be presented:

1. Status of project "PharMem - Development of a process for the elimination of pharmaceuticals and other organic trace substances from wastewater with a combined system consisting of functionalized ceramic nanofiltration membranes and advanced oxidation" – Program EFRE (Thüringer Aufbaubank), funded by the European Union. Rauschert and Fraunhofer IKTS are developing ceramic membranes for ultra- and nanofiltration on geometry types like tubes, discs and multichannel plates. The treatment of with pharmaceuticals contaminated wastewater is aimed.

2. Status of project "ReWaMem - Recycling of laundry wastewater to reuse the wastewater using ceramic nanofiltration" – Program WAVE II (Water technologies: reuse) funded by the BMBF (02WV1568A). The ceramic ultra- and nanofiltration membranes on geometries like novel tubes, discs and multichannel plates will be further developed. The treatment of laundry wastewaters of mats and towels is aimed.

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Silicon carbide - graphene composites with high thermal and electrical conductivity

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Almost fully dense silicon carbide composites with 1 vol.% equimolar Y_2O_3–Sc_2O_3 sintering additives and with different amount of graphene nanoplatelets (GNPs) from 1 to 10 wt. % or 1 wt. % of graphene oxide (GO) were sintered in rapid hot press (RHP) at 2000°C for 30 min with a pressure of 50 MPa under nitrogen atmosphere. The density of SiC/GNP and SiC/GO slightly decreased from 99% to 97% with increasing GNP or GO content from 1 wt.% to 10 wt.%. Some of the sintered materials were further annealed in gas pressure sintering (GPS) furnace at 1800 °C for 6 h in overpressure of nitrogen (3 MPa). Electrical conductivity and thermal conductivity were investigated as a function of amount of graphene, its orientation in SiC matrix and effect of annealing. The electrical conductivity of reference SiC sample (17 S×cm^{-1}) gradually increased with increasing GO or GNP content, reaching the highest value of 67 S×cm^{-1} for SiC with 10 wt.% GNPs. Remarkable improvement of electrical conductivity was achieved by annealing the samples in N_2 atmosphere and the highest value of 118 S×cm^{-1} was obtained for the sample with 10 wt.% GNPs. The highest thermal conductivities were obtained at room temperature in parallel direction to GNPs for annealed SiC samples with 1% GO ($\boldsymbol{l}$ = 238 W×m^{-1}K^{-1}) and 5% GNPs ($\boldsymbol{l}$ = 233 W×m^{-1}K^{-1}). The obtained results show that application of freeze granulation, rapid hot-pressing and annealing of samples at 1800 °C for 6h in N_2 atmosphere allows to obtain SiC ceramics with very high electrical and thermal conductivity. Acknowledgements: This work was supported by the Slovak grant VEGA 2/0007/21.

Surface bioactive silicon nitride modification by oxyacetylene flame treatment

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Silicon nitride-based ceramics with sintering additives (CaO, SiO_2, $Ca_3(PO_4)_2$), have been prepared to study the bioactivity. Dense ceramic samples were oxidized by an oxy-acetylene flame at approx. 1475 °C for 1 min, to modify the surface in terms of bioactivity enhancement and the formation of suitable porosity for cell viability and attachment. During oxidation two parallel processes occurred on the ceramic body surface: (i) formation of thin glassy layer with a composition close to that of grain boundary phase in ceramic body, and (ii) partial decomposition of silicon nitride matrix. The latter one resulted in the formation of gases (N_2 and SiO), which formed bubbles in the viscous surface glassy phase, resulting in porosity required for cell adhesion (small pores) and tissue ingrowth (large pores). The best bioactivity was observed for oxy-acetylene flame treated Si_3N_4 ceramics with $Ca_3(PO_4)_2$ as sintering additive.

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Advanced materials for energy conversion

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Few issues will concern humanity and future generations as much as climate change. To counteract this, new materials and methods are needed in many respects. One decisive sector that is largely responsible for global warming is the energy sector. The most important aspects to focus on are energy storage, transport and conversion. In this contribution, I will give an overview about recent developments in functional ceramics for energy conversion. Electromechanical energy conversion is a potential energy source for low power applications such as sensors or active vibration damping. This technique is based on the piezoelectric effect and can be realised with ferroelectric materials such as barium titanate. Using a revolutionary material design based on additive manufacturing with capillary suspensions, we recently increased the characteristic figure of merit by almost 500% compared to dense materials. By optimising the material for energy conversion, it will be possible to increase the efficiency by orders of magnitude. Since the material parameters are strongly dependent on grain size, we investigated the grain size effect on barium titanate with various techniques. Especially the in-situ characterisation with an electric field based on the STRAP method provides detailed information on the electromechanical processes. This technique can also be applied to solar energy conversion materials to elucidate the structure-properties relationships.

$Li_7La_3Zr_2O_{12}$ solid electrolyte sintered by the ultrafast high-temperature method

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All-solid-state Li batteries (ASBs) are regarded as the next battery generation. Garnet-type $Li_7La_3Zr_2O_{12}$ (LLZ) is one of the most promising oxide-ceramic based solid electrolytes (SE). However, its integration into ASBs, especially mixed cathodes, is hampered by the required high sintering temperatures for extended dwell times. These temperatures are essential to form mechanical stable battery components with suitable electrochemical properties but also form side phases with common cathode active materials (CAMs). Advanced sintering techniques with lower sintering temperature and predominantly shorter dwell times can significantly lessen thermodynamic limitations, and prevent side phase formation and loss of volatile elements such as Li. In light of this, we demonstrate that ultrafast high-temperature sintering (UHS) can be used to sinter bulk LLZ pellets without the need of sintering additives within seconds, kinetically inhibiting side reactions. The UHS applies an electric current to heat a carbon felt by Joule heating within a second to the sintering temperature. The heat is rapidly transferred to the pressed powder pellet by conduction and radiation, so that the sample placed in intimate contact inside the felt sinters within 10 seconds. Although the sintering process is short, a mechanically stable, phase pure, and sufficiently dense LLZ with sufficient ionic conductivity is obtained. The introduced UHS process is predestined for co-sintering of LLZ and CAMs, as it could help to overcome thermodynamic limitations and avoid the formation of a diffusion-based secondary phase. Hence, UHS has the potential to enable CAM/SE combinations which were unsuitable so far, and to pave the way for a completely type of ASBs.

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Hydroxyapatite scaffolds printed by fused filament fabrication

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Hydroxyapatite (HAP) as natural constituent of human hard tissues is generally considered to be good biomaterial with sufficient biocompatibility but lesser bioactivity due to its low solubility. This disadvantage was improved by preparation of biphasic composite materials prepared from HAp and better soluble tricalcium phosphate (TCP). It can be considered for an interesting feature of the HAp to decompose at high temperature reactions to defect HAP and TCP phases. This behavior was investigated in a more detailed way for HAP scaffolds produced by 3D printing from thermoplastic composite. Scaffolds with different size of interconnected pores were prepared using fused filament fabrication method and sintered in the temperature range of 1200 – 1400 °C. The temperature range was selected according to favorable results of in vitro cytotoxicity tests. The reason for improved bioactivity is based on chemical and structural changes of HAP at elevated temperatures used for sintering. The FTIR results show gradual decrease of integral intensity of structural OH stretching vibration indicating dehydroxylation reaction which was not completed even for HAP sintered at 1400°C for 2 hours.

A low-cost approach for oxide-FRCMC with tailored structure

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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 performance and reliability at elevated temperatures, e.g. fibre-reinforced ceramic composites (FRCMCs). In the past, we´ve developed at TUHH a processing route for FRCMC based on the lamination of thermoplastic prepregs which allows not only affordable manufacturing but also precise tailoring of the internal structure (both intra-bundle and inter-textile) as well as the surface topography of complex shaped products.

Basically, the route integrates processing techniques well known in the ceramic community: (a) infiltration of fibre-bundles and -textiles with low viscous suspensions, (b) manufacturing of 2 D prepregs using commercial fibre fabrics impregnated with compounds of ceramic particles embedded in an organic matrix, (c) followed by respective stacking, (d) burn out of the organic matrix, (e) preconditioning for subsequent coatings, (f) dip-coating of the compound with highly doped and therefore sinter-active suspensions and (g) final heat treatment to sinter both the FRCMC as well as the coating in one step.

In this presentation the focus is on Al_2O_3-matrix materials reinforced using NEXTEL 610 textile denier 1500 and – in case of model composites – single crystalline SAPHIKON fibers. ?-Al_2O_3 as well as Al/Al_2O_3 based mixtures are used for matrix synthesis, the later one in order to achieve matrix consolidation by reaction derived in-situ volume expansions.

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Cool-SPS : examining underlying mechanisms, sintering oxides

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Low temperature sintering methods are developing fast, opening wide ranging opportunities to develop sustainable materials and processes, new composites or even materials... Pionnering works from Heli Jantunen group established the opportunity to rely on dissolutio-precipitation mechanisms to promote room-temperature sintering. Further extension of this concept to off-equilibrium conditions led to Cold Sintering, while thermodynamic conditions are mobilized in Hydrothermal sintering. Other mechanisms can contribute to the transport of matter required for sintering, yielding a great diversity of low temperature sintering approaches. Among these, Cool-SPS has demonstrated a great versatility, especially in terms of chemical composition of sinterd materials. This communication will discuss the sintering strategies and the possible matter transport mechanisms at play for Cool-SPS.

Thermal storage systems made of mPCM and C/C-SiC for e-mobility

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E-mobility is one of the fastest growing markets forecast for the coming decades, a trend only strengthened by the decision of the UN Climate Change Conference in 2021 to phase out internal combustion technology for vehicles. However, one factor that frequently limits battery-electric vehicles is the significantly shorter range compared with conventional combustion engines. Additionally, the range is drastically reduced during the cold season, when electrical energy is also required to heat the passenger cell, which becomes particularly important for public transport, e.g., by bus. Currently, different technologies are being investigated to provide thermal energy apart from the electrical generation.

One such technology is the latent heat storage. Alloys - also known as metallic Phase Change Material(s) (mPCM) - are used as energy carriers. They are melted in a stationary state and return large amounts of energy through phase change during operation. At the German Aerospace Center (DLR), current technologies are being investigated within the Next Generation Car (NGC) project. A particular focus lies on mPCMs with a high melting point for high energy efficiency (500-700°C). For this, special encapsulation materials are required that are inert to the reaction with the alloy. A promising candidate currently researched is fiber-reinforced ceramic C/C-SiC as a HT-resistant material. The experiments focus on the interaction between C/C-SiC and mPCM, and how a possible containment concept might be envisioned. A demonstrator is currently constructed to study achievable power densities and system stability. The results will contribution to the vehicles of tomorrow, and thus to ready our mobility for the future.

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Production of ZnO nanoparticles by pulsation reactor 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. Fine Particles of ZnO has unique electrical, mechanical, optical and piezoelectric properties for ceramic applications. Due to its ability to absorb UVA-UVB radiations, it is also interesting for sun protection in cosmetics industry as an alternative to widly used TiO_2. In spite of the recent rapid developments, controlling specifications of ZnO depending on the produced particle size has remained a major challenge.
The precise control of the thermal treatment steps during particle synthesis is a beneficial method of tailoring the material characteristics to the required application. Fine ZnO particles with specific properties have been thermally synthesized by pulsation reactor (PR). The (PR) consists of a hot gas generator that produces a pulsating stream. That means: fast and unsteady-state periodic combustion of natural gas in a chamber, with a resonance tube and a separator. This work describes specifications of applied PR and properties of nano ZnO particles produced by PR. During synthesis of ZnO in PR, the effect of characteristic parameters, such as treatment temperature and reactor geometry on properties of particles were studied using different analytical methods. The temperature of PR allowed adjustment of specific surface area (SSA) of resulting ZnO up to 60 m^2/ g with a primary particles size of 10 nm. Furthermore, the produced ZnO were dispersed in different medium using milling process. The influence of the PR particle properties on the dispersing behavior of the ZnO particles was investigated. Also, the UV-Vis measurements for the ZnO powder as well as dispersion were done and the results were discussed.

Development, production and tribological characterization of ceramic coated bearings for media lubricated pumps

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Media-lubricated bearings and mechanical seals are well established within the field of mechanical engineering chemistry and process engineering. Usually, cermet coatings containing tungsten, nickel and cobalt are applied as protective tribo-coatings. However, most of these raw materials have limited supply (W, Co) or are classified as hazardous (Ni). Additionally, certain media cause tribo-corrosive wear resulting in unreliable performance. The new concept of mixed-ceramic coatings presents a high-performance alternative for the commonly used cermet coatings. Three mixed-ceramic coating systems based on TiC / TiO_2, Cr_3C_2 / Cr_2O_3 and Al_2O_3 / ZrO_2 were applied by means of high-velocity suspension flame spraying onto plain steel bearings. One of the main objectives was the production of dimensionally accurate coatings with high surface quality, to significantly reduce post-processing efforts in terms of expensive grinding operations. Presented results comprise mechanical and structural coating properties, data from surface machining as well as comparative tribometry data generated from a media-lubricated test rig operated with a silicon carbide slide bearing counterpart. Final testing was then performed using coated bearing components that were operated in a media-lubricated pump under realistic conditions. The newly developed mixed-ceramic coatings show promising results and may be suitable to replace conventional cermet coatings in near future.

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Ceramic sensors for materials characterization

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Thick-film technique on ceramic substrates as well as its further development - multilayer substrates made in HTCC or LTCC are well-known and have been established for many years. Reliability, excellent thermal and electrical properties, and good long-term stability are key features. While the ceramic sensors are often used in the automotive area, e.g., for application in the powertrain, this contribution focusses on the development of novel ceramic sensors for high temperature materials characterization. Three examples of such applications will be presented and discussed: a low-cost miniaturized all-ceramic DSC (differential scanning calorimetry) device fully made in LTCC technology, a novel ceramic sample holder to determine simultaneously temperature-dependent electrical transport parameters like the electrical conductivity and the Hall-coefficient, the therefrom derived charge carrier density and Hall-mobility, as well as the Seebeck-coefficient. A new sensor stack for a Tian-Calvet Calorimeter made in LTCC technology is also shown. Application data are shown, and advantages and disadvantages of the designs are discussed and supported by measurements. All these examples prove the potential of ceramic technologies in the field of materials characterization, especially in the high temperature range.

Prediction of crack propagation in re-entrant honeycomb ceramics by polarimetry and digital image correlation

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The fracture behavior and crack propagation of ceramic components are characterized by their brittle performance and are difficult to predict for complex and cellular structures in particular. In addition, the production of ceramic prototypes is time-consuming and many samples are required for reliable statements about the properties. For this reason, it is useful to adopt alternative but established characterization methods from other fields like glasses or brittle polymers, like polarimetry.

To predict the crack propagation of ceramic honeycombs, brittle polymer lattices were 3-D printed via stereolithography The angle of the unit cell was varied from -35° to 35°, with negative angles forming an auxetic and positive a hexagonal lattice. These were photoelastically characterized under mechanical loading, revealing regions of excess stress. For comparison, alumina unit cells were fabricated via a combination of ceramic transfer molding and 3D printing. Characterization using compression testing and Digital Image Correlation (DIC) showed a good agreement of the crack propagation. For angles of smaller magnitude, the stress distribution is much more homogeneous, which is reflected in extremely increased compressive strengths. By identifying the stress peaks with polarimetry, it was also possible to predict crack propagation, which was moreover confirmed by digital image correlation.

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Novel single phase $(Ti_{0.2}W_{0.2}Ta_{0.2}Mo_{0.2}V_{0.2})C_{0.8}$ high entropy carbide using reactive spark plasma sintering

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Single phase $(Ti_{0.2}W_{0.2}Ta_{0.2}Mo_{0.2}V_{0.2})C_{0.8}$ high entropy carbide (HEC) compacts were successfully synthesized by reactive spark plasma sintering of ball milled metal-carbon black elemental mixtures at 1400 °C , 1600 °C and 1800 °C. X-ray diffraction and elemental distribution maps indicated single phase carbide formation with small amount of TiO_2. X-ray energy dispersive spectroscopy (EDS) maps on the compacts showed uniform distribution of the transition metals in the carbide phase. The microhardness, elastic modulus, fracture toughness, thermal expansion coefficient and electrical resistivity (25 °C–600 °C) of the compact sintered at 1800 °C were found to be 25.8 ± 2.8 GPa, 461 ± 36 GPa, 3.7 ± 0.4 MPa. $m^{1/2}$, 7×10^{-6} $K^{?1}$ and $7 \times 10^{?4}$ $?/m^2$ respectively.

The influence of graphene platelets on mechanical properties of titanium diboride ceramic composites

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TiB_2-SiC-graphene nanoplatelets (GNPs) and composites were prepared using field-assisted sintering technology at 2100°C in argon atmosphere, and the influence of the SiC and different GNPs addition on microstructure development and mechanical properties has been investigated. The increasing GNPs addition to TiB_2-20 % SiC from 1 to 10 wt. % with two types of platelets (nano-size/micro-size) resulted in significantly improved densification, with almost 100 % TD, but in decreased hardness from 24 to 14 GPa, and decreased elastic modulus from 370 to 236 GPa. According to the results for optimal strength and fracture toughness, the amount of GNPs additive should be 2 wt. % for both type of additives. The highest strength was measured for the system TiB_2-20SiC-2GNP with the value of 729 MPa and the highest fracture toughness for the system TiB_2-20SiC-10GNP with values 6.2 MPa.m$^{1/2}$. Fractography revealed no fracture origin in the form of processing flaws such as pores or clusters after bending strength test and toughening mechanisms on fracture lines and surface mainly in the form of crack bridging and crack branching by GNPs were observed.

