Glass and Ceramics
Preface

In 2013 research at the Institute of Glass and Ceramics (WW 3) centred on fundamental aspects of processing of ceramics and ceramic composites. Members of WW 3 are actively engaged in a number of cooperative research initiatives including the Cluster of Excellence on Engineering of Advanced Materials, the Central Institute for Advanced Materials (Fuerth) and the Emerging Fields Initiative (Biomaterials). New research activities localized in the Energy Campus Nuremberg started on October 2013. With a major focus on solar thermal receiver materials nanoscale surface modification for optimisation of optical and thermal properties will be investigated. Furthermore, cooperation work with the Technische Hochschule Nuernberg was established on developing a novel concept for high temperature heat storage of renewable energy.

Fortunately, we could celebrate the inauguration of the European Liaison Office of the Nagoya Institute of Technology (NITech) at our Technische Fakultaet in July. Supported by WW 3 the office will coordinate and extend the scientific cooperations of NITech not only with our university but with other European academic networks in the field of materials science as well as other engineering disciplines.

Members of the institute organized conferences, workshops, advanced training courses, two summer schools in Limoges and Copenhagen, and a German-Japanese seminar on Advanced Ceramics. On the Lange Nacht der Wissenschaften a large number of guests enjoyed the presentations of our research results and glass blowing.

After one year vacation the glass group was taken over by our new colleague Professor Dominique de Ligny by December 1st. His work will focus on structure-property relations and optical properties of silicate glasses. Professor Greil was elected Vice Dean of the Technische Fakultaet for the period 2013 – 2015. Professor Roosen was appointed Adjunct Professor by the Technical University of Denmark for the period 2014 – 2019.

Peter Greil
Andreas Roosen
Dominique de Ligny
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1. **Institute of Glass and Ceramics**

Staff

**Faculties**

Prof. Dr. Peter Greil  
Head of Institute

Prof. Dr. Dominique de Ligny  
Glass

Prof. Dr. Andreas Roosen  
Functional Ceramics

PD Dr. Nahum Travitzky  
Ceramics Processing

**Administration**

Karin Bichler  
Candice Iwai

Ursula Klarmann\(^1\)  
Evelyne Penert-Müller

**Senior Research Staff**

Dr.-Ing. Ulrike Deisinger  
Ceramic Multilayer Processing

Dr.-Ing. Tobias Fey  
Cellular Ceramics and Simulation

**Research Staff**

**Advanced Engineering Ceramics and Rapid Prototyping**

M. Sc. Alexander Bonet  
M. Sc. Benjamin Dermeik

M. Sc. Ina Filbert-Demut  
Dipl.-Ing. Lorenz Schlier

Dipl.-Ing. (FH) Tobias Schlordt

\(^1\) retired
**Functional Ceramics**

Dipl.-Ing. Michael Beck  
M. Sc. Zongwen Fu  
M. Eng. Michael Hambuch  
M. Sc. Ruth Hammerbacher  
Dipl.-Ing. Daniel Jakobsen  
Dipl.-Ing. Alfons Stiegelschmitt  
M. Sc. Moritz Wegener

**Kosseleck Group**

Dr. Guo Ping Bei  
Dipl.-Ing. Kathrin Ebert\(^2\)  
Dr.-Ing. Joana Pedimonte  
Dr. Sergey Sirotkin\(^3\)

**Cellular Ceramics and Simulation**

Dr. rer. nat. Andrea Dakkouri-Baldauf  
M. Sc. Franziska Eichhorn  
PhD Guifang Han  
Dr. rer. nat. Sreejith Krishnan\(^4\)  
M. Sc. Bastian Weisenseel  
Dipl.-Ing. Bodo Zierath

**Technical Staff**

Sabine Brungs  
Evelyn Gruber  
Dipl.-Ing. Helmut Hädrich\(^5\)  
Beate Müller  
Timotheus Barreto-Nunes  
Heike Reinfelder  
Peter Reinhardt  
Alena Schenkel-Rybar  
Eva Springer  
Dipl.-Ing. Alfons Stiegelschmitt  
Hana Strelec  
Andreas Thomsen

\(^2\) now with industry  
\(^3\) now Université de Bordeaux  
\(^4\) now Vikram Sarabhai Space Centre, Thiruvananthapuram, India  
\(^5\) retired
Prof. Dr. Dominique de Ligny was appointed professor of the Glass research group at the Institute of Glass and Ceramics in December 2013.

From 2005 to 2013 he was “Maitre de Conférences” at the University of Lyon 1. As a member of the “SOPRANO team” at the “Institut Lumièrè Matière” his work was dealing with glass under extreme conditions. His work includes the development of Raman spectroscopy at high temperatures and its application to investigate the fictive temperatures of glasses as well as demixing or crystallization, respectively. Brillouin spectroscopy at high pressures was used to investigate the mechanical properties of aluminosilicate glasses. From this structural approach, many applications in various fields such as nuclear waste disposal, writing on glasses with femtolasers, strengthening of glasses, nonlinear optic or determination of natural lava flow properties etc., can be derived.

His interest on the research of glass materials started during his PhD work at the “Institut de Physique du Globe de Paris” under the supervision of P. Richet, where he focused on the characterization of the thermal properties of magma. In Alexandra Navrotsky’s group at the Princeton University and at the University of California, Davis, he studied the thermodynamic properties of clay minerals. From 1998 to 2003 he was environmental and quality manager in industry.

At the Chair of Glass and Ceramics he will continue his studies on glasses and glass ceramics focusing on the structure-property relations and optical properties of silicate glasses. The aim is to develop a specific interest for structural relaxation close to the glass transition temperature ($T_g$) as well as for the early stage of crystallization. Taking advantage of the scientific environment of FAU and Erlangen, he will certainly develop new collaborations in between a short time period.
Retirements

Ms. Ursula Klarmann retired on 31.03.2013. She joined the institute in July 1973 as a technical staff member. In the first period her work centred on organisation including preparation of charts, care of students thesis archive and publication list of the institute. Furthermore, she dealt with graphical evaluation of research results for preparation of publications. Subsequently, Ms. Klarmann assumed responsibility for the financial management of research contracts. She carried out budget controlling with great competence and responsibility in close cooperation with the university administration as well as a number of funding organisations including DFG, BMBF, AiF, EU and industry partners.

Ms. Klarmann was strongly engaged in planning and realisation of public events of the institute including the annual glass and ceramic weeks. Furthermore, she took care of the presentation of the institutes research work to the public by posters and show cases. Due to her pronounced cooperativeness and responsibility Ms. Klarmann made a continuous and appreciated contribution to the growth and life of the institute. All the staff members deeply acknowledge her for her great work. We deliver our best wishes to Ms. Klarmann and her family for the time of retirement.
Mr. Helmut Haedrich retired on 31.10.2013 after 42.5 years in public service. Working as an electronic engineer in the institute he was in charge of electrical and electronic equipment including repair as well as development of novel scientific instruments. Among his numerous contributions are the design and manufacturing of non destructive inspection and testing devices including ultrasonic and microwave technologies, furnace control and operation devices as well as thermal analyses equipment. His work was carried out in close cooperation with the electronic workshop of the faculty and the department of electronics, institute of high frequency technology. Furthermore, he was in charge of the institutes computer network, soft- and hardware management and communication technologies.

Due to his outstanding experimental capabilities combined with creative and original ideas Mr. Haedrich soon became one of the staff members contributing a lot to the successful development and prosperity of our institute. Numerous students appreciated his great competence and help for solving technical and scientific problems in their thesis works. Furthermore, he served as first aid assistant. He always represented the institute with great enthusiasm and all the members of the institute acknowledged him for his great work. We deeply wish him and his family all the best for the time of retirement.
Graduates

Bachelor Thesis

Martin Ellinger
Influence of cold low pressure lamination parameters of ceramic green sheets on the quality of the sintered structures

Sebastian Hagen
Manufacturing of ceramic multilayer structures with internal cavities

Nils Hock
Paper-derived MAX-phase ceramic sheets

Felix Keppner
Rheological studies of a colloidal alumina paste

Thomas Koch
Cellular Al$_2$O$_3$-bioceramic

Christian Leniger
Influence of the binder system on the strength of SiC ceramics produced by 3D printing

Sofia Loginkin
Manufacturing and characterization of polymer-derived ceramic springs

Daniel Schumacher
Manufacturing of partially stabilized ZrO$_2$ substrates via tape casting

Marc-André Vogelgesang
Influence of additives on the properties of fiber-reinforced paper-derived ceramics
Larissa Wahl
Manufacturing of SiC-containing composites by electrophoretic deposition

Michel Woy
Relationship between structure and property of sulfophosphate glasses

**Master Thesis**

Franziska Eichhorn
Porous piezoelectric ceramics

Ruth Hammerbacher
Manufacturing of rotation-symmetric devices based on tape cast refractory oxides

Helene Sachsenweger
Development and application of a piezoceramic actuator for bone cell stimulation

Bastian Weisenseel
Ceramic Loop-Heat Pipes with microporous SiC-Wick
Ph.D. Thesis

Birgit Joana Pedimonte

Nanoporous alumina as a functional coating on biomaterials

Sindy Fuhrmann

Pressure dependence of the topological heterogeneity of glasses
Ning Da

Pressure-assisted filling of low-melting glasses into microcapillaries

Dr. Ning Da after successful Ph.D. examination
Visiting Students and Scientists

**Dr. Marek Potoczek** (January 2013 – February 2013)
Rzeszow University of Technology, Faculty of Chemistry, Rzeszow, Poland

**Gildas Rigondaud** (June 2013 – July 2013)
National Graduate School of Chemistry and Chemical Engineering of Montpellier, France

**Rachida El Ouardi** (June 2013 – September 2013)
Ecole Nationale Supérieure d'Ingénieurs de Limoges (ENSIL), Limoges, France

**Minato Kato** (September 2013 – November 2013)
Nagoya Institute of Technology, Ceramic Research, Nagoya, Japan

**Teppei Yamazaki** (November 2013 – January 2014)
Nagoya Institute of Technology, Ceramic Research, Nagoya, Japan
Visit of a Japanese delegation of the Nagoya Institute of Technology (NITech), Nagoya, Japan (March 2013):

Sightseeing in Würzburg then with wine tasting in Escherndorf with the Franconian wine queen
Teaching

The Department of Materials Science and Engineering offers Bachelor and Master programmes in Materials Science and Engineering and in Nanotechnology.

The Bachelor course is a three years programme (six semesters) which qualifies for the Master programme (four semesters). The curriculum consists of the "Grundstudium" (basic studies) during the first 2 years, devoted to the fundamental scientific education. It introduces the student very early into materials science and engineering concepts by offering courses on materials structures, properties, thermodynamics, kinetics, chemistry, processing, product manufacturing, analysis and testing as well as practical training. Examinations follow immediately after each semester.

The subsequent advanced programme in the 5th and 6th semester broadly deepens the entire field of materials science and engineering. Courses on economics, management and other soft skills are obligatory. This period ends with a Bachelor Thesis of nine weeks duration. Additionally, the student has to perform an industrial internship of 12 weeks.

The Master programme in the 7th, 8th and 9th semester specializes in a selected "Kernfach" (core discipline), including corresponding practical courses, seminars and courses in materials computational simulation. In addition the students select a “Nebenfach” (minor subject) from the Department of Materials Science and a “Wahlfach” (elective subject) from other Departments of the University, which offers the possibility of specialization. Finally, the programme is completed by a Master Thesis of six months.

In addition to this Materials Science and Engineering programme, the Institute of Glass and Ceramics is involved in the Bachelor and Master programmes “Energy Technology”, “Medical Technology” and the Elite course “Advanced Materials and Processes”.

