FRIEDRICH-ALEXANDER
UNIVERSITÄT
ERLANGEN-NÜRNBERG

TECHNISCHE FAKULTÄT

Department of Materials Science

Glass and Ceramics

ANNUAL REPORT 2013

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

Outline

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. Eng. Michael Hambuch

Dipl.-Ing. Daniel Jakobsen

M. Sc. Moritz Wegener

M. Sc. Zongwen Fu

M. Sc. Ruth Hammerbacher

Dipl.-Ing. Alfons Stiegelschmitt

Kosseleck Group

Dr. Guo Ping Bei

Dr.-Ing. Joana Pedimonte

Dipl.-Ing. Kathrin Ebert[2]

Dr. Sergey Sirotkin[3]

Cellular Ceramics and Simulation

Dr. rer. nat. Andrea Dakkouri-Baldauf

PhD Guifang Han

M. Sc. Bastian Weisenseel

M. Sc. Franziska Eichhorn

Dr. rer. nat. Sreejith Krishnan[4]

Dipl.-Ing. Bodo Zierath

Technical Staff

Sabine Brungs

Dipl.-Ing. Helmut Hädrich[5]

Timotheus Barreto-Nunes

Peter Reinhardt

Eva Springer

Hana Strelec

Evelyn Gruber

Beate Müller

Heike Reinfelder

Alena Schenkel-Rybar

Dipl.-Ing. Alfons Stiegelschmitt

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 Lumière 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 femtolaser, 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.

Dr. Tobias Fey presenting a "survival gift" to Ms. Ursula Klarmann

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 electronical 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.

Prof. Peter Greil congratulating Mr. Helmut Haedrich

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_2O_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

Dr. Joana Pedimonte after successful Ph.D. examination

Sindy Fuhrmann

Pressure dependence of the topological heterogeneity of glasses

Dr. Sindy Fuhrmann after successful Ph.D. examination

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 Wuerzburg 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 7^{th,} 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, N_2, Ar, Vac, High-Vac, forming gas) up to 2500°C for sintering, glass melting, infiltration, debindering, 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.

Research Projects (in alphabetical order)	**Funding**	**Principal Investigator**
Bioactive ceramic cages	IN	Prof. Greil / Dr. Fey
Development of layered structures and 3D generative processing methods for innovative combustion chamber lining concepts	BMWi + IN	Prof. Roosen / PD Dr. Travitzky
Cellular ceramics for heat absorbers	EnCN	Prof. Greil
Deformation and sintering behaviour of preceramic papers	DFG	PD Dr. Travitzky
Experimental study and simulation of anisotropic effects in cast green tapes	DFG	Prof. Roosen
Flexible manufacturing of preceramic paper based refractory components	DFG	Prof. Greil
Hierarchical cellular ceramics and composites	DFG	Prof. Greil
High temperature stable ignition components based on defined 2D and 3D SiSiC structures	AiF	PD Dr. Travitzky
Lightweight cellular ceramics	EC	Prof. Greil

Manufacturing of multilayer refractories by tape casting	DFG	Prof. Roosen
Manufacturing of transparent ceramic substrates	BMBF	Prof. Roosen
Stable and metastable multiphase systems for high application temperatures	DFG	Prof. Greil
Dispers systems for electronic devices	DFG	Prof. Roosen
Polymer derived ceramics for bearing applications	IN	PD Dr. Travitzky
Robocasting of macrocellular ceramic 3D-lattice structures with hollow filaments	DFG	PD Dr. Travitzky
Self healing MAX phase ceramics	DFG	Prof. Greil
Structured carbon based catalyst support structures for CO hydration	DFG	Dr. Fey
Tape on Ceramic Technology	BMBF	Prof. Roosen

Funding organisations:

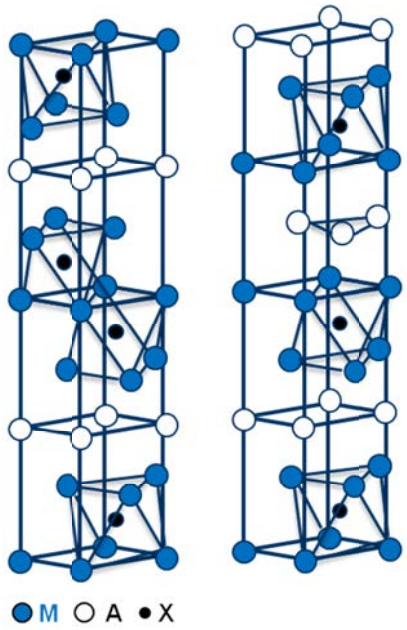
AiF: Industrial research Cooperation
BMBF: Federal Ministry of Education and Research
BMWi: Federal Ministry of Economics and Technology
DFG: German Research Foundation
EC: Cluster of Excellence (“Engineering of Advanced Materials”)
EnCN: Energy Campus Nuremberg
IN: Industry

Selected Research Highlights

Crack healing in MAX phase ceramic composites

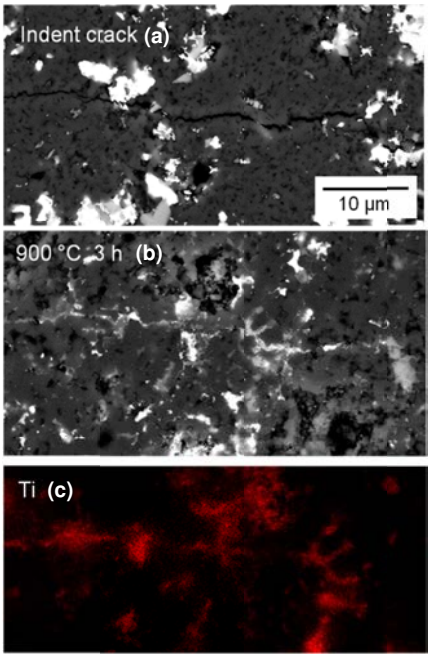
Joana Pedimonte, Guoping Bei, Tobias Fey, Peter Greil

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 Al_2O_3 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.

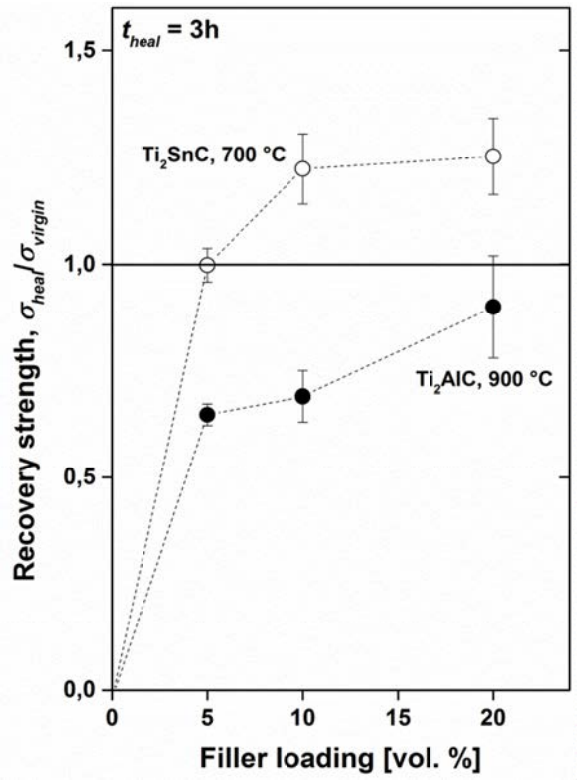
Significantly lower healing temperatures < 1000 °C may be achieved by loading Al_2O_3 with repair filler particles which undergo oxidation in near surface cracks thereby filling the crack space with oxidation products. MAX phase particles, with the general formula $M_{n+1}AX_n$ (n = 1 to 3) with M = 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 Ti_2SnC 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 $Ti_2Al_{(1-x)}Sn_xC$ (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_2AlC 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_2SnC. The enhanced healing response of Ti_2AC (A = 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_2AlC) and at 700 °C (Ti_2SnC).