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Field assisted sintering of refractory composite materials Nb-/Ta-Al_2O_3 and the influence on material properties

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For many different applications the demand for materials with tailored properties specifically designed for certain environmental and working conditions is gaining more and more attention. Especially in high?temperature applications these materials are very important, since components have to withstand very harsh working conditions. Therefore, many materials and alloys have been investigated to a large extent over the years. To improve performance as well as efficiency the approach of newly designed coarse?grained refractory composite materials made from pre-synthesized coarse grains is taken. The main goal is the development of non-shrinking materials with improved creep and thermal shock behaviour for applications in e.g. energy sector and metal working industries. This study deals with a two-step manufacturing and characterization of coarse-grained Nb- and Ta?Al_2O_3 refractory composite materials from pre-synthesized granules. As a first step, dense samples are prepared from fine?grained starting powders using Field Assisted Sintering and investigated to study the materials mechanical and electrical properties linked to the material composition and microstructure.

Key words: Nb, Ta, Al_2O_3, refractory composite, coarse-grained

Commercialization of ceramic oxygen separation membranes

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Since 12 years, Fraunhofer IKTS is developing oxygen membrane plants for on-site production of pure, cheap oxygen – O_2. The separation principle is based on a patented vacuum process using MIEC membranes (Mixed Ionic Electronic Conductor). Last devices are based on thin BSCF ($Ba_{0.5}Sr_{0.5}Co_{0.8}Fe_{0.2}O_{3?d}$) capillaries with small wall thickness enabling a high packaging density and a high O_2 flux per volume.

The actual plant concept is shown and explained in detail. Compared to the commercial technologies for oxygen production like cryogenic air separation, PSA and VPSA (Pressure Swing Adsorption, Vacuum PSA), a much lower electricity demand of 0.2 $kWh_{el.}/m^3$ O_2 was proven. Although a comparable amount of heat is also needed, the total costs are typically much lower compared to the mature technologies.

However, high investment costs affected the competitiveness of the new plants. Therefore, membrane costs were decreased by stepwise improvement of the manufacturing process during last few years. The whole plant concept was improved significantly to decrease costs further. Today, the membrane plants are already competitive in distinct applications with low but continuous oxygen demand. Presently, employees of Fraunhofer IKTS are working on the establishment of a spin-off company. It is aimed to a commercialization of MIEC membrane plants under the brand name poXos - pure oxygen on site.

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Microstructure and thermoelectric properties of electrospun Ca_3Co_4 xO_{9+}? ceramics from nanofiber mats

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Calcium cobaltite $Ca_3Co_{4-}xO_{9+}$? is known to be a promising thermoelectric material with good functional properties and thermal stability. To further enhance the functional performance of this ceramic for energy harvesting, it is desired to increase the power factor and simultaneously reduce the entropy conductivity. This can be realized by nanostructuring and texturization of the oxide-based ceramic using electrospinning. In this context, polycrystalline $Ca_3Co_{4-}xO_{9+}$? nanofibers with hollow and porous structure have been produced via sol-gel based electrospinning and calcination at medium temperatures to retain small particle sizes. The influence of the thermal treatment on the microstructure and thermoelectric properties of these electrospun nanofibers was investigated. Microstructural characterization of the nanofibers by transmission electron microscopy revealed an anisotropic particle growth along the increasement of the calcination temperature from nanoparticles to platelets, which are characteristic for $Ca_3Co_{4-}xO_{9+}$? with its layered crystal structure. These results are consistent with the evident formation of calcium cobaltite at 873 K from the X-ray diffraction pattern. Thermoelectric properties of the calcined and sintered ceramic nanofibers perpendicular to the pressing direction were determined. The Seebeck coefficient and the electrical conductivity show a strong dependence on the closed porosity and relative density.

The influence of gallium-containing mesoporous glass nanoparticles on pre-osteoblasts differentiation

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Mesoporous silica nanoparticles in the system SiO_2-Ga_2O_3 and SiO_2-CaO-Ga_2O_3 have been synthesized by the microemulsion assisted sol-gel method. The physicochemical properties of nanoparticles (NPs) were characterized by SEM, XRD, FTIR and N_2adsorption-desorption method. Electron microscopy examination confirmed that the presence of calcium influenced the morphology and textural properties of NPs. All samples exhibited spheroidal morphology and disordered mesoporous structure. The average diameter of the NPs was in the range of 80-150 nm. Ion release behavior of NPs was studied in Tris buffer (pH 7.4), Acetate buffer (pH 4.5) and cell culture medium (DMEM) for up to 7 days of incubation. Additionally, protein adsorption capability of the NPs was studied. The Ga-containing NPs were non-cytotoxic towards pre-osteoblast MC3T3-E1 cells in direct contact with nanoparticles in the concentrations of up to 100 mg/ml. To investigate cell viability further, the cells were stained with calcein-AM and propidium iodide which selectively stain live or dead cells, and the effect of the NPs on osteoblastic differentiation of pre-osteoblast MC3T3-E1 cells was studied for up to 14 days. Ga addition to NPs enhanced the alkaline phosphatase (ALP) activity and mineralization of MC3T3-E1 cells. These features make the obtained gallium containing NPs potential candidate for multifunctional applications, such as drug delivery carriers or bioactive fillers for bone tissue engineering applications.

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Material development for SOC: Quo vadis?

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Solid Oxide Cells is an enabler technology for highly efficient and reversible energy conversion from heat and electricity to chemicals and vice versa. Considerable progress in the development of materials and its processing technology enabled in pilot installations of SOC driven devices and demonstrated their technical feasibility. Besides upscaling and cost reductions issues, which are now on the front line of SOC development, the challenges of lifetime extension and increase of power density / efficiency have not lost their actuality. The degradation rates in the range of 0,4 to 0,7 % per 1000 hours achieved with planar stacks in field tests are still too high for moving for affordable large, distributed power generation and MW-scale electrolyzer applications (required lifetime of ca. 10 years). Additionally, the current density for SOC electrolyzers in base load operation should be approximately doubled to -1 A/cm^2 keeping the smallest degradation rate to become more competitive with low temperature systems. The presentation will provide an overview on status of electrodes and sealing materials as well as protective coatings for planar stack technology and discuss the options for improvement of durability and performance of SOC stacks by material development.

Surface modified ceramic materials for water treatment

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Thomas Kutschin[1,3]. Franz Jahn[2,3], Michael Stelter[1,2,3], Patrick Braeutigam[1,2,3] [1]Fraunhofer IKTS, Fraunhofer Institute for Ceramic Technologies and Systems, Michael-Faraday-Straße 1, 07629 Hermsdorf, Germany [2]Institute of Technical Chemistry and Environmental Chemistry, Friedrich Schiller University, Philosophenweg 7a, 07743 Jena, Germany [3]Center for Energy and Environmental Chemistry (CEEC Jena), Philosophenweg 7a, 07743 Jena, Germany The chemical modification of material surfaces makes it possible to impart new properties to them. For materials based on metal oxides in particular, organosilicon compounds are classically used for this purpose, which can be attached to surface OH groups by condensation reactions. The best-known example of this is the generation of water-repellent surfaces. Although the use of silanes/silanols is widespread for this purpose, the use of these compounds has some drawbacks. For example, silanes/silanols are water-sensitive and tend to self-condense, which makes the modification process more difficult. An alternative to the above mentioned compounds are modifiers based on phosphoric/phosphonic acid esters, which allow simple and controllable application and also exhibit excellent stability. In this lecture, possibilities for the application of modifiers to ceramic surfaces and their subsequent characterization will be presented. Furthermore, examples for the potential use of surface-modified adsorbent materials and ceramic membranes for water treatment will be shown.

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The influence of the C-fiber orientation on friction and wear mechanisms of C/SiC brake pads

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C/C-SiC is a high-performance friction material, due to the low density (2 g/cm^3), the high thermal shock resistance, the low wear rates and the high coefficients of friction (COF). Nevertheless, there are some drawbacks that prevent the broader application of these materials, e.g. high costs and the lack of fundamental tribological studies.

Therefore, in this work, C-short fiber reinforced C/C-SiC pads (30x30x10 mm^3) with five different fiber orientations were fabricated, applying 3k-HT-fibers (length: 12 mm; fiber volume content 50 %). The C-fibers are oriented; (a) randomly; (b) 0°; (c) 45°; (d) 90° towards the sliding direction and (e) vertically towards the frictional surface. The counterpart was a steel S235 disc (? 380 mm) on a dynamometer with a fly wheel of 800 kg (96 kgm^2) a starting speed of 10 m/s and a braking pressure of 3 MPa. The COF and the wear rate were measured and the frictional surfaces studied.
COFs between 0.45 and 0.60 were determined. The COFs of the 45° and the randomly orientated C/C-SiC pads are the highest. The wear rate is very low in general, between 29 (vertically orientated fibers) and 39 mm^3/MJ (0° orientated fibers).

Metal from the disc was transferred to the pads in different extents and fibers spalled partially away in dependence of the fiber orientation on the sliding direction. The evaluation of these mechanisms is the key towards a more fundamental studies and a more efficient friction material selection, compared to the past.

Silicon carbide ceramics with high thermal, electrical conductivity and other exceptional properties

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Freeze-granulated and afterwards under infrared lamp annealed silicon carbide powder was densified to the full density without any sintering aids by hot-pressing and/or ultra-rapid hot-pressing at 1850 °C. This densification temperature is at least 150-200 °C lower compared to the up to now known solid state sintered SiC powders. Samples densified by ultra-rapid hot-pressing reached full density. Partial beta ® alpha-SiC phase transformation was observed in the granulated and hot-pressed/rapid hot-pressed samples. The thermal and electrical conductivity of dense additive-free SiC ceramics were 164.4 W/mK and 8.3x10^{3} S/m, respectively. Bothe values are the highest pubished for SiC sintered at temperatures below 1900 °C. The creep rate of rapid hot-pressed samples at 1450 °C and 100 MPa load in 4-point bending test is 3.8x10^{-9} s^{-1} and at 1400 °C and the same load conditions is 9.9x10^{-10} s^{-1}. Creep rate of the same material at 1750 °C and higher load of 400 MPa in compression mode was only 10^{-7} s^{-1}. This is the lowest creep rate of SiC at such conditions found in the literature. Enhanced beta ® alpha-SiC phase transformation was observed after the creep test. The oxidation behaviour of this way prepared SiC ceramics at 1350-1450°C for 204 h was also investigated. The additive-free SiC ceramics exhibited a high oxidation resistance (4.91x10^{-5} mg^{2}/cm^{4}h) at 1450°C.

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Powder aerosol deposition of lead halide perovskites for optoelectronic applications

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Lead halide perovskites have gained much attention in recent years due to their remarkable optoelectronic properties. This resulted in highly efficient optoelectronic devices such as solar cells, X-ray detectors, or LEDs. Typically, lead halide perovskites are processed from solution, where after coating of a precursor solution, the perovskite forms in the course of solvent evaporation, simultaneously forming the final film. Thus, perovskite synthesis and film formation are inevitably coupled.

Here, we explore an alternative processing approach using typical ceramics technology, where in contrast to solution-based processing the perovskite synthesis and its film formation are decoupled. In a first step, we mechanochemically synthesize perovskite powder with good optoelectronic properties and high stability, which we use in a second step to produce thin films at room temperature using the dry powder aerosol deposition (PAD) method. We demonstrate the successful processing of the perovskite powder into thin and dense layers with low surface roughness as it is necessary for the fabrication of efficient solar cells. We characterize the electrical, structural and optical properties of the PAD-processed films and evaluate the effect of a post-processing thermal treatment on the PAD film properties.

A novel evaluation method for prepreg processability of oxide fiber composites (OFC)

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The processing of pre-impregnated fabrics (prepregs) emerged as a favorable route for the fabrication of ceramic matrix composites (CMC). Different measuring methods were developed to describe the property "prepreg tack" for the processing of prepregs for polymer matrix composites (PMC). Based on these studies, prepregs for the fabrication of oxide fiber composites (OFC) by a cold roll lamination process were investigated, which comprise a ceramic slurry instead of a polymer matrix. This is particularly relevant because for those types of prepregs limited information regarding their processability is available. Currently, the information gained from typical investigations of slurries, such as rheology, is insufficient for the processing of OFC prepregs. Thus, a novel testing method was designed to measure the "slurry tack" to estimate the rebound of prepreg stacks after compression. Tests were carried out with prepreg systems comprising Nextel 610 fabrics (DF-19) and an alumina-zirconia slurry with different processing parameters. The obtained results were in excellent agreement with the actual fabrication process, as it was possible to estimate the resulting fiber volume content of composite plates (110x110 mm^2) based on "mini-laminates" (35x35 mm^2). The described method offers therefore a tool for the assessment of prepreg processability, enabling quality control as well as the evaluation of new prepreg systems.

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FE simulation of co-sintering process of metal-ceramic laminates

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The potential combinations of favorable properties give metal-ceramic laminates (MCLs) a high degree of application flexibility. However, the different thermal expansion coefficient and shrinkage rate of metal and ceramic during the co-sintering process often leads to undesired deformations or even production failure. In practice, the identification of manufacturable MCLs still highly relies on the "trial and error" principle, which usually requires long development period. Therefore, there is a great demand for simulation methods that allow the prediction of deformation and manufacturability of MCLs during the co-sintering process.
The main objective of this study is to predict the deformation and the residual stress distribution in MCLs (X5CrNiCuNb17-4-4/3Y-TZP) based on the finite element (FE) simulation. To achieve this, the uniaxial viscosity, grain size evolution, and sintering stress of both materials were determined experimentally for the constitutive equation of the sinter model. In addition, the inhomogeneous density distribution of metal layer after the tape casting process which leads to the anisotropic sintering shrinkage was considered in the modelling and simulation. The results show that the developed FE simulation is capable of predicting the deformation of two-layer laminate. Furthermore, by considering the transformation-induced plasticity of the metal layer, the simulation was proved to be able to predict the residual stress distribution of multi-layer laminate.

Transfer layer evolution and influence of environment on friction in hipims W-C:H coatings

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The coefficients of friction (COF) of nanocomposite W-doped hydrogenated carbon (W-C:H) coatings are controlled by the amount of carbon in the matrix, level of its hydrogenation and environment. In humid air, oxidation of WC, Fe, water vapor dissociation and carbon hydrogenation due to flash temperatures were identified and confirmed by modelling based on the minimization of Gibbs free energy. These phases form transfer layer at the sliding interface preferentially adhered to a steel ball. Low steady COF values were achieved after quasi-static transfer layer with sufficient was formed. In dry or inert atmospheres, some of these reactions would not be possible. Subsequently, the composition of transfer layer would cause different friction behavior. The aim of the current workwas to summarize our earlier microscopic and Raman spectroscopy observations on the formation of transfer layers in W-C(:H) coatings prepared by HiPIMS during friction in humid (RH=30±3 %) air as well as in dry nitrogen, dry hydrogen and low vacuum and to support these studies by modelling.

The results showed, that in contrast to humid air, oxidation was in nitrogen atmosphere replaced by decomposition of WC producing carbon. In hydrogen, only the formation of methane from carbon and hydrogen atmosphere was predicted. Thus, assumed carbon hydrogenation would be the dominant reaction. In low vacuum (< 100 Pa), the most important was the decomposition of WC and $FeOWO_3$ into metallic W and Fe. Thus, the variations in the dominant reactions in different atmospheres and subsequently, the amount of lubricating hydrogenated carbon phase in transfer layer could explain the effects of environment on friction behavior in the studied W-C:H coatings.

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Development of Babin plots for geometry optimization of transverse TEG

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Thermoelectric generators (TEG) convert waste heat into electrical energy and can be used to power low-power applications, e.g. autonomous sensors and transmitters in sensor networks (Industry 4.0). Commercially available TEGs are based on doped Bi_2Te_3 or PbTe and are mainly fabricated in manufacture. In contrast, transverse multilayer thermoelectric generators (TMLTEG) consist of a low-cost, non-toxic and environmentally friendly oxide ceramic and a small amount of a metal. By using the ceramic multilayer technology, TMLTEGs can be manufactured as monolithic devices in large quantities.

Due to the unique properties of each oxide and metal material, the geometry of a TMLTEG must be adapted for each material combination so that the device achieves the optimum thermoelectric performance. Based on analytical calculations, we have developed so-called Babin plots that describe the power output and thermoelectric energy conversion efficiency as a function of the internal and external device geometry. The talk shows the application of these plots in the fabrication of devices made of different established thermoelectric oxide materials such as $Ca_3Co_4O_9$, La_2CuO_4 and $CaMnO_3$. Furthermore, we show the results of a material screening on suitable material combinations for a TMLTEG based on the Babin plots.