Courses offered by the faculties of the Glass and Ceramics Institute

1. Semester

• Introduction to Inorganic Non-metallic Materials, P. Greil

4. Semester

• Materials Characterization and Testing, A. Roosen

5. Semester

• Processing and Applications of Glasses, P. Greil
• Processing and Applications of Ceramics, A. Roosen
• Nanocomposites, T. Fey
• Instrumental analytics, U. Deisinger

7. and 8. Semester

• Ceramic Materials in Medicine, P. Greil
• Computational Calculation of Crack Probabilities, T. Fey
• Electroceramics I + II, A. Roosen
• Engineering Ceramics, P. Greil
• Innovative Processing Techniques for Advanced Ceramic Materials, N. Travitzky
• Non-destructive Testing, T. Fey
• Physics and Chemistry of Glasses and Ceramics: I. Thermodynamics of Condensed Systems, P. Greil
• Powder Synthesis and Processing, U. Deisinger
• Rapid Prototyping, N. Travitzky
• Silicate Ceramics: From Natural Raw Materials to Modern Applications, N. Travitzky
• Stresses and Mechanical Strength, T. Fey
• Practical Course Mechanical Testing, T. Fey
• Composite Practical Course, T. Fey
Laboratories

Technical hall (600 m²): equipped with facilities for advanced processing, shaping, melting and sintering as well as molding of glass, ceramics and composites

Main Equipment

Laboratories

- Biomaterials laboratory
- Ceramography workshop
- Functional ceramics laboratory
- Glass laboratory
- Mechanical testing laboratory
- Multilayer processing laboratory
- Polymer processing laboratory

- Powder characterization laboratory
- Processing workshop
- Rapid Prototyping laboratory
- SEM/AFM laboratory
- Simulation laboratory
- X-ray characterization laboratory
Equipment

Thermal analysis
- 3-dimensional optical dilatometer
- Push rod dilatometers (up to 1800 °C)
- Thermal analysis (DTA/TGA/DSC/TMA)
- Thermal conductivity device
- Viscometry (beam bending)

Powder characterization
- ESA acoustophoretic analyser (Zeta-meter)
- Dynamic light scattering particle size analyser
- Gas absorption analyser (BET)
- Laser scattering particle size analyser
- X-ray diffractometers (high-temperature)

Optical analysis
- FT-IR spectrometer
- High-resolution fluorescence spectrometer
- Light Microscopes (digital, polarization, in-situ hot stage)
- Scanning electron microscope (variable pressure, ESEM and high temperature with EDX)
- UV-VIS-NIR spectrometers

Mechanical testing
- High precision mechanical testing with optical tracking system (EXAKT)
- Impulse Excitation Measurement (buzz-o-sonic)
- Micro hardness tester
- Servo hydraulic mechanical testing systems (also high temperature)
- Single fibre tensile testing machine
- Viscosimeter and elevated-temperature viscosimeter

Chemical analysis
- High-pressure liquid chromatograph
- ICP-OES (Spectro Analytical Instruments)
**Structural analysis**
- 2D laser scanning microscope (UBM)
- 3D Laser scanner
- Atomic force microscope (AFM)
- Electron paramagnetic resonance spectroscopy
- He-pycnometer
- High accuracy weighing scales
- Laser-Flash LFA 457
- Mercury porosimeter
- Micro-CT Sky scan 1172
- Microwave and ultrasonic devices for non-destructive testing
- Raman-microscope with two excitation lasers

**Powder and slurry processing**
- Attrition mills
- Agitator bead mill
- Disc mill
- Intensive mixers (Eirich, powder and inert gas/slurry)
- Jaw crusher
- Overhead mixer
- Pick and Placer
- Planetary ball mills
- Planetary centrifugal mixer (Thinky)
- Rotary evaporators
- Sieve shakers
- Single ball mill
- Thermo kneader
- Three-roll mill
- Tumbling mixers
- Ultrasonic homogenizer
**Shaping**
- 3D printers
- Advanced screen printing device
- Calender
- CNC High speed milling machine
- Cold isostatic press
- Electrospinning machine
- Flaring cup wheel grinding machine
- Fused deposition modelling device (FDM)
- High precision cutting device
- Hot cutting device
- Laminated object manufacturing devices (LOM)
- Lamination presses
- Langmuir–Blodgett trough
- Lapping and polishing machines
- Low-pressure injection moulding machine
- Precision diamond saws
- PVD coaters
- Robot-controlled device
- Roller coater
- Screen printer
- Sheet former
- Spin coater
- Tape caster
- Textile weaving machine
- Twin screw extruder
- Ultrasonic drill
- Vacuum infiltration device

**Heat treatment**
- Autoclave
- Dryers
- Furnaces (air, N₂, Ar, Vac, High-Vac, forming gas) up to 2500°C for sintering, glass melting, infiltration, debinding, pyrolysis
- Gradient furnace
- High-temperature spray furnace
2. **RESEARCH**

**Survey**

Research centres on basic aspects of ceramics, glasses and composites. Materials for applications in microelectronics, optics, energy, automotive, environmental, chemical technologies and medicine were investigated. Research was carried out in close cooperation with partners from national and international universities and industries.

<table>
<thead>
<tr>
<th>Research Projects (in alphabetical order)</th>
<th>Funding</th>
<th>Principal Investigator</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bioactive ceramic cages</td>
<td>IN</td>
<td>Prof. Greil / Dr. Fey</td>
</tr>
<tr>
<td>Development of layered structures and 3D generative processing methods for innovative combustion chamber lining concepts</td>
<td>BMWi + IN</td>
<td>Prof. Roosen / PD Dr. Travitzky</td>
</tr>
<tr>
<td>Cellular ceramics for heat absorbers</td>
<td>EnCN</td>
<td>Prof. Greil</td>
</tr>
<tr>
<td>Deformation and sintering behaviour of preceramic papers</td>
<td>DFG</td>
<td>PD Dr. Travitzky</td>
</tr>
<tr>
<td>Experimental study and simulation of anisotropic effects in cast green tapes</td>
<td>DFG</td>
<td>Prof. Roosen</td>
</tr>
<tr>
<td>Flexible manufacturing of preceramic paper based refractory components</td>
<td>DFG</td>
<td>Prof. Greil</td>
</tr>
<tr>
<td>Hierarchical cellular ceramics and composites</td>
<td>DFG</td>
<td>Prof. Greil</td>
</tr>
<tr>
<td>High temperature stable ignition components based on defined 2D and 3D SiSiC structures</td>
<td>AiF</td>
<td>PD Dr. Travitzky</td>
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<tr>
<td>Lightweight cellular ceramics</td>
<td>EC</td>
<td>Prof. Greil</td>
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<tr>
<td>Project Description</td>
<td>Funding Body</td>
<td>PI</td>
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<td>-------------------------------------------------------------------------------------</td>
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<tr>
<td>Manufacturing of multilayer refractories by tape casting</td>
<td>DFG</td>
<td>Prof. Roosen</td>
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<tr>
<td>Manufacturing of transparent ceramic substrates</td>
<td>BMBF</td>
<td>Prof. Roosen</td>
</tr>
<tr>
<td>Stable and metastable multiphase systems for high application temperatures</td>
<td>DFG</td>
<td>Prof. Greil</td>
</tr>
<tr>
<td>Dispers systems for electronic devices</td>
<td>DFG</td>
<td>Prof. Roosen</td>
</tr>
<tr>
<td>Polymer derived ceramics for bearing applications</td>
<td>IN</td>
<td>PD Dr. Travitzky</td>
</tr>
<tr>
<td>Robocasting of macrocellular ceramic 3D-lattice structures with hollow filaments</td>
<td>DFG</td>
<td>PD Dr. Travitzky</td>
</tr>
<tr>
<td>Self healing MAX phase ceramics</td>
<td>DFG</td>
<td>Prof. Greil</td>
</tr>
<tr>
<td>Structured carbon based catalyst support structures for CO hydration</td>
<td>DFG</td>
<td>Dr. Fey</td>
</tr>
<tr>
<td>Tape on Ceramic Technology</td>
<td>BMBF</td>
<td>Prof. Roosen</td>
</tr>
</tbody>
</table>

**Funding organisations:**

- AiF: Industrial research Cooperation
- BMBF: Federal Ministry of Education and Research
- BMWi: Federal Ministry of Economics and Technology
- DFG: German Research Foundation
- EC: Cluster of Excellence (“Engineering of Advanced Materials”)
- EnCN: Energy Campus Nuremberg
- IN: Industry
Selected Research Highlights

Crack healing in MAX phase ceramic composites

Joana Pedimonte, Guoping Bei, Tobias Fey, Peter Greil

Engineering ceramics being able to repair cracks upon heat treatment have gained increasing attention. Recovery of mechanical properties depleted by overloading, slow crack growth, or thermal shock damage may offer a high potential for improving the reliability and prolongation of the lifetime of ceramic components subjected to mechanical loading at elevated temperatures. Crack healing behavior of Al₂O₃ was observed at temperatures exceeding 1400 °C where sintering phenomena driven by reduction of surface energy may trigger perturbation and closure of pores and cracks.

Crystal structure of 312 MAX phase, left and 211 MAX phase, right.

Significantly lower healing temperatures < 1000 °C may be achieved by loading Al₂O₃ with repair filler particles which undergo oxidation in near surface cracks thereby filling the crack space with oxidation products. MAX phase particles, with the general formula Mₙ₊₁AXₓₙ (n = 1 to 3) with M = Ti, V, ..., A = Al, Sn, ..., X = C, N, may serve as a repair filler providing crack healing capability when dispersed in a ceramic matrix composite. Due to the nanolayered nature of the structure, the crack healing mechanisms are based on oxidation of the A- and M-elements. Cracks with up to 

SEM micrograph of an indent crack on the surface of Ti₂SnC loaded (20 vol. %) alumina matrix composite a) prior and b) after annealing in air for 3 h at 900 °C and c) Ti-mapping.
approximately 200 µm length can be completely filled with Ti$_2$Al$_{(1-x)}$Sn$_x$C ($x = 0$) MAX phase oxidation products after heat treatment at $T = 900$ °C (3 h).

A full recovery of the mechanical strength was achieved in composites with MAX phase repair filler loading from 5 – 20 vol.%. For example after annealing for 3 h, composites loaded with 20 vol.% Ti$_2$AlC show full recovery at a healing temperature of approximately 900 °C whereas a similar behavior can be observed at lower temperatures of 700 °C for specimens loaded with 10 and 20 vol. % Ti$_2$SnC. The enhanced healing response of Ti$_2$AC ($A = \text{Al, Sn}$) containing Sn instead of Al offers a high potential for providing crack healing capability to ceramic matrix composites even at moderate temperatures below 1000 °C.

*Filler loading dependence of fractional strength of alumina composite after healing for 3 h at 900 °C (Ti$_2$AlC) and at 700 °C (Ti$_2$SnC).*

G.P. Bei, B. J. Pedimonte, T. Fey, P. Greil
Oxidation Behavior of MAX Phase Ti$_2$Al$_{(1-x)}$Sn$_x$C Solid Solution; *J. Am. Ceram. Soc.* 96 (2013) 1359

B. J. Pedimonte, D. Pourjafar, G. P. Bei, T. Fey, P. Greil
Oxidative crack healing in Al$_2$O$_3$ composites loaded with Ti$_2$AC ($A = \text{Al, Sn}$) repair fillers; *J. Cer. Sci. Techn. (2014) in press*

Report 2013 – Department of Materials Science and Engineering, Glass and Ceramics, University of Erlangen-Nuremberg
Nanoporous anodic alumina as a functional coating on biomaterials

Joana Pedimonte, Tobias Fey, Peter Greil

Bioceramics applied to bone replacement and regeneration should trigger minimal inflammatory responses while stimulating osteoblast adhesion, proliferation and differentiation. It has long been known that substrate topography, including grooves, ridges, islands, nodes and pores, can affect cell response and osseointegration behavior of a bioceramic implant in contact with bone. Interaction with nanotopographies can alter cell morphology, adhesion, motility, proliferation, endocytotic activity, protein abundance and gene regulation. Among diverse cell types (fibroblasts, osteoclasts, endothelial, smooth muscle, epithelial) osteoblasts were observed to interact with nanotopographical features of substrate materials.