G.P. Bei, B. J. Pedimonte, T. Fey, P. Greil

Oxidation Behavior of MAX Phase $Ti_2Al_{(1-x)}Sn_xC$ Solid Solution; *J. Am. Ceram. Soc. 96 (2013) 1359*

B. J. Pedimonte, D. Pourjafar, G. P. Bei, T. Fey, P. Greil

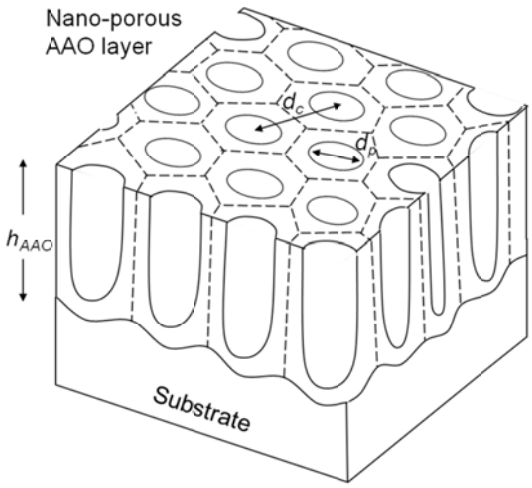
Oxidative crack healing in Al_2O_3 composites loaded with Ti_2AC (A = Al, Sn) repair fillers; *J. Cer. Sci. Techn. (2014) in press*

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.

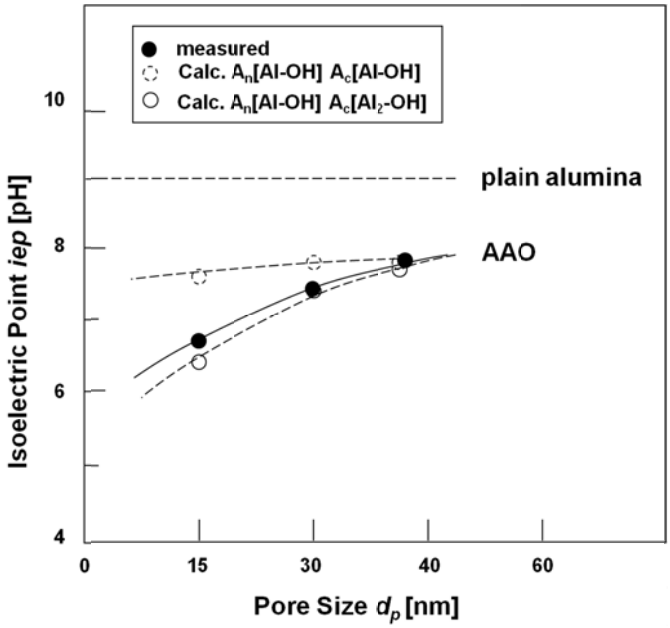
Coating of various biomaterials with a nanoporous anodic alumina (AAO) surface layer at different anodization voltages (20 – 60 V) with varying pore diameter (15 – 40 nm), a mean pore distance (40 – 130 nm) and a total porosity of ~ 10% that elicits a favorable response for osteoblasts might be attractive in bone replacement as well as bone tissue engineering.



AAO layers formed in $C_2H_2O_4$ (0.22 M) at different anodization potentials.

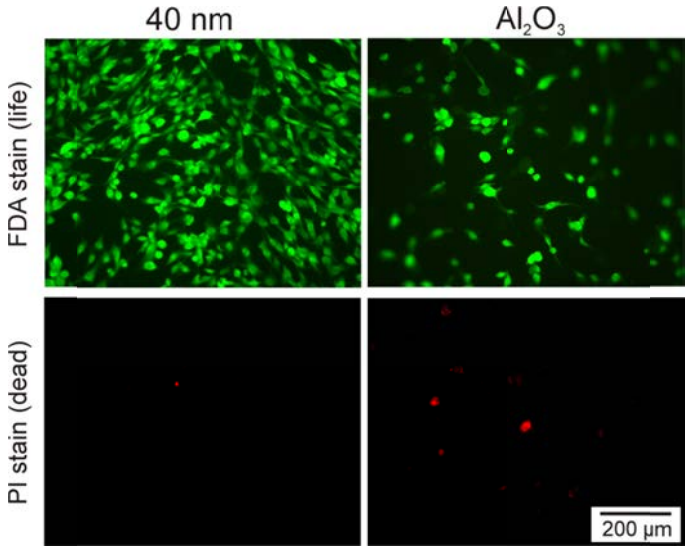
Nanoporous layers of alumina prepared by electrochemical oxidation are shown to achieve improved short-term cell attachment and activity compared to a plain alumina surface. In addition to topology variation, the control of nanoporous structure may trigger a local variation of electrostatic surface properties 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 adherence 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 nm as well as alumina reference.