Stainless steel/ceramic composites for applications with molten aluminum alloys

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The development and structural characterization of novel steel/ceramic refractory composites for contact with molten aluminum alloy have been presented. The composites were manufactured via powder metallurgy and consisted of 60 vol% 316L (1.4404 – FeCr18Ni10Mo3) stainless steel powder and 40 vol% of MgO powder addition. The corrosion resistance of developed composite was investigated by means of wettability test, long-term crucible corrosion test and finger immersion test using molten AlSi7Mg0.3 alloy at 850 °C. The wettability tests were carried out using the capillary purification technique in a hot-stage microscope. Crucible corrosion test were performed for 24 h and 168 h. Finger immersion tests were performed for 24 h. Selected composite samples undergone the surface heat treatment at 850 °C and 1000 °C for 24 h, which significantly increased their corrosion resistance against molten aluminum alloy. The composites after corrosion tests were analyzed using Laser Scanning Microscope, SEM/EDS and XRD focusing on their corrosion resistance and the phase formation at the contact interface with aluminum alloy. The impact of selected steel/ceramic refractory composites on the composition of aluminum alloy was investigated by means of adapted ASPEX PSEM/AFA and SEM/EDS/EBSD. It was revealed that developed steel/ceramic composites exhibit excellent long-term corrosion resistance against molten aluminum alloys.

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Efficient photon-harvesting technologies for water splitting reactions

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Metal oxide nanostructures with hetero-contacts and phase boundaries offer unique platform for designing materials architectures for energy harvesting applications. As viable alternative to water electrolysis, photoelectrochemical (PEC) water splitting has emerged as a competitive technology being capable of converting solar energy directly into chemical energy using stable and efficient photocatalysts for solar hydrogen production. Besides the size and surface effects, the modulation of electronic behaviour due to junction properties leads to modified surface states that promote selective decomposition of analytes and adsorbates. The growing possibilities of engineering nanostructures in various compositions (pure, doped, composites, heterostructures) and forms has intensified the research on the integration of different functional material units in a single architecture to obtain new photocatalytic materials. Even though the potential of hematite thin films for water splitting applications are widely accepted, researchers are still tackling the 'rust challenge'. We report here on the influence of external magnetic fields applied parallel or perpendicular to the substrate during plasma enhanced chemical vapor deposition of hematite (?-Fe_2O_3) nanostructures. Hematite films grown from iron precursors showed pronounced changes in crystallographic textures depending upon whether CVD was performed with or without external magnetic field. Investigations on the water splitting properties of the hematite films in a photoelectrochemical reactor revealed superior photocurrent values of hematite photoanodes deposited in external magnetic field.

Recycling possibilities for solid oxide cells

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In June 2021 the German Federal Constitutional Court made the obligatory decision to reach national greenhouse gas neutrality by the year 2045 which was accepted in the amended version of the Climate Protection Law. In order to achieve these ambitious climate protection goals, a fundamental restructuring of the complete energy sector must occur, whereby the use of hydrogen technologies for energy supply and storage will be essential in the future. Similar to current lithium-ion batteries, hydrogen electrolyser and fuel cells contain also critical and valuable raw materials that must be preserved after the end of use (EoU). However, in contrast to established recycling strategies for batteries, recycling routes for hydrogen devices are still fundamentally unknown. Within the framework of the technology platform H2Giga (BMBF), the project "ReNaRe" aims for the development of recycling concepts for different hydrogen electrolysers. Using the example of anode- and electrolyte-supported solid oxide cells, the first approaches of possible utilization routes are currently being investigated experimentally. The solid oxide cells are constructed from a nickel-yttrium stabilized zirconia ceramic (Ni-YSZ) on the fuel gas side and from perovskite-based materials on the air side, with the Ni-YSZ weight fraction predominating. The recycling concept envisages separation of the base materials used and the reuse of Ni-YSZ as a substrate for manufacturing of new solid oxide cells, while possible recycling routes for the separated perovskite-based materials are still under development. In parallel, recycling routes for the metallic components are being investigated elsewhere. This presentation shows the first contents of the recycling strategies and possibilities.

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Influence of surface treatment on the strength of a dental zirconia for implants

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Roughening of zirconia dental implants is a common clinical practice to improve ingrowth behavior. It depends on the manufacturer of the implant at which stage of the manufacturing process and by which method the surface is roughened. Systematic studies on this topic are rarely found in the literature. Therefore, the influence of surface treatment on the strength of a dental zirconia was investigated as part of a research project on the development of ceramic implants.

The material under test was a commercial zirconia consisting of a Y-TZP matrix and Ce-TZP inclusions in the sintered state. This material is characterized by a slightly higher fracture toughness and slightly reduced strength compared to typical 3Y-TZP. Sets of samples were sandblasted in the white-fired or sintered condition. The ball-on-three-ball-strength of these samples was measured and compared to the strength of as-fired samples and polished samples. The complete study was performed two times for validation of the results.

It is found that the average strength of TZP ceramics differs by almost 500 MPa depending on the surface treatment. Conventionally sintered specimens with as-fired surface exhibit a strength of 880 MPa. Sandblasting in the white fired state reduces the strength to 690 MPa. Both polishing and sandblasting in the sintered condition result in an increase in strength to about 1180 MPa. Comparative microstructural investigations, roughness measurements and X-ray phase analyses were carried out to determine the causes of these huge differences in strength. These findings may challenge the practice of white body surface treatment and give reason for further investigations on other commercial dental TZP materials.

Superparamagnetic Fe@SiCN ceramic fibers for functional applications

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Non-oxide ceramic fibers are sophisticated, key structural components with superior mechanical and thermal properties. Most of the applications are safety-relevant components, whose damage is difficult to detect. Furthermore, it would benefit to include functional properties in addition to structural aspects. Hence, we developed novel functional superparamagnetic, iron-containing SiCN ceramic fibers derived from an iron-modified, meltable polysilazane via melt-spinning, curing and pyrolysis at 1000 °C. Tensile strength values of 1.24 GPa were measured for fibers with diameters up to 40 µm. Despite a slightly reduced oxidation stability, these ceramic fibers are suitable for structural applications. Transmission electron microscopy confirms the formation of nano-crystalline FeSi, Fe_3Si, and Fe particles with a size up to 7.5 nm. Due to the well distributed iron-containing nanoparticles, the ceramic fibers exhibit superparamagnetic behavior for temperatures higher than 26 K with a saturation magnetization of 1.54 emu?g^{-1} at 300 K. These fibers are very promising for electromagnetic shielding, defect detection, and separation from polymer matrices.

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Tape casting and optimization of the slurry composition of a SiCer-compatible LTCC tape

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Silicon and LTCC tapes (Low Temperature Cofired Ceramics) are standard substrate materials in micro sensors and microsystems technology. Established methods of micro-technology, such as photolithography and micro-structuring, thick-film and thin-film technologies, as well as methods of assembly and connection technologies can be applied to both materials, resulting in efficient microsystems fabrication options for various product areas. Through combination of both materials into a common SICER substrate system, micro systems with significantly higher complexity might be developed.The presentation will deal with the development of the slurry and the casting technique of the LTCC tape for the SICER process. There is an in depth look at the goals and benefits of the technology and the processing steps. The slurry optimization is presented. The dispersant is under investigation by preparing slurries and looking at the particle distribution with a FESEM. Different plasticizers are tested by stamping, machining of green tapes and preparation of SiCer substrates. By preparing green tapes and measuring the slurry viscosity, density, shrinkage and tensile strength of the green tape the binder for further tape preparation is decided. By laborious experiments the slurry preparation has been investigated. The milling devices and milling balls and the different times for dispersion and homogenization have been tested and characterized by rheological measurements. An upscale process will be shown going from discontinuous to a continuous casting process. Finally, some examples of the SICER process built with the tape are shown.

Synthesis and characterization of SiO_2-CaO based mesoporous bioactive glass nanocarriers for delivery of therapeutic biomolecules

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In this study, SiO_2-CaO based multifunctional mesoporous bioactive glass nanoparticles (MBGNs) were prepared using a microemulsion-assisted sol-gel process. Scanning electron microscopy (SEM) and transmission electron microscopy (TEM) analysis confirmed that the particles had spherical morphology (mean particle size: 120 nm) with disordered mesoporous structure. Brunauer Emmett Teller (BET) results revealed that MBGNs exhibited high surface area (309-444 m2/g) and mesoporosity (characterized by TEM), which is a valuable feature for the loading of therapeutic amounts of biomolecules for drug delivery applications. In this context, therapeutic biomolecules (icariin and silibinin) were loaded in synthesized MBGNs. The in vitro mineralization results revealed the formation of a typical hydroxyapatite structure in physicological conditions, which suggests the bone-bonding ability of the synthesized MBGNs. The drug release study showed a sustained release of the loaded drug under dynamic conditions, which is significantly crucial for sustained therapeutic drug release. Cell viability assays revealed that icariin exhibits a dose-dependent cytocompatibility against the MG-63 osteosarcoma cell line. Silibinin-loaded MBGNs were tested using MG-63 (osteosarcoma cells), MDA-MB-231, and MCF-10A cell lines. Our initial results suggest that silibinin exhibits chemotherapeutic potential towards metastasized cancer cells. Altogether, the unique texture properties of synthesized MBGNs, their capability to incorporate metallic ions in their structure, and the loading of different biomolecules suggested that these multifunctional MBGNs are very attractive carriers for drug delivery for biomedical applications.

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Accelerated microstructure design using the high performance simulation framework Pace3D for phase-field models

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Selected highlights of microstructure simulations based on the phase-field method combined with multiphysical modelling are presented aiming for an accelerated development of advanced materials. The simulation techniques are capable to predict microstructure and material property correlations for material systems with multiple components and phases under the consideration of different process conditions including mass and heat transfer, continuum and fluid mechanics, magnetism and electrochemistry. The grain size distribution is a key criterion for the hardness and fracture toughness. Simulations allow the in-situ insight in the 3D structure formation process. Large-scale simulations of final stage sintering for realistic polycrystalline microstructures are shown, where a complex interplay of densification and grain growth dominates microstructural evolution. Grain growth starts, when pore drag effects become less important due to pore shrinkage. This grain growth then decreases the driving force available for sintering. The multiphysics simulation package Pace3D comprises modern multigrid and parallel algorithms to efficiently use high performance clusters with excellent scalability properties and extensive data analysis algorithms for the virtual and accelerated design of new materials. Finally, a new research data infrastructure: „Karlsruhe Data Infrastructure for Materials Science (Kadi4Mat)“ is introduced providing data management and processing for public usage.

Ceramic R&D in Japan –future prospects–

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Materials technologies and related industries are one of the most competitive areas of Japan. In order to maintain this competitiveness, it is critically important to assess the essential technical elements required for future development and societal sustainability and deploy the strategic research programs accordingly. This talk will give an overview of primary research items for "fine ceramics" (advanced ceramics) we will focus on during the coming several years under "Materials Strategy" formulated by the Japanese government. They include: Visualizing and monitoring the whole manufacturing processes of ceramics and their products and unraveling factors which control the variation of microstructural states; Developing computational tools which comprehensively deal with various intricate phenomena in the manufacturing processes and constructing "process informatics platform"; Exploring innovative manufacturing processes which realize high-quality products (e.g. high reliability of a minute component), low-energy production (e.g. room-temperature sintering), etc.; Evaluating and elucidating the process-structure-property-performance relationships in further details. The talk will also introduce and discuss "FC Roadmap 2050" of the Japan Fine Ceramics Association (JFCA), which deals with future visions of technologies and products of "fine ceramics" in the year 2050.

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The future of power generation starts today - with highly efficient solid oxide fuel cell systems

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Bosch is opening a new chapter in the energy transition: In 2024, the start of a full-scale production of distributed power systems based on solid oxide fuel-cell (SOFC) technology is scheduled- an annual production capacity of some 200 megawatts is foreseen.

The heart of the system is a disruptive technology for metal-supported ceramic fuel cells; these cells have besides their excellent robustness, a lower operating temperature with a longer service life and can be produced on a large industrial scale at low cost.

Recent development activities and the status quo of SOFC technology at Bosch are shown:
Pilot plants based on solid oxide fuel cells are already being successfully tested at various Bosch locations and pioneer customers. The SOFC systems can already be operated with eco-friendly biogas or natural gas – and are already hydrogen-ready for the energy system of the future. – with zero emissions of nitrogen oxides, particulates, and CO_2.

Non-destructive monitoring of internal structure during the production of high dense 3D printed alumina parts

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Fused Filament Fabrication (FFF) is, amongst other additive manufacturing technologies, considered as the most accessible, variable, and cost-effective technique. It allows prototyping of hollow and highly inner-structured objects using different materials but is not conventional for fabricating dense parts. This work presents the preparation of highly dense alumina objects (cylindrical and prismatic design) using a low-cost FFF 3D printer. After the preparation, micro-computed tomography (µ-CT) was applied to monitor internal structure after each critical processing step (3D printing, debinding, sintering) to detect possible material inhomogeneities. Results achieved showed that a 3D shape design plays a significant role in defect-free printouts. However, despite internal cracks, pores and cavities revealed by the µ-CT analysis, sintered objects achieved relative densities of up to 96 %.

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Usage of sintering aids for coarse pottery clays at low firing temperatures: an introduction

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Firing coarse pottery clay goods, temperatures in the range of 800–1000 °C are well established. As a side effect, CO_2 is released through carbonate decomposition (650–850 °C) when processing calcareous raw materials. Strongly supporting the European Green Deal, Wienerberger is also striving to find solutions for a reduction of these raw material related process emissions. Therefore, novel solid-solid or liquid-solid reactions using additives should be identified, which allow lower firing and sintering temperatures towards the decomposition temperature of carbonates whilst keeping the physico-technical parameters of the fired products comparable to the available materials. Besides, the lower fuel demand also saves CO_2. For the selection of most suitable additives, an extensive literature research with a subsequent evaluation matrix was carried out. The evaluation criteria included field of application, applied temperature range, melting temperature, effectiveness, reported dosage used, toxicity, availability, and economic aspects of possible additives. Based on the results of the evaluation, target-oriented additives appear to be (glassy) materials high in alkali and boron content. First experiments have been carried out and based on the most promising results, an extended test program and thermodynamic modeling will be performed.

Solution processed $BiFeO_3$ thin-films for photovoltaic applications

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Although metal-organic halide perovskites exhibit outstanding light-harvesting properties, their toxicity and instability may prevent their broad deployment in photovoltaic applications. Semiconducting oxides with perovskite structure are considered an emerging material class with potentially similar properties but much higher sustainability and durability. Bismuth ferrite (BFO) is a ferroelectric model system that has been widely studied as a lead-free alternative for PZT and lately for its photovoltaic performance. If a route to solution processing of BFO thin-films could be found, this technology would directly integrate into the field of printed optoelectronics for lowest fabrication costs with high design flexibility.

As of today, the thin-film synthesis via gel combustion has been challenging since solvent evaporation and the formation of gasses during combustion cause high porosity.

To achieve dense thin films in a single process step, we investigate the drying and combustion of metal-citrate complexed gels, which were obtained by sol-gel synthesis via an aqueous nitrate route and spin-coating. Different drying procedures provided control of the morphology, and a detailed microstructure analysis provides handles to the optimization towards dense BFO thin films for photovoltaic applications.

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Customized ceramic granules for laser powder bed fusion of complex alumina components

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Laser powder bed fusion is an emerging technology in industry and research, especially for metal and polymer applications. It offers a great potential to produce complex prototypes and small series by expanded boundaries for design engineers. Ceramic parts could be manufactured with high accuracy and short lead times and potentially, cost-, energy- and time-consuming hard-machining and thermal post-processing could be avoided. However, its industrial implementation for oxide ceramics remains challenging due to low thermal shock resistance, weak densification and low light absorptance in the visible or near-infrared range. Several approaches to overcome these problems were tested; however, they were only partially successful in combining high accuracies, suitable mechanical properties and total freedom of design.

In this work, a solution to increase the powder absorptance of the laser and to reduce cracking during laser processing of aluminum oxide parts is given. Different colored additives able to form low and negative thermal expansion phases during the rapid cooling of the powder bed fusion process were tested. A co-development of customized powders as well as the optimization of the laser parameters enabled the in-situ formation of these phases and led to a strong crack reduction in the final dense aluminum oxide components. X-ray diffraction finally confirmed the formation of these ternary phases.

Impact of electric fields on the microstructural evolution of strontium titanate

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New synthesis and processing routes such as SPS and flash sintering were established using knowledge of sintering and grain growth behaviour under the influence of electric fields. The present study shows novel insights into the topic by investigating the impact of an electric field on the microstructural evolution of strontium titanate under external DC and AC electric fields. We analyzed the grain growth behaviour of dense ceramics with a special focus on defect chemistry and space charge layers.

By using alumina shielded electrodes (no joule heating) and a well-defined experimental setup, we obtained a solid dataset of the microstructural evolution depending on temperatures, AC frequencies and electric field strength. Previously we explained the impact of a DC field on grain growth by combining the knowledge on field-free microstructural evolution, defect chemistry and polarization, leading to the assumption that the local grain boundary mobility is controlled by the electromigration of oxygen vacancies.

In the present study we apply low frequency AC fields, vary intrinsic defect concentrations by changing the oxygen partial pressure and use different single crystal orientations to expand the model towards kinetic effects and other defect related mechanisms, such as the influence of bulk defect concentration or the oscillation of defects on different length scales.