Coating of various biomaterials with a nanoporous anodic alumina (AAO) surface layer at different anodization voltages (20 – 60 V) with varying pore diameter (15 – 40 nm), a mean pore distance (40 – 130 nm) and a total porosity of ~ 10% that elicits a favorable response for osteoblasts might be attractive in bone replacement as well as bone tissue engineering. Nanoporous layers of alumina prepared by electrochemical oxidation are shown to achieve improved short-term cell attachment and activity compared to a plain alumina surface. In addition to topology variation, the control of nanoporous structure may trigger a local variation of electrostatic surface properties and may offer a high potential to enhance cellular interaction compared to a bioinert alumina surface. From streaming potential measurements the zeta potential and the isoelectric point (iep) were derived and correlated to the topology variation of the nanoporous AAO layers. With decreasing pore diameter a shift of iep from ~7.9 (pore diameter 40 nm) to ~6.7 (pore diameter 15 nm) was observed. Plain alumina layers, however, exhibit an iep of ~9. Compared to the plain alumina surface enhanced adhesion and activity of hFOB cells can be observed on the nanoporous AAO after 24 h
culture with a maximum at a pore size of 40 nm. The topology-induced change of the electrochemical surface state may have a strong impact on protein adsorption as well as on cell adhesion, which offers a high potential for the development of bioactive AAO coatings on various biomaterial substrates.

Isoelectric point (iep) versus pore size of the AAO layers: experimental values derived from streaming potential measurements and calculated values.

Through topological control of surface hydroxyl groups’ dissociation behavior it may also be possible to reduce the adherence of microbial entities and to provide improved control over infection and secure fixation of porous alumina-coated implants. Furthermore, the nanoporous structure offers a high potential for loading the AAO layers with bioactive materials able to stimulate or inhibit selective cellular responses. Applying these bioactive AAO coatings on versatile biomaterial substrates including ceramics as well as metals offers a high potential for developing implant materials with improved integration behavior into the living system.

FDA/PI stain of hFOB cells on AAO surface with \( d_p = 40 \text{ nm} \) as well as alumina reference.


Report 2013 – Department of Materials Science and Engineering, Glass and Ceramics, University of Erlangen-Nuremberg
Manufacture of ultrathin, particulate-based ITO layers by rotary printing

Moritz Wegener, Andreas Roosen

Transparent conductive oxides (TCOs) as indium tin oxide (ITO) or zinc oxide offer a unique combination of high electrical conductivity and high transparency. TCOs find application as transparent electrode materials in displays, touch screens, or solar cells. Conventionally, expensive vacuum-based sputtering techniques are used for the manufacturing of nanometer thin TCO layers. Sputtered TCO layers, however, are brittle and unsuitable for the use in flexible devices. Printing and coating techniques based on nanoparticular TCO materials can overcome both problems. Printed or coated films are composites of TCO particles dispersed in polymers which are manufactured at ambient atmosphere and room temperature. TCO-polymer composites offer a high mechanical flexibility.

In the past, a large number of printing and coating techniques were developed to satisfy product requirements concerning design and functionality. Rotary printing offers the possibility to print planar layers and/or structures; a high throughput based on printing velocities of several hundred meters/min can be obtained. Rotary printing techniques include, e.g., gravure printing, flat printing, relief printing, sieve printing, etc.

This research project focusses on the development of nanoparticular indium tin oxide (ITO) inks for the manufacturing of ultrathin ITO layers on flexible polymer substrates by rotary printing. Ethanol- and water-based ITO inks were prepared and the influence of different ink compositions on the printing process and the properties of the deposited film was evaluated. The transmission and the specific resistance of the printed ITO layers in dependence on the ink composition and on the printing process parameters were characterized.

The working scheme of a flexography printer, which is a relief printing technique, is shown in Figure 1; there are two rolls, a steel metering roll and a rubber-coated applicator roll. The ink is filled into the gap between these rolls. The rolls are moving in opposite direction during the coating process and the ink is applied onto the substrate by the applicator roll. The achieved wet film thickness depends on several processing parameters like the gap between the two rolls, the printing speed, the pressure on the substrate brought by the applicator roll, the viscosity of the ink, the surface tension of the ink and the substrate, etc. The amount of
powder and organic components in the ink decide which film thickness is formed on the substrate.

![Diagram of flexography printer](image)

*Figure 1: Working scheme of a flexography printer.*

In rotary printing, a very frequently arising printing artifact is the so called “ribbing”. Ribbing describes the occurrence of ribs in between the gap of two moving rolls (Figure 2). These ribs are transferred to the substrate and lead to an inhomogeneous surface topography of the deposited and dried layer. For the manufacturing of films with high layer quality, rib formation must be avoided. The appearance of ribs can be evaluated by the dimensionless capillary number $Ca$, which depends on the viscosity and on the surface tension of the ink as well as on the applied printing speed. Taking into account this $Ca$ number, the formation of ribs during the printing process could be suppressed by choosing suitable ink compositions. Thus, it was possible to manufacture highly transparent ITO films with layer thicknesses between 150 nm and 1.5 µm and high layer quality on flexible PET carrier films at printing speeds up to 3 m/min. The manufactured flexible ITO layers exhibited electrical resistivity values between 140 and 3 $\Omega \cdot$cm.

![Layer with and without ribs](image)

*Figure 2: Layer with (left) and without (right) ribs, manufactured by rotary printing.*
Influence of pore orientation on anisotropic shrinkage

Zongwen Fu, Andreas Roosen

Anisotropic shrinkage of tape-cast ceramic sheets is a major hindrance to miniaturization of multilayer structures which require high accuracy of the position of vias or circuit lines in different layers during thermal processing. It is well known that for tape-cast products the shrinkage anisotropy in three spatial directions follows $\varepsilon_z > \varepsilon_y > \varepsilon_x$, where $\varepsilon$ represents the linear sintering shrinkage and $x, y$ and $z$ denote the casting, transverse and thickness direction, respectively. The coefficient of anisotropy shrinkage, $K_{xy} = (1 - \varepsilon_x / \varepsilon_y) \times 100$, describes the shrinkage mismatch in both directions. This anisotropic shrinkage is explained by the non-uniform microstructure caused by shearing and uniaxial drying during tape casting as well as by particle rearrangement during thermal treatment. In order to establish the correlation between the anisotropic shrinkage and the textured microstructure analytically, the orientation degree of particles and pores in green tapes was investigated by means of a modified linear intercept analysis on SEM micrograph with an accuracy higher than 150 nm/pixel. The SEM micrograph was covered with a grid and the pore anisotropy factor $S$ was determined by calculating the cumulative pore space number along each grid line in both directions. The factor $S_{xy}$ is greater than zero when pores are oriented in $x$-direction. The particle anisotropy factor $R$ was determined accordingly.

Fig. 1 shows the microstructure of $xz$- cross-sections of tapes composed of spherical and platelet-shaped particles. With increasing anisotropy of the particle shape, the factors $S$, $R$ and $K$ rise simultaneously, exhibiting in most cases the same sign and a similar magnitude. Non-spherical particles and pores in tape-cast ceramics are mostly oriented perpendicular to the thickness direction which always exhibits the highest shrinkage; i.e., shrinkage perpendicular to the pore and particle orientation is always higher than in the direction parallel to the casting direction. This particle and pore orientation is caused by shearing forces during casting and constrained drying effects.

The correlation between the anisotropic shrinkage and the non-uniform microstructure was verified by mathematical modelling based on the shrinkage theory of Olevsky and Tikare. For elongated particles and pores (Fig. 2), the anisotropy shrinkage coefficient $K$ in the $xy$-plane was derived as:
\[
K_{xy} = \left(1 - \frac{\varepsilon_x}{\varepsilon_y}\right) \times 100 = \left(1 - \frac{w}{l} \cdot \frac{a^3 \cdot r_a \cdot r_b \cdot \sin \frac{B}{2} + b}{b^3 \cdot r_b \cdot r_a \cdot \sin \frac{A}{2} + a}\right) \times 100
\]

(a, b, r_a, r_b, A and B are given in Fig. 2)

Fig. 1: SEM micrographs of different alumina tapes with (a) spherical particles in \(xz\)-plane and (c) platelet-shaped particles in \(xz\)-plane

Fig. 2: Representative unit cell to calculate shrinkage anisotropy in \(x\)- and \(y\)-direction.
3. PUBLICATIONS

Papers

(in alphabetical order)

Evaluation of Angiogenesis of Bioactive Glass in the Arteriovenous Loop Model
*Tissue Engineering Part C: Methods* 19(6) (2013) 479-486
DOI:10.1089/ten.tec.2012.0572

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03/13  G. Bei, B.J. Pedimonte, T. Fey P. Greil
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Three-dimensional printing of SiSiC lattice truss structures
*Materials Science and Engineering A, 560 (2013) 851-856*
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07/13 I. Götschel, B. Gutbrod, N. Travitzky, A. Roosen, P. Greil
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*Advances in Applied Ceramics, 112 (6) (2013) 358-365*
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Dense YSZ laminates obtained by aqueous tape casting and calendering
*Advanced Engineering Materials, 15 (10) (2013) 1014-1018*
DOI: 10.1002/adem.201200362
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Journal of Advanced Research, 4 (2013) 75–82
DOI: 10.1016/j.jare.2012.01.006

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Paper-derived β-TCP
Materials Letters 98 (2013) 161-163
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Paper-derived hydroxyapatite
Ceramics International 39 (6) (2013) 7179-7183
DOI: 10.1016/j.ceramint.2013.02.062

Paper-Derived Bioactive Glass Tape.
DOI: 10.1002/adem.201200192
18/13  M. Wegener, A. Roosen, M. Gillert, F. Durst

Fabrication of functional nanoparticulate coating in the submicrometre range with the slot die process


*Annual glass week: Andreas Thomsen creates some artistic glass ware*
Evaluation of Angiogenesis of Bioactive Glass in the Arteriovenous Loop Model

Andreas Anudas, MD,1 Amelie Eitel,1 Gregor Buhrer,2 Isabel Arnold,1 Alexander Hopp,2 Rainer Detloch, PhD,2 Philipp Newby, MPhys,2 Tobias Fey, PhD,2 Peter Greil, PhD,2 Raymund E. Hensch, MD,1 Aldo R. Boccaccini, PhD,3 and Ulrich Kremer, MD.

In this study, the angiogenic effect of sintered 45S5 Bioglass® was quantitatively assessed for the first time in the arteriovenous loop (AVL) model. An AVL was created by interposition of a venous graft from the cranial side between the femoral artery and vein in the medial thigh of eight rats. The loop was placed in a Teflon isolation chamber and was embedded in a sintered 45S5 Bioglass® granula matrix filled with fibrin gel. Specimens were investigated 3 weeks postoperatively by means of microcomputed tomography, histological, and morphometrical techniques. All animals tolerated the operations well. At 3 weeks, both microcomputed tomography and histology demonstrated a dense network of newly formed vessels originating from the AVL. All constructs were filled with cell-rich, highly vascularized connective tissue around the vascular axis. Analysis of vessel diameter revealed constant small vessel diameters, indicating immature new vessel sprouts. This study shows for the first time arterial vascularization of a sintered 45S5 Bioglass® granula matrix. After 3 weeks, the newly generated vascular network already interfused most parts of the scaffolds and showed signs of immaturity. The intrinsic type of vascularization allows transplantation of the entire construct using the AVL pedicle.

Introduction

The term tissue engineering was first defined by Langer and Vacanti in 1993 as "...an interdisciplinary field that applies the principles of engineering and the life sciences toward the development of biological substitutes that restore, maintain, or improve tissue function." Since then, different strategies and technologies to grow and repair tissues have been developed. Most of these strategies are based on the use of different biomaterials or scaffolds, alone or in combination with specific cells and/or growth factors. In the field of bone tissue engineering, a multitude of different matrices have been described and evaluated. 45S5 Bioglass®, a silica-based multi-derived glass, of the composition (w%) 45% SiO₂, 24.5% Na₂O, 24.5% CaO, and 6% P₂O₅, is one of the most promising biomaterials that combines biodegradability and bioactivity. Bioactive glass was first introduced in 1971 by Hensch et al.,2 and they have been already used in a variety of clinical applications. Bioglass® is known to be osteoconductive and to promote osteoblast adherence, growth, and differentiation. Bioactive glass is not immunogenic and can control the secretion of cytokines in response to inflammatory stimuli. Angiogenic effects of Bioglass® have been also discussed.1 One of the core limitations for transferring most of the bone tissue engineering concepts from in vitro into in vivo environments and clinical applications is the inadequate vascularization of tissue-engineered constructs. Therefore, induction of vascularization is a crucial part of any successful tissue-engineering model. Nowadays, the majority of tissue engineering approaches are based on the so-called extrinsic vascular pathways.3 In this case, the construct is vascularized through the periphery; therefore, the implantation of specimens into a site of high vascularization potential is mandatory.3 Large-bone defects in regions with compromised vascularization require a reconstruction using vascularized bone constructs, which will be transplanted into the recipient site using microsurgical techniques of vascular anastomoses. These bone constructs rely on the so-called intrinsic vascular pathway with a defined vascular axis. An novel animal model to generate axially vascularized tissue construct was first introduced by Enal and Spira in 1975. Recently, our

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Accelerated processing route for KNN based piezoceramics

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It is known that the properties of potassium-sodium-niobate (KNN, $K_{x=0.7}Na_{x=0.3}Nb_{2}O_{5}$) are sensitive to processing and that the most successful way of stabilising and improving material performance is proper doping of KNN. However, this leads to more complex material systems, whose synthesis is very time consuming to assess by conventional processing techniques. On the other hand, known high throughput routes produce a serious interference with conventional processing, resulting in significant differences of the findings, or inaccessibility of certain parameters. In this paper, an accelerated processing route is introduced and compared with conventional mixed oxide processing regarding the density, large signal piezoelectric charge constant, permittivity, loss tangent planar coupling factor, specific resistivity and microstructure.