B.J. Pedimonte, T. Möst, T. Luxbacher, C. von Wilmowsky, T. Fey, K.A. Schlegel, P. Greil Morphological zeta-potential variation of nanoporous anodic alumina layers and cell adherence; *Acta Biomaterialia 10 (2014) 968*

Manufacture of ultrathin, particulate-based ITO layers by rotary printing

Moritz Wegener, Andreas Roosen

Transparent **c**onductive **o**xides (TCOs) as **i**ndium **t**in **o**xide (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.

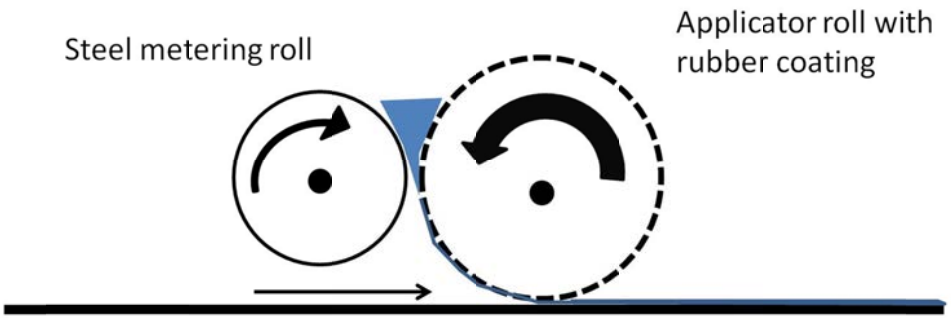


Figure 1: Working scheme of a flexography printer.

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 Ω·cm.

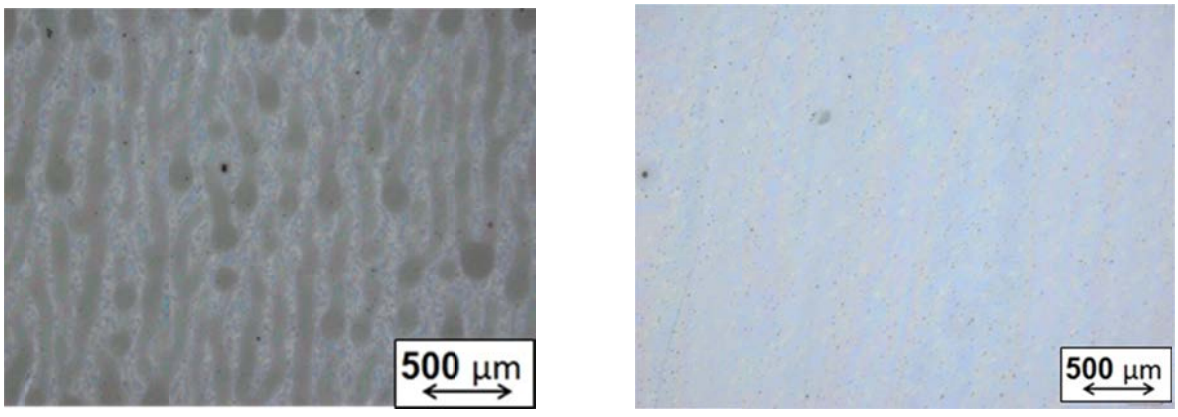


Figure 2: Layer with (left) and without (right) ribs, manufactured by rotary printing.