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Microstructure and magnetic properties of Ni-Cu-Zn ferrites for multilayer inductors

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Electronic devices have witnessed an enormous development and performance improvements over the last few decades which is fundamentally based on advancing miniaturization and the associated increase in integration density. Part of this success story may be attributed to advances in multilayer technology for passive components, including magnetic components. Ni-Cu-Zn ferrites are used as magnetic materials for multilayer inductors and a solid basis of knowledge on chemical composition, processing, microstructure, and magnetic properties of these soft ferrites exists in the literature. The standard models to correlate microstructure and permeability is the nonmagnetic grain boundary model (NMGB), which is based on the concept of a thin nonmagnetic layer between ferrite grains.
We have performed a systematic study on the correlations between microstructure and permeability behavior of Ni-Cu-Zn ferrites.
An extensive study on the sintering behavior with variations of sintering temperature and dwell times was performed, and different microstructures were generated. Special attention was paid to the appearance of Cu-oxide based grain boundary phases. Samples were characterized in terms of density, phase formation, microstructure, element distribution and magnetic permeability. The permeability was modelled using the NMGB model. The results show the limited validity of the existing models. Expansions to this model considering grain size and density are proposed.

Ox/Ox CMC: a high temperature fiber reinforced composite with great potential

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Oxide ceramic fiber reinforced oxide composites (Ox/Ox CMC) have been developed for many years and are mainly used in the field of industrial furnace construction. Nevertheless, this material, combining oxide fibers with oxide matrices, is still largely unknown in many technical fields.

In this presentation, the material properties and in particular the application-specific adaptation of the properties will be presented. By orienting the fibers and varying the layer structure, both specifically anisotropic and nearly isotropic materials can be produced, that are applicable up to 1300°C. Therefore, Ox/Ox CMC materials could be used in many industrial fields where high temperature and corrosion loads are present. However, designers and engineers are often missing a detailed understanding of materials when designing such components. The potential of Ox/Ox CMC's will be illustrated by means of some concrete application examples. The mainly thin-walled fiber-reinforced lightweight structures with appropriate design solutions are used as load-bearing racks in heat treatment processes, where lightweight and inert materials are required to realize short process times. Complex thin-walled lightweight structures are also used in unmanned aerial vehicles as components in the propulsion sector.

In addition to the pure Ox/Ox CMC, hybrid solution approaches for the combination of monolithic ceramics with Ox/Ox CMC are also demonstrated. Here, the good properties of the monolithic and fiber-reinforced materials are combined, demonstrating new application possibilities. For example, a hybrid material newly developed on behalf of BASF is used for components in chemical apparatus engineering.

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Critical processing parameters for wound oxide CMC and their effect on material properties

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Fibre reinforced all oxide ceramic matrix composites (oxCMC) have made significant advancements over the past 20 years and have found their way into a growing number of industrial applications. However, the material properties and quality vary considerably and depend strongly on the manufacturing process and respective processing parameters. With regards to industrial production capability, filament winding is the most automated and state-of-the-art fabrication technology for oxCMC today, in particular for rotational symmetric designs and components. Nevertheless, there is considerable potential to improve material properties and quality by technological optimization of critical processing parameters to meet the growing demands and requirements of future applications. Several key processing parameters for filament winding of oxide CMC were identified and their effect on the overall processability and resulting material properties will be presented, demonstrating a significant improvement of material homogeneity and reproducibility as well as an increase in bending strength by around 30%.

Pyrocatalytic oxidation: structure-activity relationship of thermally excited $BaTiO_3$ nanoparticles for water remediation

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Sascha Raufeisen[1,2], Michael Stelter[1,2,3], Patrick Braeutigam[1,2,3] [1]Institute of Technical Chemistry and Environmental Chemistry, Friedrich Schiller University, Germany [2]Center for Energy and Environmental Chemistry (CEEC Jena), Germany [3]Fraunhofer IKTS, Hermsdorf, Germany In the light of stricter regulations concerning the occurrence of micropollutants in the water cycle, there is an ongoing search for new methods like advanced oxidation processes (AOP). Within this search, the pyroelectric effect of ferroelectric materials was investigated. Pyroelectric materials are thermally excited in an aqueous medium and subsequently generated charges on the surface of the pyroelectric crystals induce an oxidation of organic compounds. Experimental studies showed that several organic compounds (2',7'-dichlorodihydrofluorescin, coumarin, terephthalic acid, RhB) can be oxidized in the presence of different pyroelectric materials like $BaTiO_3$ and $LiNbO_3$. It was also possible to disinfect a solution of bacteria and to confirm via ESR spectroscopy, chemical dosimetries and radical scavenger experiments that reactive radicals (*OH, *O_2^-) are generated during thermal excitation of these materials. Beside these fundamental findings regarding the existence of a pyrocatalytic oxidation, only little is known about the influence of critical reaction and process parameters or the exact mechanism of the process. Moreover, the influence of important material parameters (particle size/shape/porosity/surface, pyroelectric constant, crystallographic phase) on the process is still unclear. Within this contribution, new aspects of pyrocatalytic oxidation will be presented to overcome the of the above-mentioned knowledge gap.

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Development of application specific ceramic temperature cyclers

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A large number of technical manufacturing processes are based on the specific temperature control of process goods or chemical reagents. Due to their technical properties, ceramic materials and technologies are suitable for implementing selected heater applications in the geometrically mesoscale range. One example is a highly dynamic and actively cooled matrix heater for the generation of fast temperature cycles. These consist of individual pixels that are combined to form a matrix. Supported by a simulation-based design of these components, well-known technologies such as thick film technology are symbiotically combined with modern techniques of additive manufacturing of ceramic materials (CerAM) at the Fraunhofer IKTS. The production of the basic structures is based on ceramic materials and technologies. Ceramic materials can only be machined at great expense due to their typical high hardness and low ductility. Here, CerAM processes represent "game changers", since complex geometries can be realized close to the final contour and functions, such as cooling channels, even in small volumes. The use of these methods results in increased geometric degrees of freedom and completely new areas of application. The optimized CerAM components are functionalized using the well-known thick-film technologies. Electrical circuits, heating elements and sensors are printed in a structured manner and fired onto the ceramic basic structure. Electrical contacting is achieved by means of advanced assembly and connection technology. Solutions up to temperatures of 350°C and temperature gradients of up to 70 K/s have been successfully realized. Applications up to 600°C are currently being tested.

Industrial heat treatment: An established and consistently new market for CMCs

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The industrial heat treatment sector has been using monolithic ceramics and ceramic matrix composites (CMCs) for decades. In particular, due to their low density and high temperature resistance, those materials offer great advantages in terms of energy efficiency, service life and process times in the field of heat treatment and have thus been able to establish themselves over other materials, e.g. furnace racks made of metallic alloys. Current fields of applications and materials made of oxide and non-oxide ceramic matrix composites will be presented, as well as hybrid solutions for the use in heat treatment industry. A special focus will be on the processing of steels. The processes used at Schunk and resulting materials made of CFC and OFC are described, as are the advantages of CMCs for these applications. But also application limits and future potentials are highlighted.

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Does flash sintering involve plastic flow?

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During flash sintering, very rapid densification of a ceramic powder compact occurs during a thermal runaway induced by electrical power dissipation from an applied voltage and current. The mechanisms of the fast densification within seconds are still not well understood. The present study investigates the impact of plastic flow on the densification of strontium titanate during flash sintering. After sintering, a high dislocation density of almost 10^{14} m^{-2} was observed by transmission electron microscopy. Uniaxial compression tests at 1150°C revealed that deformation rates are about one order of magnitude higher for flash sintered samples compared to conventionally sintered samples.

Based on these findings, it seems as dislocations are generated and migrate during sintering resulting in the occurrence of plastic flow. This becomes possible by the very high heating rates, which conserve high driving forces for sintering up to high temperatures by minimizing neck growth by surface diffusion during heating. Accordingly, high driving forces and sintering stresses of several 10 MPa (i.e. above the flow stress) are preserved up to high temperatures. In this light, the most important parameter of flash sintering is the extremely high heating rate. Beyond extending our understating of high heating rate sintering, the presented framework offers new perspectives for materials engineering by introducing dislocations for enhanced properties.

Implications of ferroelectricity in perovskite solar cells

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Methylammonium lead iodide ($MAPbI_3$) is the archetypal semiconductor in organic metal halide solar cells. A controversial discussion about whether or not $MAPbI_3$ is ferroelectric and if ferroelectricity can influence the solar cell performance, was fueled by a wide range of seemingly contradictory results. In recent years, researchers proposed many related effects and properties of this hybrid perovskite material, such as piezoelectricity, pyroelectricity, electrostriction and ionic conductivity. Notably, polar domains in $MAPbI_3$ thin-films have been observed,providing indirect evidence of ferroelectricity. Only recently, poling of domains in an external field, which is considered a hallmark of ferroelectric materials, was demonstrated in order to consolidate the discussion.

In this work, we use Piezoresponse Force Microscopy to monitor the evolution of ferroelectric domains that is triggered by common thermal treatment of perovskite solar cell at 100°C. Our findings suggest that control of the ferroelectric domain structure in these light absorbing, semiconducting compounds may be essential in order to maximize performance of hybrid perovskite solar cells.

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New perspectives on grain boundary migration in polycrystals

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Using high-energy diffraction microscopy, we have measured the velocities of grain boundaries within a Ni polycrystal evolving by normal grain growth under a capillary driving force. Thousands of grain boundaries were tracked and their velocities and curvatures were classified by their crystallographic characteristics. Among the findings, two are noteworthy and will be discussed in this seminar. First, the velocities vary with all five crystallographic grain boundary parameters. Because boundaries are curved, and a single misorientation has a range of boundary plane orientations, one might expect the migration of grain boundaries with a particular misorientation to be independent of grain boundary plane orientation. However, on average, some orientations are faster than others and for a single misorientation, velocities can vary by a factor of three or more. Second, grain boundary velocity is independent of grain boundary mean curvature. Because curvature is an important component of the driving force, velocity and curvature are expected to be correlated positively. However, the evidence for such a correlation is poor. In the remainder of the seminar, I will briefly present three possible explanations for these observations that can be discussed as a group: constrained migration of boundaries in connected networks, defect (disconnection) mediated grain boundary migration, and the influence of grain boundary stiffness on migration.

Influence of the cutting tool on the machining performance of C/C-SiC

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Machining processes are required to generate the final shape of CMC (Ceramic Matrix Composite) components and to remove imperfections on its surface. Due to the superior properties of CMCs such as high hardness, wear resistance and high temperature resistance, the machining process is as challenging as for monolithic ceramics.

Diamond grinding is still the most effective machining method for manufacturing C/C-SiC components like ceramic brake discs. Nevertheless new CMC applications like turbine vanes, shrouds and combustion parts have 3d shapes and designs. Therefore machining with milling tools and geometric defined cutting edges becomes an interesting option for more economic and reliable production. C/C-SiC show heterogeneous and brittle machining behavior. Especially the high hardness of the SiC matrix leads to high tool loading and abrasion. In this study milling tools with different cutting materials are compared and used to machine a 2d fabric reinforced C/C-SiC. During machining the applied load was measured by a piezoelectric sensor. The cutting performance of the tools is evaluated by the wear progress.

Especially milling tools with very hard cutting materials like polycrystalline diamond show sufficient wear resistance. This makes it possible to mill C/C-SiC and overcomes the limited feed rate of grinding process.

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Design and manufacture of solid state batteries towards low cost

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Next generation of energy storage devices may largely benefit from fast and solid Li^+ ceramic electrolyte conductors to allow for safe and efficient batteries and fast data calculation. For those applications, the ability of Li-oxides to be processed as thin film structures and with high control over Lithiation and phases at low temperature is of essence to control conductivity. Through this presentation we review the field from a new angle, not only focused on the classics such as Li-ionic transport and electrochemical stability window for Li-solid state battery electrolytes, but focusing on opportunities and challenges routes in thermal and ceramic processing of the components and their assemblies with electrodes.

High-strength oxide ceramic matrix composites produced by two-step sintering

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The high strength and toughness of oxide ceramic matrix composites (Ox-CMCs) depend on their refined microstructure and matrix porosity. Nevertheless, it is relatively difficult to consolidate the ceramic matrix without causing fiber degradation using conventional sintering (CS) methods. Hence, the use of two-step sintering (TSS) for the production of high-strength Ox-CMCs is here presented. Minicomposites containing Nextel 610 fibers in a porous alumina matrix were produced by TSS ($T_1 = 1200°C$ without holding time and $T_2 = 900°C$ for 10 h) and compared to CS minicomposites (1200°C for 2 h) regarding their tensile strength and microstructure. During CS, the densification of matrix causes the formation of cracks and the strength of the minicomposites decreases with longer exposure times. In contrast, most of the densification during TSS takes place on the first step, while the matrix is slowly strengthened during the second step without impairing the minicomposite strength. Consequently, higher tensile strength was achieved by TSS minicomposites (230±27 MPa) when compared to CS (133±8 MPa). In summary, TSS is a very promising and easy-to-apply sintering method for producing Ox-CMCs with controlled microstructure.

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Rapid hot-pressed ultra-high creep resistant silicon carbide ceramics

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Freeze-granulated silicon carbide powder was densified to the full density without any sintering aids by rapid hot-pressing at 1850 °C. This densification temperature is at least 150-200 °C lower compared to the up to now known solid state sintered silicon carbide powders. Presented silicon carbide hot-pressed ceramics have excellent mechanical properties. Samples densified by ultra-rapid hot-pressing have also full density and hardness of 27.4 GPa. Partial phase transformation beta/alpha - SiC was observed in the granulated and rapid hot-pressed samples.

These additive-free **s**ilicon carbide ceramics was crept in vacuum at temperatures of 1500 °C to 1750 °C and compressive loads of 200 MPa to 400 MPa. The results showed that this way prepared ceramics had the lowest creep rate reported in the literature. The observed strain rates increased from 2.5×10^{-9} s^{-1} at 1500 °C and a load of 275 MPa to 1.05×10^{-7} s^{-1} at 1750 °C and a highest load of 400 MPa. The average creep activation energy taken from all creep tests was 315 ± 20 kJ?mol^{-1}, and the stress exponent was 2.22 ± 0.17. The suggested creep mechanism is GB sliding accommodated by GB diffusion and b-a SiC phase transformation.

Contribution of refractory materials to lower the carbon footprint of high temperature processes

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The pressure to all industries reducing their carbon footprint increases daily. The world governments agreed to keep the global temperature rise below 2°C, if possible 1.5°C, by the end of this century to avoid the worst environmental impacts. All industries including the refractory industry have to make their contribution to reach this challenging goal. As the biggest consumer of refractories, the steel industry is currently facing big changes regarding steel making processes to reduce the greenhouse gas intensity drastically. The task of the refractory producers is to supply innovative products that come with an minimized carbon footprint as well as to enable the new environmental friendly processes of the future. This presentation shares different theoretical aspects as well as practical experience from different industries of CO_2-emission optimized refractory materials and refractory solutions.

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Fracture toughness of textured alumina grains and grain boundaries determined by micro-cantilever bending tests

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Embedding textured alumina layers has been established as a novel design strategy to enhance the fracture resistance in ceramic laminates. It is hypothesized that the exceptional damage tolerance in these materials is mainly attributed to crack deflection events along the weaker basal grain boundaries within the textured microstructure. To prove this hypothesis, the fracture toughness of individual textured alumina grains and grain boundaries has been measured through micro-cantilever bending tests. Micro-scale notched cantilevers were milled using Focus Ion beam along the prismatic direction of the aligned textured grains and perpendicular to the elongated grain boundaries in basal orientation and tested with a nanoindentor. A shape function for notched pentagonal-shaped cantilevers was calculated using Finite element analysis to retrieve the critical stress intensity factor at the notch tip. The micro-scale fracture toughness of the textured alumina grain boundaries was found to be approximately 40% lower than the one of the grains. This experimental finding is paramount for understanding the macroscopic fracture behaviour of layered ceramics with textured microstructures and may be applied to other composite materials.

Ceramic additive manufacturing in prosthetic dentistry

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Lithium disilicate ($LiSi_2$) is a unique dental ceramic due to its great optical characteristics, especially translucency in combination with good mechanical properties, such as strength and fracture toughness. The translucency of $LiSi_2$ is on par with other glass ceramics such as feldspar, whereas it is exceeding them in mechanical strength. There it is only rivaled by oxide ceramics, such as Zirconia and Alumina, which in turn do not satisfy aesthetic requirements. Therefor $LiSi_2$ is favored for restorations especially in the anterior region.

Conventional processing of $LiSi_2$ is either by hot-pressing of precrystallized blanks in the so-called lost wax technique or by milling of blocks or blanks. The former method is quite intricate, time and material consuming. Milling as a computer aided manufacturing method (CAM) is embedded in the digital workflow, where patient data is acquired by intraoral scanning and the model and restoration are designed by designated computer aided design (CAD) software. The milling process however is also material consuming, as it is a subtractive method, and furthermore it is limited in the freedom of design. Especially thin restorations, such as non-prep veneers with thicknesses much below 1 mm cannot be easily produced by milling. Additive manufacturing (AM) technologies are promising approaches to overcome these limitations of the CAD-CAM production of $LiSi_2$ restorations. Especially lithography based ceramic manufacturing is an AM technology with high resolution and precision. In this work we present first results of noninvasive $LiSi_2$ restorations produced by LCM, which show exceptional optical properties, comparable adhesion and biocompatibility to conventionally produced $LiSi_2$ materials.

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Multicriteria optimization of ceramic matrix composites considering environmental impacts and mechanical properties

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During the last 40 years development activities on ceramic matrix composites (CMC) focused mainly on the improvement of mechanical, chemical as well as thermal properties and the optimization of cost-efficient production routes. Recently, more and more fields of application have opened up for CMC, in which environmental impacts are relevant. These impacts have been barely investigated so far, but receive growing interest due to increasing awareness of the environmental consequences.