By means of three differently doped KNN based compositions, it is shown that the accelerated processing route yields reproducible results, which are equal or even superior to conventional techniques, while the processing time and the batch costs are significantly reduced.

Keywords: Lead-free, Piezoceramics, High-throughput processing, HTE, High-throughput experimentation, Accelerated processing, KNN

Introduction

Lead zirconate titanate (PZT) ceramics, though highly sophisticated, very effective and well understood, should be replaced in the long run due to the potential release of harmful lead in processing and disposal. In an effort to remove hazardous substances from electronic equipment, the EU and other governments placed a general ban on lead and other heavy metals from electronic materials\textsuperscript{1}. For application fields without a proper alternative, exceptions are provided on a temporary basis, which is regularly revised. The lead free material system KNN ($K_{x=0.7}Na_{x=0.3}Nb_{2}O_{5}$) shows appealing initial performance and high Curie and application temperatures, provided the densification is high and the stoichiometry is carefully controlled\textsuperscript{2}. Thus, KNN based ceramics have become the most likely successor of PZT, especially for piezoelectric applications. Their main drawbacks include difficult processing properties like the deliquescence of sodium and potassium carbonate, as well as their evaporation in the sintering step. In addition, the well pronounced thermal dependence of important parameters like the large signal piezoelectric constant $d_{\text{33}}$ or the permittivity $\varepsilon$ is an issue. To overcome these challenges, significant effort has been dedicated to improve processing\textsuperscript{3,4,12} and to reduce the thermal drift of these properties.\textsuperscript{5,13} So far, considerable progress has been made by proper doping of KNN, leading to increasingly complex ceramic compositions. To efficiently catch up with the performance levels of PZT and to assess sophisticated material systems, experimentation approaches faster than the traditional large volume mixed oxide route are required. High throughput routes\textsuperscript{14,17} for the assessment of electronic and piezoelectric materials feature a throughput of up to several hundred compositions per week. However, they deviate strongly from the mixed oxide route, which is the most likely route for future mass production of KNN based ceramic compounds. As KNN is highly sensitive to processing, results from high throughput vapour phase, sol gel or paste dispensing processing are hardly comparable with conventional mixed oxide processing. In this paper, an accelerated mixed oxide processing route is introduced picking up aspects of high throughput approaches. As KNN based model substances, the following formulations were chosen: first, KNN-LT-T1086 ($K_{x=0.7}Na_{x=0.3}Li_{0.4}Nb_{1-x}Ta_xO_{5}$), second, KNN-LT-T88, a material doped with more Li and Sb ($K_{x=0.5}Na_{x=0.5}Li_{0.4}Nb_{1-x}Ta_xSb_{0.5}O_{5}$) and third, KNN-LT-T9 ($K_{x=0.7}Na_{x=0.3}Li_{0.4}Nb_{1-x}Ta_{x}O_{5}$). Compared to conventional processing, five times more compositions can be produced and characterized per time unit. The reproducibility is high and the properties of samples from both routes are consistent.

Experimental

For powder synthesis, commercially available raw materials were used (Table 1).

All batches shown in this paper were produced from the same raw material vessels. To cope with the deliquescence of $Na_2CO_3$ and $K_2CO_3$, these raw materials...
Oxidation Behavior of MAX Phase Ti_{2}Al_{1-x}Sn_{x}C Solid Solution

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MAX-phase Ti_{2}Al_{1-x}Sn_{x}C solid solution with x = 0.32, 0.57, 0.82, and 1 was synthesized by pressureless sintering of uniaxially pressed Ti, Al, Sn, and TiC powder mixtures. Annealing in air atmosphere at 200°C–1000°C triggered a sequence of oxidation reactions which reveal a distinct influence of solid solution composition on the oxidation process. With decreasing Al/Sn ratio, the characteristic temperature of accelerating oxygen uptake of A-element was reduced from 950°C (x = 0) to 460°C (x = 1). SnO_{2} formed at temperatures significantly lower than TiC (metal) and Al_{2}O_{3}. Substitution of A-element by MAX phase solid solutions by low-melting elements such as Sn may offer potential for reducing oxidation-induced crack-healing temperatures.

I. Introduction

MAX phases form a group of nanolaminated ternary carbides and nitrides with the general formula M_{n+1}AX_{n} (n = 1 to 6), where M denotes an early transition metal, X is A-group element (from IVA to VIA), and A is either C or N. The structure of the MAX phase (tantalum-carbides) is composed of MX slabs which are separated by planar layers of the A-element and result in strongly anisotropic characteristics. As the MX bonds are weaker than the M-X bonds, the MAX phases are able to combine both merits of metal and ceramic materials, demonstrating high thermal and electrical conductivities, excellent machinability, and oxidation resistance.

Excellent oxidation stability of Ti_{2}AlC was attributed to the formation of a protective 
Al_{2}O_{3} scale on the material surface, although Ti-Al intermetallic phases such as Ti_{3}Al and TiAl do not form a protective oxide scale during high-temperature oxidation. Although less Al is present in MAX phases Ti_{2}AlC and Ti_{2}AlC, the high diffusivity of Al along the (001) basal planes is supposed to induce a thin Al-depleted layer near the oxide scale/substrate interface and facilitate selective oxidation of Al. Originated from the pronounced differences in Ti-C and Ti-A bonding, MAX phase exhibited unusual properties such as deceleration of A-elements (Sn, In, and Sb) and Sn resulting in extrusion of metallic impurities at high temperatures. Although the driving force for deceleration of the A-element from the basal (001) planes of M_{2}XM is still discussed controversially, ab initio calculation of bonding energy as well as migration energy suggest a high mobility of low-melting A-element.

MAX phases M_{2}X_{3}A with M = Ti, V, Cr and A = Al, Si, recently gained interest for their ability to heal surface cracks by oxidation reaction at temperatures exceeding 1000°C. As selective oxidation of the A-element was claimed to govern oxidation behavior of MAX phase Ti_{2}AlC, substitution of Al by a low-melting metal element would be of interest for achieving lower reaction temperatures. Indeed, oxidation of MAX phases Ti_{2}SnC containing low-melting Sn on the A-position was reported to exhibit accelerated oxidation reaction compared with Ti_{2}AlC. This study focused on the investigation of oxidation behavior of MAX phase solid solutions Ti_{2}Al_{1-x}Sn_{x}C where x varied from 0 to 1. Variation in Ti-C and Ti-A atomic distances with x were measured by Rietveld refinement of XRD and correlated with the oxidation reaction mechanism.

II. Experimental Procedure

Ti_{2}Al_{1-x}Sn_{x}C solid solution specimens were fabricated from reactant powder mixture consisting of Ti (4.5 µm, 99.94% purity), Al (8 µm, 99.99% purity), Sn (1 µm, 99.95% purity), and TiC (2 µm, 99% purity) with molar compositions corresponding to x = 1 (Ti-Sn:0.5TiC), x = 0.32 (Ti-0.5Sn:0.2Al–0.07TiC), x = 0.07 (Ti-0.5Sn:0.5Al–0.97TiC), and x = 0.02 (Ti-0.5Sn-0.6Al-0.97TiC). A reference specimen was prepared from a Ti_{2}AlC powder (Karstahl, Sandvik Materials Technology GmbH, Mörfelden-Walldorf, Germany) with D_{50} = 1 µm and a phase purity >90%.

The reactant powder mixtures were thoroughly milled for 1 h in a Turrbox mixer (WAB, Basel, Switzerland) and cylindrical specimens with a diameter of 10 mm were uniaxially pressed (50 MPa) and pressureless sintered in a vacuum furnace (Thermal Technology Inc., Santa Rosa, CA) at 1400°C for 1 h applying a heating rate of 15 K/min. Single-phase Ti_{2}SnC samples were sintered at 1200°C under same conditions.

Analyses of the crystalline phase content were conducted by X-Ray Diffraction (XRD) (Kristallograph, Siemens AG, Munster, Germany), operated with monochromatic CuKα radiation, Material Analysis Using Diffraction (MAUD) software was applied to extract the lattice parameters of the different Ti_{2}Al_{1-x}Sn_{x}C solid solutions and atomic positions of Ti in the unit cells by means of Rietveld refinement.

The weight ratio of Sn and Al was determined by Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES), Spectro Analytical Instrument, Genesis SM3, Kleva Germany to deduce the values of the Sn content in the Ti_{2}Al_{1-x}Sn_{x}C solid solutions. Before ICP-OES analysis, the powders were dissolved in HCl/HF acid to remove metallic impurity phases such as elemental Sn and intermetallic Ti-Sn compounds.

The oxidation behavior of Ti_{2}Al_{1-x}Sn_{x}C solid solution was investigated in the temperature range from 900°C up to 1200°C by exposing the specimens to ambient air atmosphere for each 1 h (Linn High Thermal GmbH, Eschelbronn, Germany). To increase the reaction surface exposed to the oxygen, the bulk specimens were crushed and milled to a...
Thermal-induced residual stresses affect the lifetime of zirconia–veneer crowns

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ABSTRACT

Objective: The purpose of this study was to investigate the effects of thermal residual stresses on the reliability and lifetime of zirconia–veneer crowns.

Methods: One hundred and twenty eight second upper premolar zirconia–veneer crowns were manufactured for testing the initial strength (n = 48) and under cyclic fatigue (n = 48). Zirconia crowns (3Y-TZP Cubics, YTA Zahnfabrik, CTE: α-Al2O3: 10.5 ppm/°C) were milled using a CeraCADLab (Sirona) machine and sintered to a final thickness of 3.7 mm. Forty-four crowns were sintered with 10% μm alumina particles (10μm, 3 μm diameter; 60° angle, 8 μMPa pressure in order to trigger a hexagonal to monoclinic transformation and to produce a rough surface. The crowns were veneered using two different porcelains (VESTA VarioFir, VITA Zahnfabrik, CTE: α-Al2O3: 10.5 ppm/°C; and Veneer Master, CMF, CTE: α-Al2O3: 10.5 ppm/°C) so to result in crowns with either high thermal mismatch (±1.4 ppm/°C with YTA) and low thermal mismatch (±0.3 ppm/°C with Veneer Master). The porcelains were applied by the same operator and fired (YTA Variofiring 4000) according to the firing schedules defined by the manufacturers to a final thickness of 1.4 mm (final crown thickness = 1.1 mm, core/veneer ratio = 0.5). After the last glaze firing the crowns were cooled following a fast (600°C/minute) or a slow (3°C/minute) cooling protocol. The glazed crowns were submitted to a sliding-motion (0.7 mm lateral movement) cyclic fatigue in a chewing simulator (3D Mechanotest) under 200 kgf (~200 N load) weight until failure (chipping) (n = 16). The other half of the crowns were subjected to compressive loading test in an universal testing machine (Instron model 4468) until failure at a cross-head speed of 0.75 mm/min (n = 16). The failure probability for initial strength and cyclic fatigue was performed using a Weibull distribution approach at a scale factor of n = 16.

Results: The compressive strength test showed a low sensitivity to detect reliability variations regarding thermal stresses created within the veneer layer of tested crowns. Cyclic fatigue, slow cooling resulted in statistically higher cycles to failure only for the crowns that presented a high thermal mismatch between core and veneer (VESTA group).