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 ε 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:

$$K_{xy} = \left(1 - \frac{\varepsilon_x}{\varepsilon_y}\right) x100 = \left(1 - {}^{w}/_{l} \cdot \frac{a^3 \cdot r_a}{b^3 \cdot r_b} \cdot \frac{r_b \cdot sin\frac{B}{2} + b}{r_a \cdot sin\frac{A}{2} + a}\right) x100$$

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

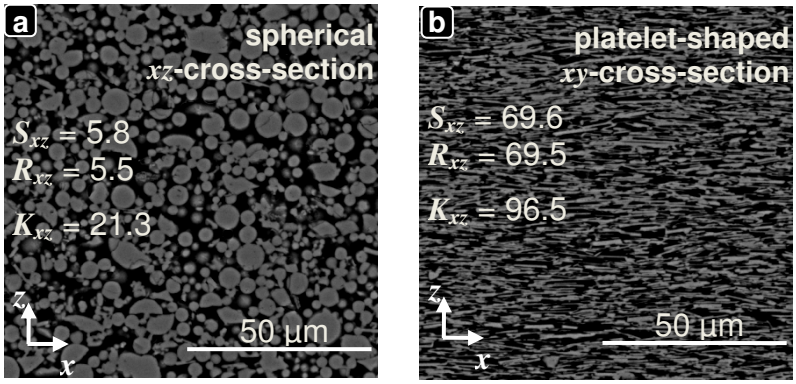


Fig. 1: SEM micrographs of different alumina tapes with **(a)** *spherical particles in* ***xz****-plane and* **(c)** *platelet-shaped particles in* ***xz****-plane*

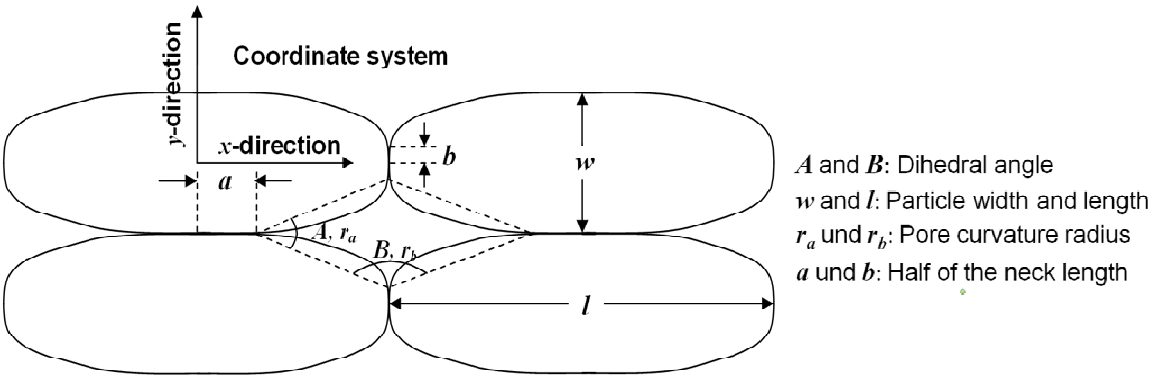


Fig. 2: Representative unit cell to calculate shrinkage anisotropy in ***x****- and* ***y****-direction.*

3. PUBLICATIONS

Papers

(in alphabetical order)

01/13 **A. Arkudas, A. Balzer, G. Buehrer, I. Arnold, A. Hoppe, R. Detsch, P. Newby, T. Fey, P. Greil, R.E. Horch, A.R. Boccaccini, U. Kneser**

Evaluation of Angiogenesis of Bioactive Glass in the Arteriovenous Loop Model

Tissue Engineering Part C: Methods 19(6) (2013) 479-486

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02/13 **R. Bathelt, T. Soller, K. Benkert, C. Schuh, A. Roosen**

Accelerated processing route for KNN based piezoceramics

Advances in Applied Ceramics, 112 (7) (2013) 430-435

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03/13 **G. Bei, B.J. Pedimonte, T. Fey P. Greil**

Oxidation Behavior of MAX Phase $Ti_2Al_{(1-x)}Sn_xC$ Solid Solution

J. Am. Ceram. Soc. 96 [5] (2013) 1359–1362

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04/13 **R. Belli, R. Frankenberger, A. Appelt, J. Schmitt, L.N. Baratieri, P. Greil, U. Lohbauer**

Thermal-induced residual stresses affect the lifetime of zirconia–veneer crowns

Dental Materials 29, 2 (2013) 181–190

DOI: 10.1016/j.dental.2012.11.015

05/13 **T. Fey, M. Götz, P. Greil**

Photoelastic imaging of residual stress distribution in epoxy interface layers of ceramics with periodic building-block structure