Our innovative approach sets mechanical properties in ratio to environmental impacts (e.g., Global Warming Potential in CO_2 emission) under varying production processes, which results in tradeoffs between mechanical properties and environmental impacts. First, the wet filament winding process of a C/C-SiC material has been investigated till the CFRP state, by simply changing process temperatures and the amount of used raw material. During the production of CFRP-plates, mass and energy flows have been tracked in each step. Tensile, three point-bending and interlaminar shear tests have been performed to identify basic mechanical properties. The environmental impacts are determined by a gate-to-gate Life Cycle Assessment (LCA) using the software SimaPro.

Resulting tradeoffs between mechanical properties and environmental impacts can show nonlinear behavior, thus revealing break points above which an improvement of mechanical properties is associated with significantly higher CO_2 emissions.

Hydrophobic ceramic membranes for membrane distillation

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Desalination processes are gaining momentum on a global scale driven by an increasing fresh water demand due to ongoing population growth, climate change and accelerated consumption. Conventional desalination technologies such as reverse osmosis (RO) have limitations (e.g. RO is limited due to the osmotic pressure) that make them unsuitable for the treatment of concentrated brines that are a by-product of seawater desalination. Therefore, unconventional desalination processes that are not limited by the salinity of a solutions are required to faciliate the treatment of these brines. One potentially suited process is Membrane distillation (MD). MD hybridizes the benefits of membrane-based and thermal desalination processes and could facilitate the further concentration of brines up to the saturation point enabling ZDL applications if coupled with crystallization processes. Due to their good mass transfer characteristics and intrinsic hydrophobic surface properties the use of polymeric membranes is state of the art in MD processes. However, the treatment of aggressive media (e.g. abrasive, high temperatures) does require the use of rebost membrane types such as ceramic membranes. Therefore, this study gives insight on the affect of relevant properties of ceramic membranes (e.g. layer design, thermal conductivity, pore size, porosity, wall thickness, functionalization, stability) on their performance in MD processes. Additionally, the impact of relevant process parameters (driving force, flow velocity, salinity) on the performance of ceramic membranes is evaluated.

This data in then used to assess the limitations and benefits of ceramic membranes in MD processes and to give stimuli for membrane adaptation.

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Thermal shock characterization of refractories and ceramics using in-situ methods

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Refractories are a key element for energy efficient thermal processes. Exact knowledge of their material properties under application conditions is a necessity for their successful selection. A particularly important topic is their resistance to thermal cycling and shock. Even though many techniques and standards have been described for testing and evaluation of thermal shock, most of these are based on fast, usually undefined temperature changes between high temperatures and room temperature e.g. by quenching. However, in most applications the more realistic stress takes place between two levels of high temperature. In order to investigate these conditions in more detail and with more accuracy, a new ThermoOptical Measuring device has been developed and is being constantly improved. It is based on a unique combination of a high-temperature furnace, a CO_2-laser and specific in-situ sensors. The furnace serves to establish a base temperature in a controlled atmosphere. The laser is used for short-term heating (thermal shock or cycling) of samples. To be representative even for complex microstructures like refractories, samples with a volume of up to 20 cm^3 are investigated. The integrated laser control software enables the definition of temperature gradients by pre-adapted laser power profiles, one- or two-sided laser irradiation, variable spot size and pulse rate. Recently, a module has been integrated which allows pre-estimation of thermal stresses. Accordingly, material adapted testing conditions can be adjusted and tedious pre-testing is avoided. The benefits of these developments will be presented based on recent tests on standard refractory materials.

Ceramic multi material vat photopolymerization of alumina and zirconia

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The cooperation between the competence center for additive manufacturing of ceramic materials (3DK) and Lithoz resulted in the examination and application of multi material vat photopolymerization ("MVPP") utilizing high solid loading alumina and zirconia slurries on a recently acquired Lithoz CeraFab Multi 2M30. The optimization of both a combined post-processing and thermal treatment program resulted in dense layered and especially non-layered composite structures. Printed series were tested for their flexural strength using ball on three balls method as well as bonding and defect building in composite interlayers using FESEM and optical analysis. In several tasks the curing process during printing has been surveyed using Raman spectroscopy and CT scans. Furthermore, it has been shown that even highly complex, hollow and overhanging forms can be safely printed with a composite pattern including the usage of several support structures regardless of the slurries viscosity.

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Magnesium aluminate spinel for optical applications

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During the last years, the use of polycrystalline transparent ceramics in optical components has constantly increased. Thereby magnesium aluminate ($MgAl_2O_4$) with its high hardness, temperature stability and high chemical stability among good optical characteristics is a promising material for applications which require optical transparency combined with resistance against wear and chemicals. Starting from different spinel powders, samples were shaped using following liquid shaping methods: slip casting, pressure slip casting and gel casting. The geometry of the samples includes lenses and complex shaped covers with diameters of up to 66mm The samples were pressureless sintered and afterwards hot isostatically pressed.

Typically, samples reached green densities of > 50 %. All samples complied after hot isostatic pressing with the necessary requirement of a transparent ceramic of a density of > 99,9 % of theoretical density. The inline-transmission measurement (measuring range: $200\ nm \leq \lambda \leq 800\ nm$) of a polished lens showed a maximum IT-value of 76,62 % at a wavelength of 800 nm and a thickness of 1.9 mm. With the help of RIT-measurements a clear transparency could be determined in a wavelength range of 425-705 nm with a value of RIT > 70 %. Optimized surface finishing of the lenses resulted in transparent components with a waviness Wt of 0.43 µm and an average roughness Ra of 0.023 µm.

Analysis of the influence of pore morphology and second phase on the dielectric breakdown strength (DBS) of Bayer alumina

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Dielectric breakdown strength defines the voltage at which a component will irreparably fail. The ever-increasing technical demands on electrical insulators and substrate carriers are the starting points for more and more innovations.

Thanks to automated imaging, it is possible to investigate the influence of pore morphology on the dielectric properties of Bayer alumina in more detail. Previous research has shown that pore size, number and location in the microstructure have a significant influence on the DBS. This can be extended to morphology through the use of automated microstructure imaging combined with statistical analysis.

In addition, the influence of various dopants and impurities on the material properties was evaluated. In particular, the manifold cross effects depending on the doping and composition of the second phase are discussed.

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Lithography-based additive manufacturing of silicon nitride by using CerAM VPP technology

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Hardness, high mechanical strength, wear and corrosion resistance or even heat resistance, for instance, are the most important properties of silicon nitride, which make the material interesting for various applications. This includes cutting tools, crucibles for molten metals, bearings or prostheses in medical technology. Up to now, the demands on the geometry of silicon nitride-based components could largely be met by using conventional shaping methods such as pressing and green machining or injection moulding. However, requirements in terms of complexity and function are constantly increasing. By using additive manufacturing (AM) technologies for ceramic materials, the complexity of components can be significantly increased and additional functions such as cooling channels for instance, can be integrated. High-resolution AM processes such as stereolithography enable the production of very accurate and complex ceramic components. Unfortunately, for silicon nitride the state of development in lithography-based AM processes is limited and commercial systems or service providers are not available. Therefore, the authors would like to give an insight into the development status of silicon nitride at the Fraunhofer IKTS for the so-called CerAM VPP process (Lithoz LCM technology) based on two research projects called "Fingerkit" and "Dynacool", which are concerned with the development of novel finger implants and heating-cooling elements, respectively.

Influence of dopants on contact resistance in anode supported solid oxide fuel cells (AS-SOFCs) between doped ceria and YSZ

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Anode-supported solid oxide fuel cells use Ni/YSZ as anode which makes them unflexible in using biogas due to strong degradation. To improve fuel flexibility, more degradation-tolerant materials have to be implemented. Doped ceria is promising since it shows increased degradation tolerance and higher ionic conductivities in the intermediate temperature regime and some electrochemical activity.

However, integration of ceria into a cell is difficult because high sintering temperatures are needed to achieve the necessary microstructure. Implementing ceria between the substrate and electrolyte leads to interdiffusion during sintering and the formation of a $Ce_{1-x}Z_xrO_2$ phase. In addition to lower conductivity of the mixed phase, the roughening at the interface between anode and electrolyte leads to an increase in polarization resistance of the electrode due to interdiffusion.

We investigated the influence of the dopant in $X_{0.1}Gd_{0.9}O_{1.95}$ (X = Sm, Gd, or Y) on the interdiffusion with 8YSZ. Powder mixtures of YSZ and XDC were prepared and sintered at different temperatures. The interdiffusion behavior was analyzed by XRD and Rietveld refinement. Interdiffusion in layered systems was investigated by SEM analysis. Impedance spectroscopy is used to analyze the influence of the dopant on contact resistance. SDC shows less interdiffusion, $Ce_{1-x}Zr_xO_2$ formation and a narrower interdiffusion zone. Impedance analysis indicates a different behavior compared to Ni-GDC and Ni-YSZ electrodes.

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Development of boron carbide/graphene platelets ceramics prepared by differnet processing technologies

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Boron carbide/graphene platelet (B_4C/GPLs) composites have been prepared with the addition of different weight percent and various types of GPLs by hot-press processing technology (HP), conventional Spark Plasma Sintering (SPS) and application of the novel and superfast processing method Flash Sintering (FS), which reduces sintering time only to 24 seconds. The effect of processing technology and influence of the GPLs addition on microstructure development, fracture toughness, electrical conductivity and tribological properties was investigated. The microstructure was studied by SEM, TEM, HRTEM, XRD and Raman spectroscopy. SEVNB method was used for fracture toughness and four-point Van der Pauw method for electrical conductivity measurement. Almost fully dense B_4C/GPLs composites have been prepared with lower wt.% of GPLs additives with relatively homogeneously distributed platelets in the matrix. With increasing amount of GPLs additives, the fracture toughness increased due to the activated toughening mechanisms in the form of crack deflection, crack bridging, crack branching and graphene sheet pull-out. The friction and wear behaviour of B_4C/GPLs composites have been investigated using the ball-on-flat technique with SiC ball under dry sliding conditions at room temperature. For revealing and observation of the wear damages under the worn surfaces, focused ion beam (FIB) technique was used for the preparation of the cross-section of wear tracks.

Twelve years of flash sintering: current status and perspectives

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Flash sintering was discovered in 2010 by prof. R. Raj and co-workers at the University of Colorado in Boulder (USA). It immediately appeared as a revolutionary method to consolidate ceramics in a few seconds at temperatures much lower than the conventional ones and by using relatively simple equipment. Flash sintering has attracted a strong interest from the scientific community because it could represent a promising sintering route to reduce economic, energetic and environmental costs.

Flash sintering can be defined as a consolidation process where the green ceramic component is subjected to an external electric field while being moderately heated. At an on-set combination of applied field and external temperature -this depending on the green body conductivity- the material flashes, i.e. a bright glowing, a rapid drop in the ceramic resistivity, an accelerated diffusion kinetics and almost instantaneous densification occur.
Since it discovery, flash sintering has been applied on a multitude of ceramic materials, insulators, semiconductors and conductors with the aim to understand the fundamental phenomena behind it and to identify strategies to up-scale it for the production of real ceramic components.

In this presentation, the fundamental results obtained in the past twelve years from the analysis of flash sintering phenomena and mechanisms, its applicability to different classes of ceramics, the fundamental issues related to its evolution into an industrial practice are presented. Emerging challenges associated with flash-induced innovative routes like flash-synthesis, -joining or -softening are discussed, as well, together with possible new applications in materials processing.

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Fabrication of dense NaSICON solid electrolyte films via aerosol deposition method

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Solid-state batteries are promising energy storage devices that can be widely used in near future in stationary and mobile applications. Commercial lithium ion batteries contain liquid electrolytes, which need to be replaced with nonflammable solid electrolytes to provide increased safety. NaSICON solid electrolytes have gained much attention in recent years for their remarkable electric and chemical properties. One of the most attractive candidates for sodium ion conductive solid electrolytes is NaSICON regarding its low cost and abundant resources.

The powder aerosol deposition (PAD) method enables to deposit dense films (> 95% of theoretical density) in the µm-range at room temperature on a wide variety of substrate materials. Therefore, it is to be expected that dense NaSICON solid electrolytes can be deposited with high deposition rates cost effectively by utilizing this method in contrast to conventional methods that require a final sintering step.

In the current work, we produced NaSICON solid electrolyte powders by the mixed oxide technique using a planetary ball mill. X-ray diffraction analyses including Rietveld refinement were carried out on the calcined powders. The powders with the desired phase were sprayed on different substrates via PAD. Subsequently, characterization of the electrical properties of thin films and evaluation of the effect of annealing temperature on the ionic conductivity of NaSICON PAD films were presented. Our work demonstrates a successful room temperature application of dense NaSICON electrolyte films on different substrates, which is promising for stationary energy storage applications of solid-state sodium batteries.

Structural determination of the Curie temperature with temperature-dependent X-ray diffraction on barium bismuth potassium titanate

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Barium bismuth potassium titanate (BBKT) is a potential candidate as a lead-free PTCR (Positive Temperature Coefficient of Resistance) ceramic. PTCR ceramics are used as self-regulating heating elements, where the Curie temperature (T_C) plays an important role. T_C describes the transformation from ferroelectric to paraelectric state and therefore the operating point of the PTCR ceramic. BBKT is chosen because of the working range of $T_C = 120$-380°C.
The most common method of determining T_C is to determine the relative permittivity as a function of temperature. This method reaches its limits as soon as the phase transformation changes from 1st to 2nd order. As a result, the transformation becomes diffuse and extends over a wide temperature range. In addition, the permittivity does not exhibit a clear maximum. With increasing the amounts of Bi and K in barium titanate, the phase transformation changes from 1st to 2nd order. This leads to an overestimation of T_C or complicates a determination for materials with a 2nd order behaviour.

Since T_C is defined as a, transformation from a ferroelectric to a paraelectric cubic phase, T_C can also be determined structurally. This can be done with temperature-dependent X-ray diffraction. However, the diffuse transformation over a wide range of temperatures impedes the accurate determination of T_C. In this contribution we present a method to define T_C for materials with a 2nd order behaviour.

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Effects of synthesis parameters on the fabrication of high-flux CHA membranes for CO_2/CH_4 separation

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Motivation
Burning processes, natural gas and biogas production generates huge amount of CO2. Subsequently, CO2 must be captured. CO2-separation process with membrane technology has a few benefits in comparison to other approaches. Zeolites are crystalline porous oxides, which can separate molecules based on pore size and polarity. CHABAZITE (CHA) with a pore size of 3.72 Å is an ideal material to separate CO2 from CH4 because the pore diameter of CHA is between the kinetic molecule diameters of CO2 (3.3 Å) and CH4 (3.8 Å). The aim of this work is to investigate the effective synthesis parameters on the fabrication of CHA zeolite membranes.

Experimental
The CHA membranes were fabricated on a porous ?-alumina tubes. For the preparation of the initial gel, chemicals were mixed and aged for few hours, and the hydrothermal treatment was performed for 48 h at 160 °C. After the drying step, the membrane was calcined at different temperatures to remove any possible organic impurity. The as-synthesized CHA membranes, were analyzed by XRD, FTIR, FESEM and finally single and mixed gas permeation measurement.

Results
The obtained results illustrated that how would several synthesis parameters affect the final performance of CHA membranes. Excellent CO2/CH4 selectivities of 150 were determined with the best membranes in measurements in artificial CO_2/CH_4 mixtures, which is a very good basis for further experiments on the processing of industrial gases.

In-situ graphene platelets formation and its suppression during reactive spark plasma sintering of boron carbide/ titanium diboride composites

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Boron carbide composites with 10 vol.% TiB_2 were prepared by reactive sintering of B_4C, TiO_2, and carbon black powder mixture at the temperature of 1800 ?C, under a pressure of 70 MPa in a vacuum. The combined effects of electric current and in-situ reactions led to a significant overheating of the central part of the sample, while no overheating was observed for hot press and non-reactive SPS processes. A lower electrical resistivity of TiB_2 produced a significant Joule heating of boron carbide, leading to its partial decomposition to form gaseous boron and graphene platelets. Homogenous, fully dense and graphene-free samples were obtained when employing an insulating Al_2O_3 paper during reactive SPS. A short dwell time (30 s after a degassing step of 6 min) and the uniform distribution of fine TiB_2 grains were the main advantages of isolated SPS over the reactive hot press and SPS processes, respectively.

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Structural characterization of $Na_3Fe_2(PO_4)_{3-x}(SiO_4)_x$ NaSICON and evaluation of the electrochemical properties

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$Na_3Fe_2(PO_4)_{3-x}(SiO_4)_x$ NaSICON-type compound was synthesized through liquid-phase co-precipitation method with increasing molar amounts of silica. A detailed XRD, XPS, FTIR, and Raman spectroscopic investigation carried on the as-synthesized samples revealed a progressive enlargement of the monoclinic lattice concordant to the addition of dopant, and evidenced the formation of Fe-O-Si complexes in the classical "lantern unit" structure of NaSICON. Electrochemical tests confirmed the sensitivity of the electrochemical properties to the silicon addition, particularly an improvement of the electronic conductivity at room temperature was associated with the amount of silicon substitution in the NaSICON structure.

Quality assurance of solid-state electrolytes for energy storage applications by non-contact vibration analysis

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Ceramic solid-state electrolytes play a central role in various types of batteries for energy storage applications. They must be defect-free, so quality assurance is important. In this contribution, we study non-contact vibration analysis for Na-ß-aluminate cups used as solid electrolytes in Na-$NiCl_2$ batteries.