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Photoelastic Imaging of Residual Stress Distribution in Epoxy Interface Layers of Ceramics with Periodic Building-Block Structure

By Tobias Fey,* Michael Götz and Peter Greil

Ceramic composites with regular periodic assembly structures of space filling building blocks having dimensions orders of magnitude larger (10–1000 μm) than the particle size (0.1–10 μm) may offer a high potential for near net shape manufacturing as well as toughening of brittle ceramic materials. Furthermore, ceramics with a three-dimensional periodic structure such as porous lattices or photonic crystals have found increasing interest for a variety of application fields including sensors, catalytic substrates, and tissue engineering scaffolds, as well as for photonic and electromagnetic wave guidance, circuits, filters, cavities, lenses, antennas, and absorbers. Recently, we reported on ceramic-based composites with 2D and 3D periodic arrangement of space filling building blocks manufactured by a vibration assisted self-assembly technique. In contrast to monolithic ceramics the properties of composites with periodic structure depend not only on the intrinsic ceramic properties but will strongly be influenced by the symmetry of building-block arrangement and the bonding phase. Interface bonding may be achieved by a brittle ceramic or glass, a plastic metal, or a viscoelastic polymer layer.

Interface design and interface stresses play a key role regarding to the fracture behavior of multiphase ceramic composites when subjected to mechanical or thermal loading conditions. The stresses may be caused by thermal expansion mismatch between dissimilar constituents, e.g., matrix and reinforcement phase(s). As clamping stresses increase the interfacial frictional stress increases and eventually may trigger brittle cracking. Complementary when tensile stresses exceed a critical value it results in spontaneous interface debonding. In addition to interfacial sliding, residual stresses influence the conditions for crack deflection and hence may have a strong impact on toughness and work-of-fracture of brittle ceramic matrix composites. Residual interface stresses generated by thermal expansion and elastic mismatch between reinforcing particles and matrix in ceramic composites were analyzed by experimental as well as theoretical fracture mechanics approaches. A variety of sophisticated test configurations for the mechanical evaluation of interfaces in ceramic composites evolved in literature. For example, indentation push-in or push-through techniques received the greatest attention as they provide information on frictional stress acting on the interface of individual fibers. Micromechanical models that correlate global fracture behavior with local interface stress state were derived for a variety of composite materials structures including dispersed particle composites, fiber-reinforced ceramic matrix composites and laminar composites. Superposition of interface stresses and applied external loading stresses may give rise to critical conditions able to initiate energy dissipating mechanisms such as debonding, deflection, and pull-out. Overall, the micromechanical processes mentioned above have a strong impact on deformation behavior, toughness, and work-of-fracture of brittle ceramic materials.

In this work, an epoxy resin bonded alumina composite with a periodic arrangement of cuboidal alumina building blocks served as a model system for imaging residual stress distribution in the interface bonding layer. Making use of optical transparency of the polymer bonding phase, a photoelastic measurement technique was applied to image stress distribution in the interface bonding layer. Non-contact, non-destructive photoelastic measurements are widely used for stress analysis in glass products even of complex shape, in prosthetics, as well as for analysis of fracture processes. Illuminated by polarized light fringe patterns that are related to the difference in principal stresses in a plane normal to the light propagation direction (isochromatic) give information about the local stress state governed by the material properties and the building-block arrangement. Periodic structure patterns with tetragonal and monoclinic unit cell symmetry were prepared which differ in four- and three-fold interface bonding layer node topology, respectively. Stress interface distribution in building-block and epoxy resin composite was analyzed experimentally. Simulations on idealized structures were done by FE calculations and corroborated by experimental data.

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Three-dimensional printing of SiSiC lattice truss structures

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ABSTRACT
Silicon/silicon carbide–ceramic composites were fabricated by the three-dimensional printing (3DP™) process from SiSiC (cermet) powder blends. After printing, the Si/SiC preforms were infiltrated with a liquid silicon resin for transient phase stabilization. The green bodies were pyrolyzed at 1000°C in nitrogen atmosphere resulting in a residual with a density of ∼45%. The porous preforms exhibit excellent infiltration behavior for liquid Si at 1500°C in vacuum. Bending strength, fracture toughness and Young’s modulus were analyzed with respect to Si volume fraction.

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1. Introduction
Silicon-infiltrated silicon carbide ceramics (SiSiC ceramics) are used for a wide range of engineering applications due to their excellent near-net shape fabrication and good mechanical properties combined with high chemical stability up to elevated temperatures [1-3]. SiSiC ceramics with a silicon volume fraction of 10-15% exhibit typical bending strength, fracture toughness and Young's modulus values of approximately 350 MPa, 4 MPa m^{1/2} and 350 GPa, respectively [4-6]. The mechanical properties were found to scale linearly with Si-content [7].

The reactive infiltration of a porous carbonaceous preform with liquid Si (SIS-process) offers the possibility to near-net shape manufacturing of dense SiSiC composites at relative low temperatures and reasonable costs [22]. According to the ISG-process, the RIFSE-process and the SECOMP-process were developed for industrial production of SiSiC since the 70s [8-10]. It should be noted that silicon is distinguished by a volume expansion of 10% upon solidification of the melt [4,11]. The volume expansion during solidification may lead to residual microstresses in the SiSiC composites [12,13]. In addition, due to different thermal expansion coefficients between Si and SiC, compressive residual microstresses in Si phase may arise. This microstress-induced strengthening effect may contribute to the improving mechanical properties of SiSiC ceramics [12,13].
Processing of preceramic paper and ceramic green tape derived multilayer structures

I. Götschel, B. Gutbrod, N. Travitzky, A. Roosen* and P. Greil

Multilayer laminates with gradients in material composition and porosity were fabricated by the combination of ZrO₂, Al₂O₃-ZrO₂ and Al₂O₃-MgAl₂O₄ preceramic papers with MgO-MgAl₂O₄, Al₂O₃ and MgAl₂O₄ ceramic green tapes. A ZrO₂-loaded adhesive based on an aqueous dispersion of copolymerised polyvinyl acetate served as the interface adhesive. The shrinkage behaviour of the individual layers was adapted by combining coarse and fine grained ceramic powders in order to avoid crack formation and delamination during firing. Defect-free multilayer laminates were obtained after sintering at 1700°C for 6 h which offer a high potential for application in refractory functional components.

Keywords: Preceramic paper, Ceramic green tapes, Multilayer laminates

Introduction

Refractories are exposed to high temperatures and must withstand thermal and mechanical stresses as well as corrosion by molten metal, slag, fluxes and corrosive atmospheres.¹,² Degradation of refractories during service in harsh environments is a complex phenomenon and the overall wear rate of refractories is dependent on the contributions of all degradation mechanisms involved.¹,² Though carbon bonded refractories exhibit superior degradation stability,³ carbon dioxide emission and carbon dissolution in steel melts are considered major disadvantages. The development of advanced refractory materials with low-to-no carbon content is a great challenge since chemical properties like wetting behaviour, compatibility between slag and refractory, and thermal shock behaviour must be taken into account. The thermal shock behaviour can be improved by tailoring the porosity, but porosity facilitates penetration of the metal or slag melt into the refractory material, and leads to interface reactions and corrosion degradation. Refractories of very high density, however, tend to be more susceptible to thermal shock damage.⁴ Designing multilayer refractories by combination of dense and only porous layers with corrosion resistant dense layers is of great interest for the fabrication of advanced carbon-free refractories. The multilayer design allows for the production of low carbon steel and reduces the amount of carbon dioxide emission. In this work, graded refractories are fabricated by alternating stacking and laminating of preceramic papers and ceramic green tapes.

Preceramic paper and ceramic green tapes

Preceramic paper has recently demonstrated the capacity to fabricate ceramics with various composition and porosity ranging from 15 to 60%.⁵ The pulp fibre network provides shape stability and strength to the green preceramic papers which, upon thermal decomposition and sintering, results in an interconnected pore structure with elongated pores. Tape casting is a well established low cost industrial process for the manufacture of thin ceramic sheets distinguished by low porosity of <1%.⁶,⁷ The tape sintering shrinkage and microstructure formation can be tailored in a wide range by applying multimodal powder mixtures.⁸,⁹ The preceramic paper and the ceramic green tape both contain plasticisers and binders, which provide high flexibility and excellent shaping ability and may facilitate multilayer laminate manufacturing.

Multilayer laminate processing

Ceramic multilayer technology is commonly used for the fabrication of electronic components like capacitors, inductors, high integrated circuits and actuators.¹⁰,¹¹,¹² Multilayer processing also offers the possibility to manufacture composite structures for structural applications, which can be composed of different layers with varied composition and microstructure. Typically, stacked ceramic green tapes are laminated by thermocompression where the adjacent green tapes are joined together at elevated temperatures and pressures. Mass flow is induced above the glass transition temperature of the binder-plastisol system and the particles of the neighbouring tapes interpenetrate across the interface. The properties of a multilayer composite can be made superior to those of the constituents by the appropriate laminate design.¹³ Mismatch in the layers’ shrinkage behaviour, however, can result in constrained sintering and give rise to an increased residual porosity.¹⁴,¹⁵ Furthermore, a mismatch in the coefficients of thermal expansion (CTE) may generate residual stresses upon cooling. While these internal stresses may increase fracture toughness, stresses exceeding a critical threshold can cause delamination and crack formation.¹⁶,¹⁷
Influence of coatings on microstructure and mechanical properties of preceramic paper-derived porous alumina substrates

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ABSTRACT

Preceramic papers loaded with inorganic fillers may be used as preforms in a novel manufacturing technique to fabricate lightweight ceramic structures. In order to reduce the porosity caused by burning out cellulose fibers and organics, porous preceramic paper-derived alumina substrates were post-treated via two different coating routes using silica suspension or methylvinylic/hydrgenic polylsine. Sintering of the alumina-filled preceramic papers in air at 1000°C for 2 h resulted in a non-uniformly distributed open porosity ranging from 25 to 26% after coating and infiltration. All samples were additionally heat treated up to 1500°C for 2 h. Thermal analysis (DTATC) was applied to determine the pyrolysis temperature of polysilane. Microstructure and phase analysis were performed respectively by SEM and XRD. After sintering, water absorption, apparent density and open porosity of test pieces were determined and mechanical properties of the substrates were evaluated before and after coating. For the samples coated with silica suspension, the mechanical strength remained in the same range of those for uncoated samples, while for the polysilane-coated samples the mechanical strength steadily increases after repeated impregnation steps, reaching ~350 MPa.

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1. Introduction

The search for increasing the strength and the stiffness, while decreasing the weight of materials for structural applications, boosted the research on the field of lightweight products, such as the ones produced from preceramic papers. As Travitzky et al. (2008) had shown, the preceramic process involves the conversion of a preform into a ceramic product through the removal of the organic polymer and the consolidation of the inorganic fillers. The packing characteristics of fibers, filler powders, and chemical additives, as well as the processing conditions are used to control the porosity of products obtained from the preceramic paper. Gutbrod et al. (2011) found, that the preceramic paper presents a porosity that may vary from 15% to 65%, after sintering in air, with the pore shape and size distribution templated by the pulp fiber morphology.

Porous ceramics can be used for different applications as heat-insulation structures, kiln furniture, porous burner substrates, fire protection structures, and catalyst supports.

In order to improve properties and the overall performance of the components, the surfaces of ceramic bodies are often subjected to treatments. For the purpose of form a multi-phase component, Marple and Green (1989) combined several materials to offer the possibility of adapting systems for specific applications. A path of introducing additional phases into body and allowing the matrix of relatively fine scale is the infiltration of powder composites with suitable liquid media. After this process, the sample can subsequently be heated to obtain a dense multi-phase component.