Advanced Engineering Materials, 15, 11 (2013) 1099-1104

DOI: 10.1002/adem.201300034

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Three-dimensional printing of SiSiC lattice truss structures

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Processing of preceramic paper and ceramic green tape derived multilayer structures

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Influence of coatings on microstructure and mechanical properties of preceramic paper-derived porous alumina substrates

Journal of Materials Processing Technology 213 (2013) 308–313

DOI: 10.1016/j.jmatprotec.2012.09.005

09/13 N. Kölpin, M. Wegener, E. Teuber, S. Polster, L. Frey, A. Roosen

Conceptional design of nano-particulate ITO inks for inkjet printing of electron devices

J Mater Sci 48 (2013) 1623–1631

DOI 10.1007/s10853-012-6919-8

10/13 T. Kühnlein, A. Stiegelschmitt, A. Roosen, M. Rauscher

Development of a model for the sintering of PZT multilayer ceramics and their dielectric properties

Journal of the European Ceramic Society 33, 5 (2013) 991-1000

DOI: 10.1016/j.jeurceramsoc.2012.10.018

11/13 V. Moreno, D. Hotza, P. Greil, N. Travitzky

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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Synthesis and characterization of laminated Si/SiC composites

Journal of Advanced Research, 4 (2013) 75–82

DOI: 10.1016/j.jare.2012.01.006

13/13 T. Schlordt, S. Schwanke, F. Keppner, T. Fey, N. Travitzky, P. Greil

Robocasting of alumina hollow filament lattice structures

Journal of the European Ceramic Society, 33 (15-16) (2013) 3243-3248

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14/13 S.L. Stares, M.C. Fredel, A. Aragones, E.Y. Gutmanas, I. Gotman, P. Greil, N. Travitzky

PLLA/HA Composite Laminates

Advanced Engineering Materials, *15, 11 (2013) 1122-1124*

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Paper-derived β-TCP

Materials Letters 98 (2013) 161-163

DOI: 10.1016/j.matlet.2013.02.021

16/13 S.L. Stares, M.C. Fredel, P. Greil, N. Travitzky

Paper-derived hydroxyapatite

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DOI: 10.1016/j.ceramint.2013.02.062

17/13 S.L. Stares, A. Kirilenko, M.C. Fredel, P. Greil, L. Wondraczek, N. Travitzky

Paper-Derived Bioactive Glass Tape.

Advanced Engineering Materials 15, 4 (2013) 230–237

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

CFI Ceramic Forum International, 90 (10) (2013) E35-E42

Annual glass week: Andreas Thomsen creates some artistic glass ware

TISSUE ENGINEERING: Part C
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Evaluation of Angiogenesis of Bioactive Glass in the Arteriovenous Loop Model

Andreas Arkudas, MD,[1] Amelie Balzer,[1] Gregor Buehrer,[1] Isabel Arnold,[1] Alexander Hoppe,[2] Rainer Detsch, PhD,[2] Phillipa Newby, MPhys,[3] Tobias Fey, PhD,[4] Peter Greil, PhD,[4] Raymund E. Horch, MD,[1] Aldo R. Boccaccini, PhD,[2,3] and Ulrich Kneser, MD[1]

In this study, the angiogenetic 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 contralateral 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 axial 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".[1] Since then, different strategies and technologies to growth of new tissues have been developed. Most of these strategies are based on the use of different biomaterial matrices 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 melt-derived glass, of the composition (wt%) 45% SiO_2, 24.5% Na_2O, 24.5% CaO, and 6% P_2O_5, is one of the most promising biomaterials that combines biodegradability and bioactivity.[2] Bioactive glasses were first introduced in 1971 by Hench *et al.*,[3] and they have been already used in a variety of clinical applications.[2] Bioglass® is known to be osteoconductive and to promote osteoblast adhesion, growth, and differentiation.[4–6] Bioactive glass is not immunogenic and can control the secretion of cytokines in response to inflammatory stimuli.[9] Angiogenic effects of Bioglass® have been also discussed.[10]