First, extensive numerical modal analysis was carried out using finite element modelling (FEM). Beside the complete mode spectrum of the free-floating perfect component, the influence of the suspension as well as deviations from the ideal geometry to the eigenmodes were studied. Additionally, the influence of different defect parameters, such as shape, location, and size, on the eigenmodes was investigated.

For experimental study an arrangement with a soft suspension, impact excitation pendulum and near-surface microphone array rack was designed and built. Initially defect free samples with exact reference geometry were measured. Eigenfrequencies, damping ratios and mode shapes were extracted from the microphone array recordings using the operational modal analysis (OMA) algorithms, as the impact excitation signal was not traced. Experimental and numerical data have shown good agreement. Further, the samples with reference defects, induced by laser cuts of different length and position, as well as laser drilled holes were studied. Depending on their type, size and position, the defects lead to a decrease of some eigenfrequencies and to a splitting of formerly degenerate modes. Same effects are shown for a real crack.

Based on these results, preliminary application boundaries and potential development patterns for non-contact modal testing to detect defects in the electrolyte cups are discussed.

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Ablation resistance of ZrB_2–SiC composites prepared by reaction sintering of $ZrSi_2$, B_4C, C, and rare-earth based additives

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ZrB_2-SiC composites were fabricated using Field Assisted Sintering Technology (FAST). The composites containing 25 vol% SiC particles were prepared by in situ reaction of $ZrSi_2$, B_4C and carbon black powders, which is a method to densify ZrB_2-based composites at low temperatures. Furthermore, rare-earth (RE) based additives were used to improve the ablation resistance of ZrB_2-SiC composites. The microstructures of the ZrB_2 based composites were characterized by X-Ray Diffraction and Scanning Electron Microscopy. Both the room temperature (hardness, strength, fracture toughness) and high temperature (ablation resistance using an oxy-acetylene flame with a temperature up to 2700 °C) properties were investigated. The results showed that homogeneous microstructure and nearly fully dense ZrB_2-25vol.%SiC composites with a relative density above 99% were obtained after sintering at the temperature of 1600°C under the pressure of 70 MPa for 10 min. During sintering, the additives were completely transformed into ZrB_2 and SiC particles, which were homogeneously distributed in the ZrB_2 matrix. The RE-based additives were also uniformly distributed at the grain boundaries of ZrB_2. The mechanical properties of ZrB_2-SiC composite, such as hardness, strength and fracture toughness, were slightly improved by the addition of RE additives. The ablated zone of ZrB_2-SiC consisted of three distinct ablation layers. Most importantly, the ablation resistance of ZrB_2-based materials was significantly improved by the addition of RE, and further improved with their increasing amounts.

Alteration of microstructures by stressing surfaces and interfaces

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The application of external mechanical, electrical or chemical stress on surfaces or interfaces has the ability to change surface and interface energies. For instance, during sintering the application of electric fields can accelerate densification of powder compacts, lower processing temperatures, and suppress or enhance grain growth. In this presentation a series of recent bicrystal diffusion bonding experiments will be reviewed that were designed to interrogate electric field effects on the atomic and electronic structures of individual grain boundaries. The results demonstrate the ability to alter grain boundary configurations and, hence, interface energies in an emerging microstructure. In a separate study growth of NiO nanorods from individual Ni nanoparticles was observed during in-situ electron microscopy at high temperature and in the presence of water vapor. High-aspect ratio growth of NiO is favored for sufficiently high total surface energies. In-situ high resolution transmission electron microscopy experiments were used to explore growth modes.

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Molecular sieving graphene-based membranes produced by dip-coating

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Many extraordinary properties have been reported for graphene, a 2D carbon nanostructure, and its oxygenated derivative graphene oxide (GO). A few years ago, it has also been discovered that thin films made of GO show unique permeation properties for small molecules and, to date, a few groups have been able to prepare GO membranes with nanofiltration, desalination, pervaporation and gas separation properties. However, before such membranes can be practically used, there are several subjects that remain to be addressed, such as making large areas of membranes on porous ceramic supports and the stability of the material.

The membranes reported here are fabricated by dip-coating a water-based GO dispersion on a specially designed 8YSZ mesoporous support, which has been recently commercialized. The latter consists of two 8YSZ membrane layers with a pore size of ~5 and ~3 nm on top of a macroporous a-Al_2O_3 support disc or tube. The resulting macro-/meso-/microporous membrane systems are found to be permeable for the smallest molecules He, H_2 and H_2O, while larger molecules are rejected. For example, with the use of a reductive thermal treatment at 750 °C, a 10 nm thick graphene type membrane with high permselectivities (> 30) for the gas pairs He/N_2, H_2/CO_2, H_2/N_2 and H_2/CH_4 is obtained. High H_2O permeance and selectivity is achieved for the GO type membrane, which shows H_2O separation selectivities as high as > 500 in pervaporation with H_2O/propanol mixtures.

Glycerol-modified SBA-15 rods as bioactive and biodegradable platforms for the fabrication of multifunctional composite systems

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There is a great clinical demand for smart artificial materials intended to repair or regenerate human tissues. SBA-15 mesoporous silica has been established as an ideal system due to its outstanding features, including its interconnected mesoporous network and silanol-rich (SiOH) surface. Moreover, its versatility allows it to act as starting platform for the production of a wide range of complex structures such as 3D hybrid scaffolds, theranostic devices, 3D printed composite hydrogels, electrospun membranes, etc. This work reports the synthesis and characterization of degradable and bioactive SBA-15 rod particles and the influence of the addition of glycerol and phosphoric acid (H_3PO_4) on their degradability and bioactivity. Solvent extraction for surfactant removal played a key role in the final properties, making it possible to maintain/produce a large number of free silanols at the silica surface. The formation of needle-type hydroxycarbonate apatite (HCA) crystals was observed after 14 days of immersion of the SBA-15 particles in simulated body fluid (SBF). The size of the rods was adjusted by the incorporation of H_3PO_4, increasing their length with decreasing the ratio and concentration of H_3PO_4. Degradability was facilitated by the addition of glycerol and H_3PO_4, being higher when the concentration of H_3PO_4 increased. The synthesised SBA-15 rods can be thus considered suitable candidates for the fabrication of multifunctional biomedical devices.

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Mullite-based ceramic composite for fused filament fabrication of complex ceramic shapes

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The extrusion of composite ceramic/polymer material into filament was performed from pre-mixed materials in single homogenisation step using double screw extruder with ceramic 65 wt. % powder loading.

The printability of prepared filament was tested by production circular segments of scaled down industrial steam desalination turbine model from General Electric were used. The models were obtained from public repository (CC-Attribution license). The segments were up?scaled and printed using commercial direct-drive printer Creatr, the Repetier-Host 2.0.1, and slicer software Slic3r 1.3.0.

The tested material showed good printability and all the objects kept their characteristic features. The defects created in the AM process are unaffected by the thermal processing. These findings suggest the examined material can be a suitable feedstock for preparation of complex ceramic shapes in applications that benefit from the nature of the ceramic materials - heat-resistant components, complex-shaped insulation elements, etc.

Preparation of fully dense boron carbide ceramics by fused filament fabrication (FFF)

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Boron carbide is a refractory material with excellent properties such as hardness, wear resistance, low density, etc. However, the shaping and sintering of boron carbide components with complex geometries without additional machining remains a challenge. This work is focused on the preparation of fully dense boron carbide ceramics fabricated by fused filament fabrication. Newly developed composite filament containing 65 wt. % of micron-sized boron carbide powder was tested on a commercial low-cost desktop printer with a 0.4 mm nozzle for manufacturing complex-shaped B_4C ceramics with dimensions up to 4 centimeters. The 3D-printed objects were successfully debound and sintered at 2300°C. The samples after sintering achieved relative densities higher than 96 %.

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Multi-material printing of reaction bonded carbides by robocasting

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In the present work, reaction bonded boron carbide (RBBC) and reaction bonded silicon carbide (BRSC) composites were fabricated using multi-material robocasting followed by liquid silicon infiltration (LSI). The fabrication of carbide based materials using LSI is of great technological interest because it avoids the need for the relatively high temperatures and pressures required in traditional fabrication. Multi-material robocasting offers the possibility of combining materials and thus their properties, as demonstrated in this work with silicon carbide (SiC) and boron carbide (B_4C).
The feasibility of combining RBSC and RBBC was demonstrated by printing multilayer bending bars. The microstructure of the samples was investigated and, in particular, the interfaces at each processing step were studied. After LSI, cracks were observed in the B_4C layers due to differences in thermal expansion, but the interfaces showed no delamination and good interfacial bonding between the layers was evident. Hardness measurements were performed to investigate the interfacial bonding and residual stresses in the materials used. The manufactured RBSC and RBBC samples showed a hardness of 18.3 GPa and 17.1 GPa, respectively. The residual stresses in the layers were calculated theoretically and also based on the crack pattern generated by Vickers indentation. Tensile stresses in RBBC and compressive stresses in RBSC were determined and can explain the cracking in the laminated structures.

Robocasting of reaction bonded silicon carbide/ silicon carbide platelet composites – Influence of porosity on infiltration with liquid silicon

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Reaction bonded silicon carbide (BRSC) and reaction bonded silicon carbide/ silicon carbide platelet composites were fabricated using robocasting followed by liquid silicon infiltration (LSI). During printing, the anisotropic SiC platelets align in the printing direction due to shear forces. This texturization was confirmed by SEM image analysis and EBSD measurements.

It was found that the LSI result was strongly dependent on the heat treatment of the robocasted samples and thus on their porosity. Especially the composites with platelets showed strongly different results depending on whether the samples were sintered or only pyrolyzed. Samples without platelets were successfully infiltrated after pyrolysis and after sintering, whereas samples with platelets could only be completely infiltrated after the additional sintering step. In order to investigate this aspect further and to determine the influence of the platelets, the pore size, pore distribution, and structure were investigated by means of µCT and mercury porosimetry.

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Numerical simulation of electric current assisted sintering of 8YSZ

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Electric current assisted sintering (ECAS) is a promising powder consolidation technique which can achieve short-term sintering with high heating rates and low dwell times. Currently main methods of performing ECAS are indirect heating of the powder compact in a conductive tool or direct heating with current flowing through the conductive powder compact. Various factors can lead to the rapid densification during ECAS, such as ultra-high heating rates, extra-high temperatures, and electric field. However, the determining factor and the key mechanism are still under debate. This study aims at understanding the densification mechanisms during ECAS of 8 mol.%Y_2O_3 doped ZrO_2 (8YSZ) by finite element (FE) simulation. Two different heating modes, ultrafast high-temperature sintering (indirect heating) and flash sintering (direct heating) were studied. A numerical strategy was developed to predict the thermal profile, densification, and grain growth during both sintering processes. Furthermore, a comparison was made with conventional sintering to investigate the densification mechanisms besides the conventional thermal effect. In addition, the electrical polarity on microstructure during flash sintering of 8YSZ was also investigated, the simulation results of grain size inhomogeneity agree well with the experimental data.

Ni/YSZ solid oxide fuel cell anodes: correlation of microstructural aging with electrochemical behavior

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The solid oxide fuel cell (SOFC) offers great potential for stationary energy conversion applications because of its high combined heat and power efficiency. Identification of the predominant aging mechanisms during cell operation is therefore of great interest to optimize material selection and manufacturing conditions. This also concerns the ceramic-metal framework of the nickel/yttria-stabilized zirconia (Ni/YSZ) anode, which is highly dependent on an intact electronic transport path through the nickel due to the nature of pure ionic transport in YSZ.

This contribution presents a study on the impact of a variation in operation parameters such as temperature and fuel humidification on Ni/YSZ anode aging. The electrochemical performance of the anode-supported cells was monitored by electrochemical impedance spectroscopy. We combine optical microscopy, focused-ion-beam/scanning-electron-microscopy (FIB/SEM) tomography, and energy dispersive X-ray spectroscopy (EDXS) combined with scanning transmission electron microscopy (STEM), with equivalent circuit modeling to visualize predominant aging mechanisms. It will be shown that local microstructural aging is superimposed by long-range loss in electrical connectivity of the Ni-phase. Therefore, the amount of electrically isolated nickel is a key indicator for understanding the long-term electrochemical behaviour of Ni/YSZ anodes.

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Controling carrier diffusion via strain in methylammonium lead iodide perovskite

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Polycrystalline thin films and single crystals of hybrid perovskites – a material group successfully used for photovoltaic and optoelectronic applications – reportedly display heterogeneous charge carrier dynamics often tied to grain boundaries or crystalline strain. We found an anisotropic carrier dynamics directly correlated with the arrangement of ferroelastic twin domains in methylammonium lead iodide ($MAPbI_3$) perovskite crystals. These domains form to release internal strain introduced by the cubic-tetragnonal phase transition. Comparing diffusion constants parallel and perpendicular to the domains showed carriers diffuse around 35% faster parallel to the domains. Extensive piezoresponse force microscopy (PFM) experiments on the nature of the domain pattern suggest that the diffusion anisotropy most likely originates from structural and electrical anomalies at ferroelastic domain walls. We believe that the domain walls pose as shallow energetic barriers, which delay the transversal diffusion of carriers. To control the strain and thereby the arrangement of ferroelastic domains in $MAPbI_3$, we developed a chemical method for controlling the strain in methylammonium lead iodide ($MAPbI_3$) perovskite crystals by varying the composition in the precursor solution. The PFM images show an increase in the average size of ferroelastic twin domains upon increasing the $PbCl_2$ content, indicating an increase in crystal strain. Additional time-resolved photoluminescence (TRPL) show an increased carrier lifetime in the $MAPbI_3$ films prepared with higher $PbCl_2$ content, suggesting a decreased trap density in films with larger twin domain structures. The results demonstrate the potential of chemical strain engineering as an easy method for controlling strain-related effects in lead halide perovskites.

Multi material jetting (CerAM MMJ) of ceramic-based functionally graded components (FGC)

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Ceramic-based functionally graded Components (FGC) are ceramic parts with a variety of different properties, which change gradually throughout their dimensions by realizing a graded microstructure or material gradients. As a result, innovative, multi-functional property combinations, such as hard and ductile, electrically or thermally conductive and insulating or magnetic and nonmagnetic combinations can be created. Applications are conceivable in a variety of industrial and medical fields - for example, as cutting tools, wear resistant components, ceramic heaters, energy and fuel cell components or as bipolar surgical tools.

Multi Material Jetting was specially developed for the manufacturing of ceramic-based functionally graded and multi material components. In our study, we investigated the AM of ceramic-based Functionally Graded Components (FGC) by CerAM MMJ, like zirconia components with a varying microstructure as well as Si_3N_4 -based components, which combine an electrically conductive phase and a non-conductive phase.

The Multi Material Jetting technology is currently in the commercialization phase. The project is being launched as part of the EXIST research transfer programme "EXIST: CerAM MMJ" (funded by the BMWi, project management organization PTJ, funding code: 03EFQSN180). The presentation will give an overview of the current state of the technology development, the available material portfolio, some applications and the progress of the commercialization activities.

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Investigations of the structure formation of hierarchical porous Freeze Foams for use as bone graft substitutes

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The combination of foaming gas and freezing ice during the Freeze Foaming (developed by Fraunhofer IKTS) of biocompatible materials results in a hierarchical porous structure consisting of macro-structural foam cells and micro-structural porous struts. Porous calcium phosphate ceramics are highly suitable as bone graft substitutes due to their proven biocompatibility. In a successfully completed DFG-project main process influencing parameters for controlled foaming of hydroxyapatite ceramics were identified.
In the follow-up project it is of interest to control both pore hierarchies, the foam cells, and the strut pores, with the aim of developing tailored foams for biomedical applications. Following this aim, a model-suspension with stable foaming behavior was chosen to investigate the influence of most important process parameters: the water content of the suspension, temperature, air content and the pressure reduction rate during foaming.
Furthermore, the strut pore formation was investigated in detail by varying pressure reduction rate and the temperature of the freeze dryer shelf, which resulted in different freezing rates.
Foam cells were examined by computer tomography analysis and strut pores with electron microscopy and subsequent microstructural analysis.

This way insights in the mechanisms during foam growth, but also during freezing of the foam could be gained. A discrete investigation of macro- and microstructure development was possible for the first time. With the help of the gained knowledge, it will be possible to manufacture tailored Freeze Foams for specific biomedical applications, but also others like catalyst support or thermal insulators if different materials are used.

Development of functional ceramics for optical and lighting applications

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Since the development of the first highly translucent ceramics based on polycrystalline alumina for high-pressure vapor lamps in the 1960s, a very dynamic and innovative field of research and development with a wide range of applications has emerged in the form of light-transmitting ceramics. After a historical review with examples from a wide range of applications of optically passive and active ceramics, own work on luminescent converter ceramics for light-emitting diodes (LEDs) as well as highly refractive ceramics will be presented as examples. In addition to the very application-specific materials and microstructure design, the special requirements for material selection and synthesis and further manufacturing steps in the ceramic process chain will also be discussed. Finally, an outlook is given on possible future developments and applications.

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New steel melt filtration approaches in industrial ingot casting and continuous casting of steel

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Melt filtration is a popular approach to remove non-metallic inclusions from metal melts, especially in foundry processes. In steelmaking processes, the application of filters usually is significantly more challenging due to the higher melt temperature, larger volume of the processed melt batches and long casting durations, especially during continuous casting. Premature clogging, corrosion or failure due to thermomechanical stresses are very demanding for common filter materials and implementation concepts. In order to address these challenges, new filtration approaches for industrial casting processes are investigated in the framework of the Collaborative Research Center 920.