Lee et al. (2011) developed an infiltration technique to densely sinter a matrix in a high performance SiC/SiC composite. The slurry infiltration process was also used by Lu and Miao (2005) to produce highly porous SiC-doped alumina ceramics. In this case, well-dispersed alumina slurry was employed to infiltrate the pore space in polystyrene molds. Kem and Gadow (2004) presented liquid phase coating with ceramic precursors solutions, with subsequent drying, curing, pyrolysis and calcination, as a cost efficient process to deposit dense
Conceptional design of nano-particulate ITO inks for inkjet printing of electron devices

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Abstract This manuscript presents the conceptional design of indium tin oxide inkjet inks for the manufacture of electron devices. For this purpose, the processing window of the printed ink is identified and the inks are conceived to meet the requirements. The nano-particles are effectively stabilized in different dispersion media. The rheological, the wetting and the drying behavior of the inks are adapted to the inkjet process and the substrates to be coated. To assemble a field effect transistor (FET), the most suitable ink is chosen and source and drain contacts are printed. In the device, a nano-particulate ZnO layer acts as a semiconducting layer and the gate electrode as well as the dielectric layer is formed by a thermally oxidized silicon wafer. The electronic device assembled shows the typical FET characteristics proving its functionality.

Introduction

Inkjet printing is an emerging technology with many potential applications in the field of electronics and biotechnology such as the assembly of organic electronic devices, the direct printing of electronic bends on ceramics or the manufacture of "gas chips" [1–3]. Compared to more conventional printing methods like screen printing or offset printing, inkjet printing is more flexible as it is a direct printing technique and it is also contactless, which can be advantageous for sensitive substrates.

The inkjet printing technique was initially developed in the 1960s and 1970s using a continuous jet for industrial applications. Later on in the 1970s and 1980s the drop-on-demand printers using piezoe or bubble jet technology were developed [4]; the printers with piezo heads are still very frequently used for research activities today because they can be applied to a broad variety of inks [5–8].

Fromm [9] made a contribution to understanding the printability of the inks by numerically calculating the fluid dynamics of drop-on-demand jets using Navier–Stokes equations. In order to describe the fluid properties, he used the Reynolds $N_R$ number and the Weber number $N_W$ of the ink:

$$N_R = \frac{va \rho}{\eta}$$

$$N_W = \frac{v a \rho}{\sigma}$$

where $v$ is the velocity, $a$ is a characteristic dimension, i.e., the radius of the printing orifice, and $\rho$, $\eta$, and $\sigma$ are the fluid density, viscosity and surface tension, respectively. These two parameters can be summarized to the so-called parameter $Z$, which is the inverse of the Ottesen number $Oh$ [10, 11].

$$Z = \frac{1}{Oh} = \frac{N_R}{\sqrt{N_W}} = \frac{\sqrt{\frac{a \rho}{\eta}}}{\frac{v a \rho}{\sigma}}$$

For inkjet inks, the $Z$-parameter should lie between 1 and 14 [10, 11]. If the ink fulfills this condition, then a drop
Development of a model for the sintering of PZT multilayer ceramics and their dielectric properties

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Abstract

Piezoelectric multilayer ceramics are increasingly used in sophisticated applications for high-precision positioning systems. The reproducibility of the piezoelectric properties is of major importance for the manufacture of high-quality products. This study focuses on the variation of the sintering parameters and its effect on the poling behavior as a contribution to the establishment of an understanding of PZT multilayer processing.

To cover the complexity of the sintering process, the experiments were conducted with the design of experiments method. As parameters the sintering temperature, the holding time, the airflow in the furnace and the lead oxide atmosphere were investigated. As target variables the grain size, density and mass loss were investigated. In the following the correlations between the target variables and the sintering parameters were discussed and summarised in a model. The ceramic properties were correlated to the dielectric properties and the influence of the poling process was evaluated.

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Keywords: PZT; Sintering; Multi-layer; Microstructure-macro; Dielectric properties

1. Introduction

Lead zirconate titanate (PZT) ceramics are today widely used in industrial sensor and actuator applications, such as for pressure and ultrasonic sensors and actuators for fuel injection, due to their excellent piezoelectric properties.\textsuperscript{1,2} The application-specific adjustment of the material properties is achieved by the ratio of zirconate to titanate and by doping.\textsuperscript{3} Depending on the size of the incorporated ions, a soft or hard PZT is obtained by doping.\textsuperscript{4} A high strain PZT ceramic is achieved by doping with Sr, K and Nb. This so called PZT-SKN reaches elongations up to 2% and is therefore well suited for high strain applications.\textsuperscript{5,6}

The sintering process defines basically the microstructure, which in turn defines the electrical and mechanical properties of the multilayer, even though the result depends on the composition of the PZT and electrode material. During the sintering process the parameters temperature, time and the atmosphere have to be controlled to adjust the sintering density, shrinkage, mass loss and grain size.\textsuperscript{7}\textsuperscript{8} Typical sintering temperatures for the co-firing of PZT multilayers lie in the range of 950–1150°C, which depends on the stability of the electrode material and the liquid phase content in the ceramic during densification.\textsuperscript{7}

The sintering of PZT is a complex process due to the melting and evaporation of PbO starting at low temperatures of around 890°C. Therefore different methods exist to prevent a depletion of the Pb in the PZT. A PbO excess can be added to the PZT, which generates a liquid phase during sintering and enhances the densification.\textsuperscript{7} For such ceramics the overall sintering process can be described by the liquid phase sintering theory.\textsuperscript{8,9} For stoichiometric PZT an encapsulation with a Pb containing atmospheric powder, in which the Pb has a higher partial pressure than in PZT, is often used.\textsuperscript{7} In this case the sintering follows the solid state sintering regime.

For PZT multilayers which are co-fired with inner electrodes consisting of Ag and Pd, an additional influence of the electrode material on the sintering has to be considered. Donnelly et al.\textsuperscript{10} found reactions between Pb and Pd in the temperature range up to 800°C, which lead to the formation of a thin PbPdS\textsubscript{2} layer between the electrode and the ceramic. In contrast, Zno
Dense YSZ Laminates Obtained by Aqueous Tape Casting and Calendering**

By Veronica Moreno,* Dachom’ir Hotza, Peter Greil and Nahum Travitzky

Solid oxide fuel cells (SOFCs) are high temperature devices aimed to stationary heat and power generation.1-10 Compared to other fuel cells, SOFCs present the highest efficiency, being flexible in relation to fuel characteristics and operational temperature range (700–1000°C). SOFC devices consist of a dense electrolyte and two porous electrodes.4-11 The efficiency of the cells is determined by the performance of the electrolyte, which depends in turn on the ionic conductivity and density of the material.

Yttria-stabilized zirconia (YSZ) is widely used as the electrolyte material due to high chemical stability and mechanical toughness, as well as ionic conductivity above 700°C.10-14 Alternatively, scandia-stabilized zirconia (ScSZ) electrolytes show high ionic conductivity above 700°C comparable to that of YSZ at 1000°C. However, high cost and toxicity of ScSZ at temperatures below 600°C may limit the widespread use of this material. Doped ceria oxides, like gadolinia-doped ceria (GDC) and samaria-doped ceria (SDC), have a higher ionic conductivity, however, they tend to be chemically instable in reducing atmospheres.15-37

Different routes have been applied for the fabrication of planar SOFC electrolytes. By tape casting, 10–500 μm thick films may be produced. Typical electrolyte thickness in an electrolyte-supported cell is 150–200 μm.24 In order to produce dense SOFC structures, usually two or more cast tapes are laminated to produce dense SOFC structures. Interlayer delamination and interconnected porosity are not desirable because they decrease the ionic conductivity. Warm pressing is commonly used to laminate cast tapes.10,11,14 An alternative technique was reported23-24 so that 0.25–2.5 mm thick films were prepared by rolling a plastic ceramic mass to produce laminated with two or more layers.

The aim of the present work was to fabricate high dense YSZ electrolytes by aqueous tape casting followed by warm pressing or calendering. The influence of different processing routes on the density, microstructure, and mechanical properties of the laminates was investigated.

1. Experimental

1.1. Materials and Processing

SOFC electrolyte green tapes were produced by aqueous tape casting. Prior to casting, the slurry with 55 wt% YSZ powder (95Y2O3, 8 mol% Y2O3-stabilized ZrO2, Sigma-Aldrich) was deagglomerated in deionized water with 1 wt% dispersant (Darvan 82A, Vanderbilt) using ball milling for 24 h. After deagglomeration, binder (Mowilith LDN-613B, Clariant), antifoam (Antifoam A, Sigma-Aldrich) and surfactant (coconut diethanolamide, Stepan) were added and the slurry was mixed for further 30 min. The as-fabricated slurry was cast at 25°C by a tape caster (CC-1200, Mßßller) with a Mylar carrier coated with a silicon layer (G10/RM, Mßßller). A casting speed of 6 cm min−1 was used. The gap between the blade and the carrier was adjusted to obtain a tape thickness of 90–200 μm. The green tapes were dried at 25°C for 24 h, cut into 5 × 5 cm2 samples and laminated at 40°C by warm pressing and calendering.

Two sheets were laminated and laid one in the cast direction and the other perpendicular to the cast direction. Lamination by warm pressing (LA-4,5, Bürklen) was carried out in a steel die. Pressure varying between 16 and 19 MPa was applied for 5 min. The lamination by calendering (CAS, Sumitomo) was performed at 10–15 MPa with a roll speed of 10 m s−1. Tapes were further passed freely through the calender or placed between two copper sheets with 0.5 mm thickness and passed through calender rolls.

Debinding and sintering were carried out in an electrically heated furnace (HT-16/17, Naberntherm) in air. Debinding of the green laminates was performed by heating up to 550°C with the rate of 0.5 °C min−1 and holding time of 1 h. The laminates were then heated up to 1000°C with the rate of 5°C min−1. After holding time of 1 h, the laminates were cooled down with the rate of 5°C min−1.
ORIGINAL ARTICLE

Synthesis and characterization of laminated Si/SiC composites

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KEYWORDS
Laminates; Si/C composites; Microstructure; Porosity; Oxidation resistance

Abstract Laminated Si/SiC ceramic were synthesized from porous preforms of digerous carbon impregnated with Si slurry at a temperature of 1500 °C for 2 h. Due to the capillarity infiltration with Si, both intrinsic micro- and macrostructure in the carbon preform were retained within the final ceramics. The SEM micrographs indicate that the final material exhibits a distinguished lamellar structure with successive Si/SiC layers. The produced composites show weight gain of ±5% after heat treatment in air at 1000 °C for 20 h. The produced bodies could be used as high temperature gas filters as indicated from the permeability results.

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Introduction

Porous SiC ceramis have drawn attention in the field of porous ceramics due to their superior properties, such as low thermal expansion coefficient, high thermal conductivity and excellent mechanical strength [1–3]. However, their brittleness limits their use in most structural applications. To improve fracture resistance in brittle material–matrix composites, the use of a weak interface that promotes crack deflection is necessary [4]. The earliest ceramics composites used as interfaces are boron nitride or carbon; however, these materials are prone to oxidation at high temperature. Porous oxide layers seem to be an attractive alternative and have been successfully demonstrated as effective interface layers in laminated ceramic composites [5,6]. Laminated system consisting of porous-Al2O3 interfaces between Al2O3 bars showed markedly improved fracture resistance for these composites as compared with monolithic Al2O3 [5].

Clegg et al. [7] have produced laminated SiC with graphite interface layers. These monolayer SiC composites showed apparent toughness and fracture energy 5 and 200 times, respectively, higher than the typical values of monolithic – SiC. However, it was shown that laminated composites without weak interfaces also exhibited damage-tolerant behaviors [8,9].
Robocasting of alumina hollow filament lattice structures

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Abstract

Robotic controlled deposition (robocasting) of an aqueous colloidal α-Al₂O₃ gel for manufacturing of cellular ceramics with periodical lattice structure was investigated. The colloidal gel was loaded with 50 vol% α-Al₂O₃ and exhibits shear-thinning behavior, a shear modulus of 288 kPa and a yield-stress of ~70 Pa. Tabular filaments of circular and rectangular cross section having an outer diameter of 1.5 mm and a capillary diameter of 0.75 mm were deposited in an oil bath to fabricate lattice truss structures with free spanning filaments. After freeze drying the robocast grids were sintered in air at 1550 °C. X-ray μ-CT revealed continuity of the tabular filaments for long distances (~550 mm). Critical conditions to avoid capillary collapse were discussed by considering bulging stress and pressure distribution within the hollow filament. At short filament length its instability into the capillary driven by capillary suction supports the tabular filament whereas oil flow driven by movement of the tool nozzle causes pressure difference to increase linearly with increasing filament length.