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 bone tissue-engineering model. Nowadays, the majority of tissue engineering approaches are based on the so-called extrinsic vascular pathway.[11] In this case, the construct is vascularized from the periphery; therefore, the implantation of specimens into a site of high vascularization potential is mandatory.[12] Large-bone defects in regions with comprised 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 constructs was first introduced by Erol and Spira in 1979.[13] Recently, our

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Accelerated processing route for KNN based piezoceramics

R. Bathelt[1], T. Soller[1], K. Benkert[1], C. Schuh*[1] and A. Roosen[2]

It is known that the properties of potassium–sodium–niobate (KNN, $K_{0.46}Na_{0.54}NbO_3$) 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 impose 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 on 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.[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 ($K_{0.46}Na_{0.54}NbO_3$) shows appealing initial performance and high Curie and application temperature, provided the densification is high and the stoichiometry is carefully controlled.[2–5] Thus, KNN based ceramics have become the most likely successor of PZT, especially for piezoactuation purposes. 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 piezoconstant d_{33}^* or the permittivity ε is an issue. To overcome these challenges, significant effort has been dedicated to improve processing,[6,7] the performance in general[3,8–12] and to reduce the thermal drift of these properties.[2,13] 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[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, KNNL4T10S6 $[(K_{0.46}Na_{0.54})_{0.96}Li_{0.04}].Nb_{0.84}Ta_{0.10}Sb_{0.06}O_3$,[3] second, KNNL4T11S8, a material doped with more Li and Sb $[(K_{0.46}Na_{0.54})_{0.96}Li_{0.04}].Nb_{0.81}Ta_{0.11}Sb_{0.08}O_3$, and third, KNNL3T19 $[(K_{0.46}Na_{0.54})_{0.96}Li_{0.03}].Nb_{0.81}Ta_{0.19}O_3$.[8] Compared to conventional processing, five times more compositions can be produced and characterised 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

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Rapid Communication

Oxidation Behavior of MAX Phase $Ti_2Al_{(1-x)}Sn_xC$ Solid Solution

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MAX phase $Ti_2Al_{(1-x)}Sn_xC$ solid solution with $x = 0$, 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 accelerated oxidation reaction of A-element was reduced from 900°C ($x = 0$) to 460°C ($x = 1$). SnO_2 was formed at temperatures significantly lower than TiO_2 (rutile) and Al_2O_3. Substitution of A-element in MAX phase solid solution 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, A is an A-group element (from IIIA to VIA), and X is either C or N.[1–3] The structure of the MAX phase (nanolaminates) is composed of MX slabs which are separated by planar layers of the A-element and result in strongly anisotropic characteristics. As the M–A 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.[1]

Excellent oxidation stability of Ti_2AlC was attributed to the formation of an adhesive and protective Al_2O_3 scale on the material surface,[4–9] although Ti–Al intermetallic phases such as Ti_3Al and TiAl do not form a protective oxide scale during high-temperature oxidation.[10] Although less Al is present in MAX phases Ti_3AlC_2 and Ti_2AlC, the high diffusivity of Al along the (0001) basal plane was supposed to induce a thin Al-depleted layer near the oxide scale/substrate interface and facilitates selective oxidation of Al.[9] Originated from the pronounced differences in Ti–C and Ti–A bonding, 211 MAX phase exhibited unusual properties such as deintercalation of A-elements Ga, In, and Sn resulting in extrusion of metal ligaments upon cooling.[11] Although, the driving force for deintercalation of the A-element from the basal (0001) plane of M_2AC is still discussed controversially, *ab initio* calculation of bonding energy as well as migration energy suggest a high mobility of low-melting A-element.[12]

MAX phases M_2AC 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.[13–16] As selective oxidation of the A-element was claimed to govern oxidation behavior of MAX phase Ti_2AC, substitution of Al by a low-melting metal element would be of interest for achieving lower reaction temperatures. Indeed, oxidation of MAX phases Ti_2SnC containing low-melting Sn on the A-position was reported to exhibit accelerated oxidation reaction compared with Ti_2AlC.[17] This study focused on the investigation of oxidation behavior of MAX phase solid solution $Ti_2Al_{(1-x)}Sn_xC$ 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