In cooperation with the company Deutsche Edelstahlwerke Specialty Steel, different concepts for improved melt cleanliness are investigated. Foam filters with tailored geometry were developed based on the replication of foam templates produced by selective laser sintering and subsequent flame spraying. First prototypes of these filters were already tested in industrial bottom-teeming ingot casting. Furthermore, alumina feeders for ingot casting were conceptualized and manufactured by pressure slip casting. The functionalization of the feeders with reactive carbon-bonded coatings and the application of flame-sprayed filters with active filter surface enable the combined operation of reactive and active filtration mechanisms in one casting process. For continuous casting, extruded filter materials were investigated regarding their mechanical and structural properties as well as their thermal shock resistance. Based on the findings, first prototypes of cellular casting starter tubes for the application in the tundish were developed.

Ceramic membranes with unique properties through engineering the pores on the nanoscale

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Membranes, based on ceramic materials, are interesting for industrial applications under aggressive environments, because of their robustness, and can therefore be applied under several demanding conditions, like industrial gas separations and recovery of products and (organic) solvents. Not only robustness, but also pore size and membrane-feed interactions are important parameters to control, in order to obtain membranes with the required separation properties. In the presentation fabrication, microstructure and transport properties will be discussed of several types of ceramic membranes, divided in three categories modifying ceramic pores with nanotunable polymer brushes zeolite membranes with tuned properties In the context of modifying porous ceramics with organic brushes a/o examples of PDMS, and PI as well as a novel strategy for the preparation of a thin thioether-based crosslinked membrane layer on top of a ceramic support for Nano Filtration (NF) application are treated. Finally, strategies are discussed for the reproducible fabrication of defect-free zeolite membranes, having uniform pore sizes and high thermal/chemical stability. Elimination of defects between zeolite crystals in the membrane is a challenge in order to obtain separation on the sub-nanometer level with high permeances of the desired product. In conclusion, the inherent chemical and mechanical resistance of ceramics coupled with the multitude of possible modifications can result into three major benefits for ceramic membranes: long membrane lifetimes, suitability to harsh conditions, and the ability to be tailored to a large range of applications.

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$NaNbO_3$-based antiferroelectric ceramics for energy storage

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Antiferroelectric (AFE) materials are currently being considered for a variety of electronic applications, including high-energy density capacitors and electrocaloric refrigerators. These applications are mostly based on the electric-field-induced phase transition from an AFE state to the corresponding ferroelectric (FE) state. $NaNbO_3$ is a prototypical lead-free AFE material, but it is characterized by irreversible phase transition behavior, which makes it impractical for energy storage applications. Moreover, systematic studies of the fundamental mechanisms and effective strategies to make the transition reversible are still missing.

In this work, the irreversible phase transition of $NaNbO_3$ from Pbcm to $P2_1ma$ phase is investigated using a combination of ex situ and in situ electrical and structural characterizations. To modify the irreversible nature of the phase transition, a new composition (1-x) $NaNbO_3$-$xSrSnO_3$ was developed using first-principles calculations. Characteristic double polarization hysteresis loops were observed in the new material with an energy storage density of 0.90 J/cm^3, which is more than 7 times higher than that of the unmodified $NaNbO_3$ (0.12 J/cm^3). However, the energy storage efficiency is still low (21%) due to the large remanent polarization. The defect chemistry and microstructure of the newly designed material were further tailored by doping and thus the energy storage density was improved to 1.70 J/cm^3 with an efficiency of 33%. Moreover, both the density and efficiency were found to be temperature independent over a wide temperature range of 20 140 °C.

Synthesis of niobium-alumina composite aggregates and their application in coarse-grained refractory ceramic-metal castables

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Niobium-alumina aggregate fractions with particle sizes up to 3150 µm were produced by crushing pre-synthesised fine-grained composites. Phase separation with niobium enrichment in the aggregate class 45-500 µm was revealed by XRD/Rietveld analysis. Phase assemblage and morphologies of the synthesised aggregates are characterised by SEM and SEM/EBSD.

No organic additives were used for the castable mixtures to reduce the amount of carbon-based impurities, which resulted in water demands of approximately 27 vol.% for the fine- and coarse-grained castables. As a consequence, open porosities of 18 % and 30 % were determined for the fine- and coarse-grained composites, respectively. Due to increased porosity, the modulus of rupture at room temperature decreased from 52 MPa for the fine-grained composite to 11 MPa for the coarse-grained one. However, even the compressive yield strength decreased from 49 MPa to 18 MPa at 1300 °C for the fine-grained to the coarse-grained composite, the latter showed still plasticity with a strain up to 5 %.

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Posters

Dislocation and grain boundary interaction in oxides: slip transmission or cracking?

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Grain boundaries (GBs) play a crucial role on the mechanical properties of polycrystalline materials. Grain boundaries could act as effective barriers for dislocation glide. For brittle ceramic materials, the interaction between dislocation and grain boundary has a crucial influence on the deformation behavior. Here, we study the room temperature GB-dislocation interaction in $SrTiO_3$ (STO) as a model oxide material, with bi-crystal samples having low- and high-angle GBs. Large ball Brinell indentation was carried out close to the GBs to generate dislocations without cracks and interact with GBs. The dislocation structure was revealed by chemical etching, and etch pits were characterized using optical and scanning electron microscopy to study the dislocation-GB interaction. Depending on the GB type, we found both slip transmission across GB and GB cracking occurred. For low-angle GB, evidence is shown that dislocation could propagate through the GB into the adjacent grain. Whereas for high-angle GB, dislocation pile-up and cracking dominate. Our findings provide insights into potential improvement of deformation of polycrystalline oxides at room temperature via grain boundary engineering.

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Silver based metallization pastes for next generation sensors in harsh environments designed by the SiCer technology

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The combination of silicon chip technologies with micro-electro-mechanical systems (MEMS) is opening the door to robust sensing in harsh environments and improved cost efficiency at the same time. The patented SiCer technology allows a unique combination of silicon technology (Si) with ceramic multilayer technology (Cer). Several design concepts were established and this innovative substrate technology is gaining importance in sensor integration (Stehr et al. 2019). The LTCC is adopted to the thermal properties of the silicon wafer to ensure strong bonding at the silicon-LTCC interface. This contribution focuses on the development of a silver-based metallization that is suitable for cofiring within the LTCC during the SiCer process. To benefit from the low costs of silver in comparison to gold, challenges like strong interaction with the LTCC, silver migration, adjustment of shrinkage or enhancement of solder wettability needed to be overcome. The elaborated paste family will strengthen the competitive position of sensors designed and produced by SiCer technology.

Effect of crystallite size of Si/Graphite anodes on electrochemical properties for li-ion batteries

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Increasing the lifetime along with the capacity of Li-ion batteries by changing the production methods of the base materials, implementing or doping new elements into the active materials, is quite challenging for researchers. The main aim of this study was to optimize the ball milling conditions and obtain contamination-free Si/Gr composite powders for better cell performance. In the first step, 20:80 Si/Gr powders were used as active material and 4factor-2level full factorial design was used for finding main effects and interactions. Contamination levels were investigated by using weight loss of milling balls. SEM analyses were carried out to check the microstructure of milled composite powders. Weighted average composite grain sizes were calculated by using XRD data and D10-D90-Span was obtained with particle size distribution analysis. Regression equations were created with high R-sq(adj) values respectively %91, %93, %90 for contamination, crystallite size and span. Three different powders from different ball milling conditions were chosen to compare the effect of crystallite size on charging-discharging capacity at different current densities from 0.2 A/g to 4 A/g. Si/Gr anodes were prepared by coating N-Methyl pyrrolidone-based slurries on Cu foil and compositions were 60:30:10. Specific charge-discharge capacities were calculated and results showed that smaller crystallite size provides higher capacity with lower first cycle coloumbic efficiency. It was determined that thicker and continuous SEI formation due to the high surface area increased the irreversible capacity loss. At 1 A/g, it was reached to %80 SoH value at 600th, 1150th, and 1500th cycles with following 23nm, 36nm, 38nm weighted average crystallite sizes.

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Phase development of inorganic binders under conditions simulating geothermal wells

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Multicomponent cement pastes based on the Class G cement substituted by 30 wt.% of binary or ternary mixtures of silica fume (SF), metakaolin (MK), and ground granulated blast-furnace slag (BFS) were submitted to hydrothermal curing at 150 °C and 18 MPa for 7 days. A detailed study of the formed phases and their relationship to compressive strengths (CS) was performed by a combination of X-ray diffraction, Fourier Transform Infrared analyses in the mid-IR region, and thermogravimetric-differential scanning calorimetry. The structure and the amount of the formed phases depended on the used combination of additives, at which their reactivity and the CaO/SiO_2 ratio seemed to be the key factors. The highest amount of C-S-H and C-A-(S)-H phases disposing of the highest polymerization degree and leading to the highest value of CS (~ 48 MPa) was determined for SF – MK combination when they were used in the same amount. The majority of the phases had an amorphous character and among the detected crystalline phases belonged thermally stable tobermorite, hibschite, and katoite. The CS of the samples prepared with the content of BFS did not differ significantly (~ 10 MPa), which was caused by the lower amount of the formed hydration products as well as by the higher condensation degree of their structure. When a higher amount of BFS was used, temperature-induced decomposition and transformation of primary hydration phases led to the formation of crystalline ?-C_2SH and the decrease of CS.

Grain size correlation with field-induced ferroelectric phase transformation in barium titanate ceramics

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Piezoelectric materials are part of our daily routine: whether as microelectronics, sensors or actuators. Barium titanate (BT) is the classic model for ferroelectric systems and it is indispensable for dielectric applications. In addition, its properties are highly temperature and grain size dependent. BT has excellent properties with grain sizes around 1 µm, but undergoes marked weakening as the grain size decreases. However, the fundamental mechanisms behind the electromechanical behaviour are still not fully understood. A wide range of unimodal grain size distributions between 0.4 µm and 15 µm was successfully sintered via spark plasma sintering (SPS), quenched sintering and two-step sintering, avoiding abnormal grain growth. Samples with intermediate grain size, showed excellent electromechanical and dielectric properties. They possess a balance between microstructural strain, existence and mobility of domain walls, which in turn allows the field induced crystal phase transformation. In order to elucidate the structural response to an applied electric field, the samples were measured with in situ high energy X-rays. The structural data obtained were processed by means of STRAP and the field induced phase transformations were quantified. This induced phase transformation is stronger in samples whose grain size distribution curve is located around 1 µm. These results contribute to the understanding of fundamental questions about the ferroelectric effect.

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Optimization of reactive DC magnetron sputtering and high target utilization sputtering of hard TiZrHf(Me)-N (Me = Nb, V, Ta) coatings and their thermal stability

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The introduction of several refractory metals into ceramic coating systems offers a possibility of significant increase of their thermal stability combined with sufficiently high hardness. Thus, multicomponent ceramic systems analogous to high entropy ceramics are promising candidates for coatings for extreme conditions. The aim of the current work was to investigate the influence of the additions of Nb, V or Ta in TiZrHf targets sputtered in Ar + N2 atmosphere during reactive DC magnetron sputtering and High Target Utilization Sputtering (HiTUS) on hardness and thermal stability of the obtained nitride coatings in vacuum.
The microstructure and subsequent X-ray diffraction observations showed, that the coating structures vary from practically amorphous in pure metallic state to nanolamelar textured when stoichiometry of the content of nitrogen in the coating increased with the increase of the flow of in the sputtering atmosphere. Their hardness and indentation moduli increased from around 15 GPa in metallic state to over 30 GPa when nitrogen content reached stoichiometric composition. Thew highest mechanical properties were obtained with Nb additions in both, DCMS and HiTUS made coatings. The annealing in vacuum at 1000°C and 1200°C for 5 min resulted in small changes in phase composition related mostly to the growth of crystallite size and relaxation of microstrains present in the coating in the as-deposited state. The relaxation and growth processes induced by annealing resulted in certain degradation of mechanical properties which varied depending on type of additional dopant.

Photocatalytic properties of Ti-Mo-O nanotube arrays

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Mo-Ti-O nanotube arrays were prepared by anodic oxidation of co-deposited Mo-Ti precursor films. D? magnetron sputtering from combined Mo-Ti target allowed to increase the molybdenum content in the precursor film up to 60 at. %. The anodization was performed in ethylene glycol/ ammonium fluoride electrolyte ($C_2H_6O_2 + NH_4F$) using a two-electrode configuration with a platinum rod as a counter electrode. Nanotubes with a length of about 450 nm, where the photocatalytic activity was optimized, were obtained at 40 V, 30 min, and sample-cathode distance of 50 mm. The current density was kept at 1,5 µA per cm^2. The samples were annealed in a tube furnace at 600 ºC for 2 h in the 5% H_2/ Ar atmosphere. Mo-Ti-O nanotube showed good photocatalytic activity towards Rhodamine B degradation under simulated solar light irradiation. The degradation extent was ~65% after 4 h with a photocatalytic rate constant (***k***) of 0.00759 min^{-1} per cm^2 of the optimized sample.

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A simulation-based sustainable approach for energy savings and CO_2-reduction

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Ceramics have a wide application area and are very important for new innovative products to enable new technologies and applications, e.g. in the field of renewable energies and sustainability or aircraft turbine components. Processing of ceramic components is very energy intensive, particularly the firing process. In this work, as an example for e highly energy intensive ceramic process, graphitization of cylindrical rods is investigated by FEM-based process simulation. Typically, graphitization is performed in so called lengthwise graphitization furnaces by direct resistive heating. The product to be graphitized is arranged in parallel columns called strands. Such a furnace is isolated by special coke types called packing media. Energy consumption is calculated for different product assemblies within the furnace. Furthermore, the effect of the thermal isolation is investigated by varying the isolation material (packing media). Finally, different firing curves are assumed. For all configurations, the energy consumption is calculated to reach the same final product temperature. Based on the simulation could be shown, that energy savings of up to 20% could be achieved. Based on German electrical energy mix in 2020, CO_2 emissions between 1.1 and 0.84 kg per kg product are calculated. This simulation-based approach could be transferred to other ceramic processes and even to the hole ceramic process chain. This would enable to identify and assess promising energy saving measures.

Synthesis and characterization of lithium-doped mesoporous bioactive glass nanoparticles for bone regeneration applications

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Mesoporous bioactive glass nanoparticles (MBGNs) have the potential to be extensively used in various biomedical applications due to their highly ordered mesoporous arrangement, nanostructure, and large specific surface area. Incorporating metallic ions with pro-osteogenic effects such as lithium (Li) into MBGN is an attractive strategy for developing biomaterials with enhanced hard tissue regeneration/repair. This study investigated the effects of Li incorporation into MBGNs (Li-MBGNs) on the morphological characteristics, bioactivity, and cytocompatibility of osteoblast-like cells (MG-63). MBGNs were prepared in the system 70 SiO_2 – 30 CaO (mol%) with lithium substituting calcium in different amounts, ranging from 5 to 20 mol% by a modified Stöber process. All synthesized particles were amorphous structures and exhibited spherical or ovoid shapes measuring approximately 200 to 300 nm in diameter. In comparison to undoped MBGNs, Li-MBGNs demonstrated higher bioactivity through the formation of hydroxyapatite crystals in SBF solution. Furthermore, the water-soluble tetrasodium (WST) test confirmed that Li-MBGN was biocompatible and promoted the proliferation of MG-63 cells. The results suggested that the substitution of Li in MBGNs may be successfully used as a candidate for bone void fillers.

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Development of proton conducting electrolysis cells with tailored interface design

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The total energy transition from carbon based to climate-neutral fuels requires green hydrogen as energy carrier. The innovation pool "Solar Hydrogen: Pure and Compressed" collects research efforts regarding green hydrogen within the Helmholtz Association. The Institute of Energy and Climate Research IEK-1, Forschungszentrum Jülich GmbH partakes with a project on Solid Oxide Electrolysis Cells (SOEC) with proton-conducting electrolytes (PCEC). These cells are closely related to the conventional cells using oxygen-ion conducting electrolytes, yet they differ in two ways. Firstly, they can operate at lower temperatures. Secondly, the generated hydrogen is pure because it is not diluted with steam and can therefore be compressed directly without an additional gas separation step.
The goal is to develop multilayer proton-conducting electrolysis cells with a tailored interface design and improved performance by means of industry relevant scalable methods such as tape-casting. Such a cell consists in the simplest architecture of a thin electrolyte layer made of $BaZr_{0.7}Ce_{0.2}Y_{0.1}O_{3-d}$ and $BaZr_{0.4}Ce_{0.4}Y_{0.1}Yb_{0.1}O_{3-d}$; a fuel composite electrode made of the electrolyte material and Ni; a steam ceramic electrode based on e.g. $BaCo_{0.4}Fe_{0.4}Zr_{0.2}O_3$.
A systematic characterisation of the shrinkage rates, thermal expansion, microstructures, phase compositions of single layers and assemblies were explored aiming at determining the optimum conditions for processing (incl. co-firing, lamination) of flat and reproducible cells with reasonable thermal and chemical compatibility between the constituting layers.