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Keywords: Robocasting; Colloidal gel; Rapid prototyping; Filament based writing; Hollow filaments

1. Introduction

Robocasting is a filament based writing technology which creates ceramic patterns of variable architecture and composition. In contrast to droplet-based deposition techniques such as 3D printing or inkjet printing, a continuous rod-like filament is delivered through a nozzle of defined shape making it possible to build up three dimensional ceramic patterns. Filament formation and shape retention are achieved by tailoring the rheological behavior and solidification kinetics of the feedstock suspension. Aqueous colloidal gels were applied to manufacture three dimensional space filling monoliths as well as lattice structures even with free spanning filaments. While robocasting in air required nozzles with diameters exceeding 500 μm, decoupling the deposition kinetics from the drying process by extrusion into an oil bath allowed generation of filament diameters less than 100 μm. Ceramic gels based on silica, alumina, mullite, lead zirconate titanate, titania, hydroxyapatite, lead magnesium niobate (PMN), porcelain and barium titanate were successfully applied to robocasting. Applications of the ceramic robocasting process include for example highly porous grid structures for bone restoration, catalyst carriers and meshes for filters. Robocasting requires a feedstock which exhibits rapid recovery of gel elasticity after leaving the tool nozzle for shape retention of the extruded filament. Flocculated colloidal suspensions (colloidal gels) were demonstrated to provide suitable rheological properties (G’ > 100 kPa, G” > 100 Pa, η > 10 Pa s⁻¹). The shear thinning behavior to enable continuous deposition of filaments with feature sizes down to 100 μm was also free spanning structures. Gelation of powder suspensions with schematic volume loading fractions ranging from 0.3 to 0.5 applied in robocasting shaping process was induced by a rapid increase of interparticle bond strength either by lowering the pH-value, increasing the ionic strength or adding a polymeric flocculant, respectively. In order to prevent sedimentation and synergy cellulosic derivatives (Hydroxypropylmethylcellulose, Ethylhydroxyethylcellulose) were added which give rise for appreciable yield-stress of the particle suspension. Depending on the extrusion speed and the rheological behavior, flow of a colloidal gel through the extrusion nozzle may cause a pronounced shear rate gradient over the filament diameter which results in plug flow with an unyielded core and a surface region depleted of
PLLA/HA Composite Laminates

By Steferson Luiz Stares, Márcio Celso Fredel, Aguendo Aragones, Elazar Y. Gutmanas, Irena Gotman, Peter Greil and Nahum Travitzky

In recent decades, it has been carried out studies on different absorbable materials for the fabrication of implant in order to eliminate a number of complications associated with the use of metallic implants. Facing a complex biological and sensitive system as the human body, an ideal absorbable material must meet certain medical and mechanical requirements, in order to be safe e.g. in fracture fixation surgery. The medical requirements are mainly related to the biocompatibility and biodegradability of the material. In order to attend mechanical requirements, has to be observed: (a) a high initial strength to withstand the stresses during the surgical procedures of implantation and support the external and physiological loads during the early stage of tissue integration; (b) an appropriate elastic modulus, i.e. the material should not be excessive rigid or flexible to its intended use and (c) should not exhibit brittle fracture mechanism, as this would cause a concentration of inflammatory cells due to detachment of fragments. The aim of the present work is to investigate some properties of novel PLLA/HA multilayer composites with the further goal of producing devices for use in bone fixation.

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1. Experimental

1.1. Materials and Sample Preparation

The materials used in this research: poly-l-lactide – PLLA (Laboratory of Biomaterials, PUC-Sorocaba, Brazil) as the matrix of composite and synthetic hydroxyapatite – HA (Ca10(PO4)6(OH)2 – Merck, Darmstadt, Germany) as reinforcement. PLLA with an Mw of ≈151 kDa and density of 1.23 g cm⁻³ was supplied in the form of granules (60 μm × 3 mm). The HA mean particle size used was 4 μm and density of 3.15 g cm⁻³. The HA content in the composites were 5, 10, and 20 wt%.

The polymer granules and the HA powder were mixed by hand in desired proportions and melt extruded to cylindrical rods of a diameter approximately 5 and 50 mm length using a twin-screw extruder (HAAKE PolyLab System PTW 16/25 – Thermo Electron Corp., Wallingford, USA). The extrusion temperature was held at 170 °C and the screw speed 10 rpm. By using a mold these rods were compression molded to form sheets with approximately 250 μm thick. Thereafter, double sheets were symmetrically stacked and again compression molded to make two groups of samples relative the HA content. The first group was formed by a sequence 21-10-5-0-5-10-20 (denoted as “20-20-20”) and the second group was formed by a sequence 0-5-10-20-10-5-0 (denoted as “0-20-0”) related to the reinforcement content. Compression moulding was conducted at 100 °C and 20 MPa in an axial press (Polystat 207 – Servitec Maschinenbau GmbH, Wustermark, Germany).

1.2. Characterization

Samples were freeze-fracture using liquid nitrogen to enable examination of interior cross-sections and fracture surfaces. The exterior and interior of samples were examined to assess the influence of HA particle addition on morphology and microstructure by using SEM micrographs (JEOL, Quanta 200, FEI, Czech Republic).

Bending strength and elastic modules were measured using a three point flexural loading device configuration. The distance “L” applied between the supports was 20 mm. The radius of the supports was 5 mm and the dislocation velocity of the headstock was 5 mm min⁻¹. At least 10 specimens were tested for each composition and the mean values and standard deviations calculated. The tests were conducted in a universal testing machine (Instron 5565, Instron Corp., Canton, MA, USA) with load cell capacity of 500 N.
Paper-derived β-TCP

S.L. Stare, A., M.C. Fredel, P. Creij, N. Travitzky

1. Introduction

β-TCP (tricalcium phosphate) is a synthetic bioactive equivalent to human bone. Some investigators have reported bone ingrowth (osteoconduction) through the use of β-TCP scaffolds leading to bone regeneration in critical-sized defects [1,2]. Pre-ceramic paper processing is a novel, economic approach for the manufacturing of ceramic components. The process can be used to create a wide variety of shapes with tailored macro- and microscopic properties for a broad field of applications [3-6]. Pre-ceramic paper is made up of inorganic fibers and loaded with inorganic powders [5]. The processing approach used for the deposition of fibers or mixtures with or without the addition of fillers defines the paper properties [3]. The organic fraction of the paper substrate is burned out during firing in air, leaving a porous ceramic residue [7]. In the present work, a novel pre-ceramic paper derived β-TCP has been developed with the goal of fabricating porous structures for use in bone reconstruction surgery. Paper wash formation, binder behavior and microstructure were studied as well as the compressive strength and compressive modulus.

2. Experimental

Preparation of preceramic paper: Pre-ceramic papers loaded with β-TCP spheres and pulp fibers were prepared from dilute aqueous suspensions β-TCP (Ca₃(PO₄)₂) spheres were obtained from Sigma-Alrich, Steinheim, Germany with a mean particle size of 3 μm and density of 3.14g cm⁻³. The aqueous suspension of a pulp mixture containing 0.60 w/w% non-refined softwood pulp (Celi Sarl, France) with an average diameter of 0.5 μm and an average length of 0.7 μm was homogenized by vigorous stirring at a pH=7.4 for 1h. Solid retention was obtained by flocculation in the feedstock suspension, induced by addition of 0.005 wt% anionic starch ether (Rhönhoff A5, Friedrich Gmbh, Germany). Pre-ceramic paper sheets were formed on a Rapid Elithen sheet forming device (Haage Labortechnik BBS-2, Haage GmbH, Mühlenbach an der Ruhr, Germany). Circular sheets with a diameter of 200 mm were obtained after drying under vacuum < 10⁻³ Pa. The as-formed specimens were dried at 93°C for 15 min resulting in pre-ceramic paper sheets denoted series A, B, and C. Table 1 summarizes the feedstock composition excluding water. The pre-ceramic paper sheets were sintered in air atmosphere in an electrically heated furnace (HT 16/27, Zabertherm, Unternötting, Germany). A single step annealing sequence was applied where the temperature was raised with a constant heating rate of 2°C min⁻¹ up to 200°C, followed by a heating rate of 1°C min⁻¹ up to 450°C. Temperature was held at 450°C for 2 h followed by subsequent heating to 800°C at 1°C min⁻¹ to allow complete removal of pulp fibers and paper aid chemicals. The sheets were sintered at 1200°C for 2 h. Heating from 600 to 1200°C and cooling were set to 5°C min⁻¹. Characterization of samples: The apparent density of the specimen (ρApp) was determined from weight and volume measurements. The thickness of the samples was measured with a digital dial indicator. Skeletal density (ρSkelet) of the samples was measured by He-Pycnometry (AccuPyc 1330, Micromeritics Instrument Corporation, Norcross, GA, USA). Total porosity of
Paper-derived hydroxyapatite

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Abstract

Porous hydroxyapatite structures were manufactured via a novel preceramic paper process. Preceramic paper sheets were produced from aqueous suspensions loaded with different contents of pulp fiber and HA fiber. Pressure loading was applied in order to increase the packing density in the paper sheets. The sheets were sintered at 1250 °C for 1 h. The porous ceramic specimens were characterized for density, porosity, microstructure and mechanical properties. A pronounced volumetric shrinkage was observed, but no surface flaws or inhomogeneous areas were detected. The mechanical strength using the ball on three ball test (B3 test) and elastic modulus of sintered specimens vary between 18 and 28 MPa and 0.65–1.53 GPa, respectively.

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Keywords: Preceramic Paper; Hydroxyapatite, Bone tissue

1. Introduction

Recently a preceramic paper processing approach was used for the manufacturing of ceramic components. The process can be used to create a wide variety of shapes with tailored macro- and microscopic properties for a broad field of applications [1–6]. Preceramic paper is made up of inorganic fibers and loaded with inorganic powders [7]. The processing approach used for the deposition of fibers or their mixtures with or without the addition of fillers defines the paper properties [1]. The organic fraction of the paper substrate is burned out during firing in air, leaving a porous ceramic residue [8].

In the present work, a novel preceramic paper derived hydroxyapatite has been developed with the goal of fabricating porous structures for use in bone reconstruction surgery. Paper web formation, sintering behavior and microstructure were studied as well as the strength and elastic modulus. In order to increase packing density in the sintered ceramic product, the effect of pressure on the paper properties was also studied.

2. Experimental

2.1. Preparation of preceramic paper

Preceramic papers loaded with hydroxyapatite spheres (HA) and pulp fibers were prepared from dilute aqueous suspensions. HA (Ca10(PO4)6(OH)2) spheres were obtained from Merck, Darmstadt, Germany, with a mean particle size of 4 μm and density of 3.15 g cm⁻³. The aqueous suspension of a pulp mixture containing 0.30 wt% non-refined softwood pulp (Celti PP, Celulose Beira Industrial (CBI) S.A., Figueira da Foz, Portugal) with an average diameter of 15 μm and an average length of 657 μm was homogenized by vigorous stirring at a pH = 7.6 for 1 h. Solid retention was obtained by flocculation in the feedstock suspension, induced by addition of 8.0 vol% anionic starch ether (Fibrafilm A5, Südstraße GmbH, Schorndorf, Germany).

Preceramic paper sheets were formed on a Rapid Köthen sheet forming device (Haage Laborblattbildner...
Paper-Derived Bioactive Glass Tape**

By Steherson Luiz Stares,* Alina Kirilenko, Márcio Celso Fredel, Peter Greil, Lothar Wondraczek and Nauman Trovitzky

Porous bioactive glass (BaG) structures were manufactured applying novel preceramic paper process. Preceramic papers were produced for aqueous suspensions loaded with different contents of pulp fiber and BaG filler. Pressure loading was applied in order to increase the packing density in the paper sheets. The paper sheets were sintered at 630°C for 1 h. The porous glass–ceramic specimens were characterized for density, porosity, composition, microstructure, and mechanical properties. A pronounced volumetric shrinkage was observed, but no surface flaws or inhomogeneous areas were detected. The mechanical strength using the ball on three balls test and elastic modulus of sintered specimens vary between 21 and 35 MPa and 0.30–0.85 GPa, respectively.