Rapid thermal sintering of screen-printed $LiCoO_2$ films

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A rapid photonic sintering process (rapid thermal processing) was used to obtain compact films of cathode active material of lithium batteries. $LiCoO_2$ (LCO) thick films were screen-printed on a steel substrate followed by a contactless thermal processing at 1000 °C in only 90 seconds. A homogenous densification of such layers could be verified by scanning electron microscopy, while the structural consistency of the LCO phase in these layers could be detected by X-ray diffraction and Raman spectroscopy. Furthermore, electrochemical activity of the layer was confirmed by electrochemical cell tests with a liquid electrolyte. This is the first report of the manufacturing of highly dense LCO cathode layers by photonic sintering on conventional steel substrates.

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Artificial intelligence-supported analysis of the porosity of functional ceramic microstructures using optical image segmentation

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The further development of functional ceramic components depends strongly on the analysis of the microstructure. Conventional analysis methods, such as buoyancy weighing, can only capture insufficient data regarding the distribution and number of pores in the microstructure. By using automated image analysis, it is possible to capture significantly more pores than with classical methods. In this context, an AI-supported evaluation of segmented microstructural images helps to classify the measurements and provides the statistical basis for a comparison with material characteristics resulting from these microstructural features. The use of coaxial illumination in optical microscopy helps to significantly accelerate the speed of analysis compared to SEM-based methods.

Nanoceramic fibrous materials and composites prepared by needle-less electrospinning

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Electrospinning has been known for decades, but in recent times, it received a new wave of interest because of its great potential and possibilities. It is universal easily tunable technology, thanks to which a wide range of fibrous materials and composites can be produced. Heat treatment in different atmospheres or plasma treatment allows the transformation of the precursor fibers directly to a ceramic, carbon or composite fibers, which combines the properties of ceramic and polymer materials. Due to the specific properties of nanofibers, such as high surface-area-to-volume ratio, high porosity, appreciable mechanical strength, these materials in nanofibrous form show better characteristics, compared to bulk ones, and are used in advanced composite materials. This study describes in detail the production of different oxide and non-oxide ceramic micro/nanofibers prepared from single precursor material. As a precursor were used composite titania/polyvinylpyrrolidone microfibers prepared by new needleless electrospinning technology. The fabrication process consists of three steps: i) preparation of spinning solution; ii) electrospinning; iii) post spinning treatment – heat treatment (conventional calcination or pyrolysis in argon, for the transformation of electrospun precursors to ceramics) and DBSPD – low temperature dielectric barrier surface plasma discharge in air (for composites preparation). For spinning solutions preparation polyvinylpyrrolidone, titanium tetraisopropoxide, ethanol, and acetic acid were used. The influence of the plasma and heat treatment including its atmosphere and temperature on the phase/chemical composition and grains size of the final fibers were studied and described.

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Bulk vs thermal sprayed alumina for insulation applications: A comparison of electrical and dielectrical properties

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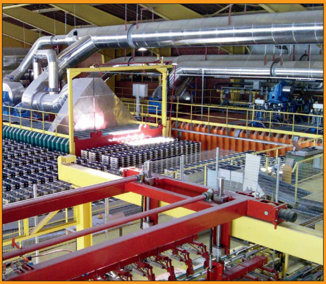
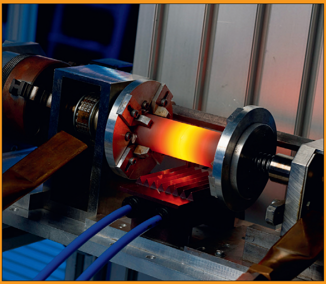
Additive manufacturing (AM) processes are opening new design possibilities for large scale electrical devices such as power generators. Conventional manufacturing methods use copper rods which are wrapped, vacuum impregnated, bend and welded. These processes are labor-intensive and time-consuming. The introduction of AM methods for manufacturing the copper conductor and electrical insulation can reduce the size of the generator head, the most complex part of the generator.

In this study, the electrical and dielectrical properties of additively deposited ceramic layers are investigated and compared with the properties of conventionally fabricated bulk ceramics. The ceramic layers are thermally deposited by atmospheric plasma spraying of a commercially available alumina powder. Bulk ceramics are fabricated by dry pressing and sintering of the same powder. Microstructure and porosity were analyzed by scanning electron microscopy (SEM). Electrical and dielectrical properties such as DC resistance, dielectric strength, dielectric loss, and relative permittivity were determined according to the standards.

The microstructures of sprayed and sintered alumina show significant differences with respect to grain form and porosity. The density of the bulk ceramic is lower than the density of the sprayed layer due to the coarse particle size ($d_{50} = 33$ µm). Therefore, data from dense samples of the same chemical composition but lower particle size alumina powder were used for comparison.





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

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Potassium-sodium-niobate piezoceramics $(K_{1-x-z}Na_xLi_z)Nb_{1-y}Ta_yO_3$ (KNNLT) represent a promising, environment-friendly substitute for lead-zirconate-titanate $Pb(Zr_{1-x}Ti_x)O_3$. For multilayer actuator fabrication the use of inexpensive base metal alloys (nickel or copper) is desired. This requires firing in reducing atmosphere. For the fabrication of defect-free, high-performance piezoelectric components, a thorough understanding of the complex interplay between piezoceramic oxide and metal layers during debindering, sintering and reoxidation is necessary. Sintering protocols which avoid oxidation of the metal electrodes during sintering at low oxygen partial pressure and to tailor the oxygen vacancy concentration in the piezoceramic upon reoxidation have to be developed. The KNNLT piezoceramic was successfully cofired with nickel electrodes. A KNNLT multilayer actuator was obtained after debindering the multilayer in air at 350°C, firing under low partial pressure of pO2 = 10-10 atm at 1000°C, and subsequent reoxidation at pO2 = 10-6 atm at 850°C. Manganese dopant segregation at the interface between the ceramic layer and the nickel electrode was observed. The obtained multilayer shows a normalized strain coefficient of , an effective coupling factor of keff = 0.226 and a total strain of S = 0.64 ‰ at 3 kV/mm. However, further development is required to optimize the performance of KNNLT multilayer actuators with nickel electrodes.

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Synthesis of sodium ion-conducting glass ceramics (NaRSiO) for use as a solid electrolyte in sodium solid-state batteries

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New sodium-based battery concepts represent an alternative to lithium-based batteries due to the good availability of sodium, lower costs and better environmental compatibility of suitable active materials. Some of these sodium batteries require dense, solid electrolytes, which act as ion-conducting separators between anode and cathode. The aim of this Master thesis is the sintering of sodium rare earth silicates NaRSiO with R= Y, Sm, Gd. These are synthesized as glass frits in the system Na_2O?Si_2O?R_2O_3 with the molar ratio 35.7:57.1:7.2. It was easily possible to synthesize corresponding NaRSiO?powders from the basic composition by substituting different rare earth elements. These powders could be pressed into compacts, which became dense samples after sintering using tailored sintering profiles. The microstructures have been characterized by SEM/EDX and the phase composition was determined by XRD. High fractions of the conductive phase $Na_5RSi_4O_{12}$ (N5-type phase) has been achieved in the sintered samples, as well as the minor phases $Na_{16,5}R_{2.5}Si_{12}O_{36}$ and $Na_3RSi_2O_7$ could be determined. A 2-stage sintering stage for the corresponding amorphous samples led to dense specimens. Likewise, pre-crystallised powders resulted in dense sintered bodies in a 1-stage sintering step. Ionic conductivities have been analysed by impedance spectroscopy at different temperatures to deduce activation energies. Ionic conductivities at 30°C for the grain phase of 2.2?10^{-3} S/cm for the Y-doped and 7.2?10^{-4} S/cm for the Sm-doped materials have been measured while the total conductivities were about one order of magnitude lower. These values are similar to those of sodium ion-conducting solid electrolytes such as NASICON and ??alumina.

Development and additive manufacturing of a ceramic bone implant using multi material jetting

GOTTLIEB, Lisa Katharina

Human bone has a unique structure and special mechanical properties. Tissue defects resulting from accidents, tumors or other diseases and their replacement represent a medical problem that has not yet been satisfactorily solved. There is still a great need for research, both in finding the right material and in the choice of the manufacturing process. Additive manufacturing (AM) is becoming increasingly important in implantology. The aim of this work was to implement a more suitable biomimetic material that can be used as a bone substitute and to benefit from the advantages of additive manufacturing by producing a demonstrator using the Multi Material Jetting process developed at Fraunhofer IKTS. EvoCera, the material used, is biocompatible and combines the properties of the latest high-performance ceramics with the ductility of steel. It was systematically investigated and adapted from the smallest volume unit that can be dispensed by MMJ, the droplet, to a complex component. Hybrid structures with a porous interior and a compact well-defined outer shell were produced by combining the two manufacturing processes Freeze Foaming and Multi Material Jetting. Finally, the AM process was run through from the detection of a bone tumor in the Magnetic Resonance Imaging, through the three-dimensional reconstruction of the affected area, to the additive manufacturing of a personalized implant structure.

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Performance evaluation of high-performance symmetric electrode $La_xSr_{1-x}(TiFeNi)yO_{3-?}$ as both cathode

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In comparison to other power generating devices/energy conversion system, solid oxide fuel cell(SOFC) has gained much popularity because of their remarkable efficiency, reliability, and low pollution. This study aimed to synthesize and evaluate a novel electrode material, which reduces the cost and the operating temperature of SOFCs. The purpose was achieved successfully, and the Modified pechini method was adopted to synthesize $La_xSr_{1-x}(TiFeNi)_yO_{3-?}$ perovskites and utilized it as an anode and cathode for intermediate-temperature symmetrical solid oxide fuel cell (IT-SSOFC). The XRD patterns for LSTFN and L_xS_{1-x}TFN revealed better reversibility with the cubic perovskite phase in dry H_2 and air atmosphere. The conductivity of LSTFN was increased up to 1.1 Scm^{-1} at a temperature of 700 ^oC but was further decreased with the increase in temperature due to the small polaron mechanism. However, at the same temperature (700 ^oC) in the presence of air, conductivity was increased up to 318 Scm^{-1}, fulfilling the requirement of utilizing the material as anode and cathode. Low polarization resistance (Rp) of 0.047 and 0.201 $?cm^2$ was exhibited by LSTFN in the presence of dry H_2 and air respectively at a temperature of 750 ^oC. While L_xS_{1-x}TFN exhibits much lower polarization resistance (Rp) than LSTFN at 750 °C. LSTFN-01, 02 and 03 exhibits a Rp value of 0.069 $?cm^2$, 0.043 $?cm^2$ 0.046 $?cm^2$ and in air, whereas 0.21 $?cm^2$, 0.15 $?cm^2$, and 0.16 $?cm^2$ in dry H_2 respectively. The fabricated cell L_xS_{1-x}TFN/LDC|LSGM|LDC/L_xS_{1-x}TFN maintained good stability and no measurable degradation for ~500 hrs in air and ~100 hrs in dry H_2.

Investigation on the sintering trajectory and monte carlo simulation of 3y-tzp using field-assisted sintering technology (fast)

HUANG, Jiaqi; LIU, Chao; DENG, Yuanbin; KALETSCH, Anke; BROECKMANN, Christoph

3mol%-yttria-stabilized zirconia (3Y-TZP) is one of the most valuable ceramic materials due to its outstanding chemical and mechanical characteristics. In this study, a series of sintering experiments were conducted using Field-assisted Sintering Technology (FAST) to investigate the sintering mechanisms of 3Y-TZP. By monitoring the change of relative density and measuring the average grain size at different soak time and temperatures, the 'relative density-grain size' trajectory of 3Y-TZP was obtained. Based on this trajectory, the stress exponent, grain size exponent and activation energy in the kinetic equation of high temperature creep were computed. In addition, the underlying mechanisms throughout the sintering process were identified: grain boundary sliding accompanied by diffusional creep is the dominant mechanism at low-temperature/ low-relative density region; interface reaction takes over at the medium phase while dislocation climb is responsible at high-temperature/ high-relative density region. Grain growth was found to be the result of a combination of lattice diffusion, vapor transport and solute drag. Furthermore, a numerical approach of two-dimensional Monte Carlo method was adopted to simulate the microstructural evolution and validated against the experimental results. The simulation showed high consistency with the experimental data at low-temperature/ low-relative density region while a deviation was inspected at later sintering stage.

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Contact damage tolerance of alumina-based layered ceramics with tailored microstructures

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Surface contact damage is a major cause for rejection of ceramic components. This work demonstrates strategies to enhance contact damage tolerance on the example of alumina-based ceramics. A bioinspired strategy involves texturing the microstructure to deflect cracks along the elongated grains, resembling the fracture of nacre. Further improvement is achieved by embedding such textured layers in a multilayer architecture to induce compressive residual stresses, acting as a "protective shield" against crack propagation. Contact damage resistance of such layered alumina system was investigated under Hertzian contact loading and compared to monolithic equiaxed and textured materials. Acoustic emission detection was used for "in-situ" monitoring of the materials response. It was found that a textured microstructure can accommodate the damage below the surface by shear-driven, quasi-plastic deformation instead of the classical Hertzian cone cracking observed in equiaxed alumina. In the multilayer system, a combination of both mechanisms, namely cone cracking on the equiaxed surface layer and quasi-plastic deformation within the embedded textured layer, was identified. Owing to the combined action of textured microstructure and compressive residual stresses, the propagation of cone cracks was hindered even at higher applied loads. These findings demonstrate the potential of embedding textured layers as a strategy to enhance contact damage tolerance in alumina ceramics.

Inducing photoelectric effects with dislocations in oxide ceramic single crystals

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Dislocations in ceramics can change the material's mechanical and functional properties and therefore are emerging as a new research field. Previous studies elucidated that dislocations enhance properties like ionic and electronic conductivity. But how do dislocations change the conductivity under illumination? Here, we tackle this question by fabricating ordered dislocation structures in strontium titanate single crystals and investigating their effect on the photoconductivity.

Using a recently established deformation procedure, single crystalline strontium titanate was uniaxially compressed to reproducibly induce well-ordered dislocation structures. Their photoelectric properties were characterized via a conductive atomic force microscope as well as with patterned microcontacts in a cryostat. Owing to the sample surface preparation including selective etching, it was visually proved that the out-of-plane photoconductivity around dislocations is significantly increased. This increase reached up to orders of magnitude when measuring a collective behavior by using microcontacts. Thereby, interesting side effects such as a resistive switching behavior under illumination were observed. The overall photocurrent was found to be strongly dependent on many parameters which are complexly interrelated. While dislocations are often seen as detrimental one-dimensional line defects, we demonstrate that ordered dislocations in strontium titanate instead can massively increase the photoconductivity. This brings into discussion the significance of dislocations for photoelectric processes in oxide ceramics.

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Synthesis and characterization of $In_2O_3(ZnO)_m$ / graphene oxide composites (m = 4, 5 and 7) for high temperature thermoelectric applications

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Currently, the search for new environmentally friendly and efficient energy sources is of great interest. Thermoelectric materials offer an interesting possibility by allowing us to harvest waste heat and convert it into electricity. A promising thermoelectric candidate is indium zinc oxide (IZO), which features a superlattice structure and offers a homologous series with different amounts of zinc oxide.
In this work, the synthesis of $In_2O_3(ZnO)_m$ with m = 4, 5 and 7 and up to 3 wt% of reduced graphene oxide (rGO) was conducted with the assistance of spark plasma sintering (SPS). Herein, the formation of metallic indium was observed, a result of the carbon presence during sintering. The influence of rGO on the composites was analysed by varying sintering conditions and characterized with X-ray diffraction, X-ray photoelectron spectroscopy and thermogravimetric analysis. The formation of metallic indium in the specimens appeared above 700 °C during SPS and led to the partial decomposition of the homologous compound. The presence of metallic indium and zinc oxide inside the IZO / rGO composites altered the thermoelectric properties interestingly. The existence of rGO and metallic indium vastly improved the electrical conductivity at the cost of the overall Seebeck coefficient. Combined with the rise in thermal conductivity, $In_2O_3(ZnO)_4$ exhibited 0.04 at 500 °C, a higher ZT than the IZO / rGO composites.

Sintering without shrinkage – the strange case of pure tin oxide ceramics

SIMONOVA, Petra; GREGOROVA, Eva

Tin oxide (SnO_2) in pure form is an unusual ceramic raw material, because non-densifying mechanisms (evaporation-condensation and surface diffusion) are dominating during sintering. Since essentially no shrinkage occurs after sintering, also the bulk density and porosity remain essentially the same. At the same time, however, the grain size and pore size increase and the geometry and topology of the pore space changes. This fact makes pure tin oxide ceramics an exceptional model material that allows one to study property changes due to other microstructural parameters than porosity.

The samples were prepared by uniaxial pressing (50 MPa) from commercial SnO_2 powder and sintered in the temperature range 500-1400 °C with different dwell times (0.5 to 6 hours), resulting in samples with porosity 51.6 ± 0.7 %. The ceramics were characterized by the Archimedes technique (bulk density 3.385 ± 0.046 g/cm^3). X-ray powder diffraction was used to check phase purity and estimate crystallite size (via line broadening). SEM micrographs of sintered samples were evaluated using image analysis (grain sizes with median values 155 nm-4.8 ?m). Mercury porosimetry was used to obtain the pore throat size distributions (median values 70 nm-3.5 ?m). Young's modulus was measured via high-temperature impulse excitation, resulting in relative Young's modulus increases of 30, 70 and 120 % after sintering at 1000, 1200 and 1400 °C, although the porosity of all samples is the same.

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