The ultimate goal in the area of reconstructive medicine is the structural and functional restoration of tissues to its natural state. Bioactive glasses (BaGs) have been given a lot of attention as candidate implant materials since they possess highly desirable characteristics for some applications.5–21 The main advantage is their high biocompatibility.22–24 Specialy formulated BaGs are chemically active and favorably react with body fluids to form chemical bonds with soft and hard tissue. Some investigators have varied the composition of the former in order to obtain maximum induction of direct bonding bone.5–6 However, clinical application of BaGs is limited due to their inherent properties: brittleness, weak tensile strength, and difficulty in deposits on the surface of other stronger materials. For these reasons, the majority of applications are limited to the maxillofacial and dental areas,5–6 although some load-bearing applications have also been attempted.5,11 BaG implants were manufactured by different techniques including: injection molding, extrusion, solvent casting, foaming replication method, plasma spraying, 3D-printing between others.11–40

Preceramic paper processing is a novel, economic approach for the manufacturing of ceramic components. The process can be used to create a wide variety of shapes with tailored macro- and microscopic properties for a broad field of applications.15–29 Preceramic paper is made up of inorganic fibers and loaded with inorganic powders.15 The processing approach used for the deposition of fibers or their mixtures with or without the addition of fillers defines the paper properties.15 The organic fraction of the paper substrate is burned out during firing in air, leaving a porous ceramic residue.15

In the present work, a novel preceramic paper-derived BaG has been developed with the goal of fabricating BaG structures for use in bone reconstruction surgery. Paper web formation, sintering behavior, and microstructure were studied as well as the strength and elastic modulus. In order to increase packing density in the sintered BaG product the effect of pressure with different processes: (a) axial pressure and (b) calender, on the fabricated samples properties was explored.
Fabrication of Functional Nanoparticulate Coating in the Submicrometre Range with the Slot Die Process

Introduction

The tape casting method [1] is used for industrial processing of suspensions prepared from ceramic powders to films. The obtained green tape then being the precursor product for ceramic multilayer technology [2]. In tape casting, the ceramic slurry is in a special chamber, which is equipped with a doctor blade, under which a moving substrate draws out the slurry to form a thin coating. Typical coating thicknesses range between 100 and 1000 μm, but coating thicknesses around 1 μm are also achieved. With the application of another doctor blade concept, known as the profile rod technique, coating thicknesses of around 200 nm are obtained [3]. The drawing speed of this process reaches up to 60 m/min. Much faster speeds for such thin coatings up to >100 m/min are achieved with the application of the suspension onto the moving substrate with a broad slot die operated in the bead coating mode.

In all the above-mentioned processes for obtaining coating thicknesses <10 μm, the colloidal preparation of the particles is crucially important. Unlike the application of solutions, for the application of particulate systems, the powder particles must be first deagglomerated and stabilized in a solvent by means of surfactants or ultrasound, the latter being achieved with the use of appropriate dispersants. In the processing of nanoscale powders for coating thicknesses <1 μm, the selection of a short chain dispersant is crucial [4]. This article looks at the preparation of nanoscale indium tin oxide powder and the subsequent processing of these suspensions to submicrometre thin films by means of the slot die process.

Coating methods

In many fields of semiconductor technology, tests are conducted to produce coatings in some 10 nm ranges and in some 100 nm ranges with the help of the wet film method to apply functional coatings to large-area film substrates at low cost. In today's coating technology, a wide range of methods is applied to apply thin liquid coatings onto sheet-like substrates or films [5]. In today's coating technology, mainly self-metering methods (e.g., doctor blading) are applied to apply thin liquid coatings onto substrates. Here, the achievable thickness of the coating is dependent on the substrate preparation (difficult to control and heavily dependent on the fluid properties), the selected coating method and the freely selectable parameters as well as the coating speed. In contrast to the state of the art, with pre-metered coating systems (broad slot dies operate in the bead [6], web-tensioned, extrusion, short-curtain or curtain-coating mode [7]) the desired wet film coating thickness can be determined based on the measured, forcibly metered mass flow and the known substrate speed. Particularly, when the coating quality must meet very high requirements, like, for example, in the processing of ceramic powders, suspensions to large-area functional coatings in the nano- to micrometre range, the pre-metered broad slot die method boasts key advantages with regard to controllability and reproducibility.

Self-metering coating methods

The self-metering methods (roll, doctor blade, anilox roll coating) are characterized by the fact that the wet film coating thickness is determined by the coating process and cannot be determined by the adjustment of the mass flow during operation of the coating tool. The coating thickness on the substrate is relatively uncontrolled and is heavily dependent on the fluid properties, the selected coating method and freely selectable parameters and the coating speed. In the case of self-metering coating processes, changes in the coating weight are...
Proceedings

T. Früh, U. Deisinger, A. Roosen
Manufacture of Highly Anisotropic Ceramic-Polymer Composite Films Mimicking the Structure of Natural Nacre

M. Hambuch, F. Gora, K. Beart, F. Wittmann, A. Roosen
Joining of Sintered Alumina Substrates and LTCC Green Tapes via Cold Low Pressure Lamination

D. Jakobsen, I. Götschel, A. Roosen
Fabrication of Multilayer Composites for Refractory Applications via Tape-Casting of Ultra-Thick Green Tapes

D. Jakobsen, I. Götschel, A. Roosen
Tape Casting of coarse-grained oxide powders for the manufacture of advanced refractory multilayer composites
Books

P. Greil, T. Fey, C. Zollfrank
Biomorphous Ceramics from Lignocellulosic Preforms

Patents

Slide ring sealing and method of manufacturing
4. **CONFERENCES, WORKSHOPS, LECTURES, AWARDS**

Conferences and Workshops organised by Members of the Institute

**A. Roosen**

6th Advanced Training Course on “Tape Casting and Ceramic Multilayer Technology”, University of Erlangen-Nuremberg, 19 February 2013

*Members of the 6th Advanced Training Course on “Tape Casting and Ceramic Multilayer Technology”*
A Roosen
Session Chair, 5th International Conference on Shaping of Advanced Ceramics, Mons, Belgium, 29-31 January 2013

A. Roosen
Member of the Program Committee and Session Chair, Annual Meeting of the “Deutsche Keramische Gesellschaft”, Weimar, 18-20 March 2013

A. Roosen
Session Chair, 13th International Conference of the European Ceramic Society, Limoges, France, 23-27 June 2013

A. Roosen
Member of Advisory Board and Session Chair, 12th Intern. Conf. on Ceramic Powder Processing Science ICCPS-12, Portland, OR, USA, 4-7 August 2013

A. Roosen
Member of the Program Committee and Session Chair of the DKG-Symposium: “Verfahren zur Herstellung keramischer Schichten”, Erlangen, 3-4 December 2013

N. Travitzky
Symposium Organizer: PACRIM 10 The 10th Pacific Rim Conference on Ceramic and Glass Technology – “Innovative Processing and Manufacturing: Symposium 3: Novel, Green, and Strategic Processing and Manufacturing Technologies”, San Diego, California, USA, 2-7 June 2013

N. Travitzky
Opening Ceremony of the European Liaison Office of the Nagoya Institute of Technology (NiTech)

On July 15 the President of the NiTech, Professor Minoru Takahashi officially opened the European Liaison Office in the presence the General Consul of Japan at Munich, the Dean of the Technical Faculty delegations of Japanese and German professors. NiTech (http://www.nitech.ac.jp) is among the leading engineering schools in Japan with more than 4500 students and a broad spectrum of engineering disciplines. Starting in 2010 cooperation between NiTech and FAU focused on the area of high performance ceramics, biomaterials as well as materials for electronics and energy systems. More than 60 japanese students, postdocs and faculties already attended the joint seminars on materials science held at Erlangen.

The European Liaison Office hosted by the Technical Faculty is the second international liaison office of NiTech after the first one established in 2011 at the Beijing University of Chemical Technology (BUCT). It is the aim of the office to strengthen the cooperation in science and teaching between NiTech and FAU at least in the fields of materials science. Furthermore, cooperation will be extended to other engineering disciplines. The European Liaison Office will support international promotion and careers of young Japanese students by triggering formation of novel academic networks throughout Europe.
Opening Ceremony of the European Liaison Office of the Nagoya Institute of Technology (NiTech)

NiTech Europe Liaison Office at FAU on July 15, 2013
Science Night

Science Night (Lange Nacht der Wissenschaften) on 19 October 2013 involved a large number of institutes in the region of Erlangen, Fuerth and Nuremberg. From 6 pm up to 1 am the Department of Materials Science opened the lab doors to show interested public exclusive experiments and latest research results in a common way.

The Institute of Glass and Ceramics offered the following attractions:

- 3D-movie with red/green glasses on cellular ceramic structures
- Ceramic implants in a human skeleton model
- Fantastic handcraft in blowing beautiful glass ware.
Impression of the Science Night 2013 in our technical hall
Invited Lectures

**T. Fey**, M. Stumpf, P. Greil

Microstructure evaluation and simulation of micro cellular ceramic

*2nd International Symposium on Ceramics Nanotune Technology, Nagoya Institute of Technology, Japan, 6-8 March 2013*

B. Ceron-Nicolat, F. Wolff, A. Dakkouri-Baldauf, **T. Fey**, H. Münstedt, P. Greil

Processing and Characterization of graded cellular polymer derived ceramics

*PACRIM 10 The 10\(^{th}\) Pacific Rim Conference on Ceramic and Glass Technology, San Diego, CA, USA, 2-7 June 2013*


Cellular polymer derived ceramics: microstructure characterization and simulation

*8th International Conference Series on High Temperature Ceramic Matrix Composites – HTCMC-8, Xi’an, China, 22-26 September 2013*

**P. Greil**, M. Götz, T. Fey

Ceramics with Periodic Microstructures

*Shanghai, China, Shanghai Institute of Ceramic Advanced Ceramics Conference, 21 September 2013*

**P. Greil**, L. Schlier, N. Travitzky

Surface Healing of Polymer Derived Ceramic Matrix

*8th International Conference Series on High Temperature Ceramic Matrix Composites – HTCMC-8, Xi’an, China, 22-26 September 2013*
**A. Roosen**

Tape casting: Design diversity in building planar multilayer structures

*Colloquium "Recent trends and developments in ceramic process technology",
Waldkraiburg, 5 June 2013

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**A. Roosen**

Powder preparation and forming methods

*Summer school “Ceramic science and technology for the 21st century: basic principles and modern trends”*, Limoges, France, 20 June 2013

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**A. Roosen**

Advances in ceramic green tape technology

*13th International Conference of the European Ceramic Society, Limoges, France, 27 June 2013*

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**A. Roosen**

Manufacture of particulate structures in the micrometer range via coating and printing techniques

*12th Intern. Conf. on Ceramic Powder Processing Science ICCPS-12, Portland, OR, USA, 7 August 2013*

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**A. Roosen**

Shaping of advanced ceramics.

*1st International PhD Summer School "Optimized Processing of Multi-Material Architectures for Functional Ceramics", Risø, Denmark, 26-30 August 2013*
N. Travitzky

Ceramic-metal composites

PACRIM 10 The 10th Pacific Rim Conference on Ceramic and Glass Technology, San Diego, CA, USA, 2-7 June 2013

M. Wegener, J. Kaschta, H. Münstedt, A. Roosen

Gelling of PVB-based slurries with addition of Ti-esters

6th Usermeeting Rheology TA Instruments, Erlangen, 11 October 2013

M. Wegener, N. Kölpin, A. Roosen

Tape casting of submicron thick TCO layers and their processing


Awards

T. Früh

Manufacture of highly anisotropic ceramic-polymer composite films mimicking the structure of natural nacre

Winner of the Student Poster Contest, 5th International Conference on Shaping of Advanced Ceramics, 29-31 January 2013, Mons, Belgium

A. Roosen

In December 2013 Prof. Dr. Andreas Roosen was appointed by the President of the Technical University of Denmark (Copenhagen) as Adjunct Professor of the Department "Energy Conversion and Storage". The appointment will strengthen existing activities in research and teaching in the field of ceramics processing, in particular in the field of multilayer ceramics.
5. ADDRESS AND MAP
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By car:
Highway A3 exit Tennenlohe; direction to Erlangen (B4).
Follow the signs “Universität Südgelände“.
After junction “Technische Fakultät“ please follow the map.

By train:
Railway station Erlangen.
Bus line No. 287 direction “Sebaldussiedlung“.
Bus Stopp “Technische Fakultät“: 50 meters to a layout plan; search for “Department Werkstoffwissenschaften“.

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6. IMPRESSUM

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