$Ti_2Al_{(1-x)}Sn_xC$ solid solution specimens were fabricated from reactant powder mixtures consisting of Ti (4.5 μm, 99.4% purity), Al (<45 μm, 99.5% purity), Sn (2 μm, 99.4% purity), and TiC (2 μm, 99% purity) with molar compositions corresponding to $x = 1$ (Ti–Sn–0.9TiC), $x = 0.32$ (Ti–0.8Sn–0.2Al–0.9TiC), $x = 0.57$ (Ti–0.5Sn–0.5Al–0.9TiC), and $x = 0.82$ (Ti–0.2Sn–0.8Al–0.9TiC). A reference specimen was prepared from a Ti_2AlC powder (Kanthal, Sandvik Materials Technology GmbH, Mörfelden-Walldorf, Germany) with $D_{(0.5)} \approx 7$ μm and a phase purity >90%.

The reactant powder mixtures were thoroughly milled for 1 h in a Turbula 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_2SnC samples were sintered at 1200°C under same conditions.

Analyses of the crystalline phase content were conducted by X-Ray Diffraction (XRD) (Kristalloflex, Siemens AG, Mannheim, Germany), operated with monochromatic CuK_α radiation. Material Analysis Using Diffraction (MAUD) software[18] was applied to extract the lattice parameters of the different $Ti_2Al_{(1-x)}Sn_xC_2$ 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 Optic Emission Spectroscopy (ICP-OES; Spectro Analytical Instrument, Genesis SM3, Kleve Germany) to deduce the x-values of the Sn content in the $Ti_2Al_{(1-x)}Sn_xC$ 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 $Ti_2Al_{(1-x)}Sn_xC_2$ solid solution was investigated in the temperature range from 200°C up to 1200°C by exposing the specimens to ambient air atmosphere for each 1 h (Linn High Thermal GMBH, Eschenfelden, Germany). To increase the reaction surface exposed to the oxygen, the bulk specimens were crushed and milled to a

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Thermal-induced residual stresses affect the lifetime of zirconia–veneer crowns

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ABSTRACT

Objectives. The purposes of this study were 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 ($n = 64$) and under cyclic fatigue ($n = 64$). Zirconia copings (YZ Cubes, VITA Zahnfabrik, CTE: alphac = 10.5 ppm/°C) were milled using a Cerec3 InLab (Sirona) machine and sintered to a final thickness of 0.7 mm. Sixty-four copings were sandblasted with 105 μm alumina particles (15 s, 3 cm distance, 45° angle, 0.4 MPa pressure) in order to trigger a tetragonalmonoclinic transformation and to produce a rough surface. The copings were veneered using two different porcelains (VM9, VITA Zahnfabrik, CTE: alphaVM9 = 9.1 ppm/°C, Lava Ceram, 3M ESPE, CTE: alphaLava = 10.2 ppm/°C) so to result in crowns with either high thermal mismatch (+1.4 ppm/°C with VM9) and low thermal mismatch (+0.3 ppm/°C with Lava Ceram). The porcelains were applied by the same operator and fired (VITA Vacumat 4000) according to the firing schedules defined by the manufacturers to a final thickness of 1.4 mm (total crown thickness = 2.1 mm, core/veneer ratio = 0.5). After the last glaze firing the crowns were cooled following a fast (600 °C/min) or a slow (30 °C/min) cooling protocol. The glazed crowns were submitted to a sliding-motion (0.7 mm lateral movement) cyclic fatigue in a chewing simulator (SD Mechatronik) under 20 kg (~200 N load) weight until failure (chipping) ($n = 16$). The other half of the crowns were subjected to a compressive loading test in an universal testing machine (Instron model 4240) 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. For 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 (VM9 group).

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ADVANCED ENGINEERING MATERIALS

COMMUNICATION

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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,[1] as well as for photonic and electromagnetic wave guides, circuits, filters, cavities, laser, antenna, and absorbers.[2] 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.[3] 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.[4] The stresses may be caused by thermal expansion mismatch between dissimilar constituents, e.g., matrix and reinforce-ment 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.[5] Residual interface stresses generated by thermal expansion and elastic misfit 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.[4] 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.[6] 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,[7,8] fiber-reinforced ceramic matrix composites,[9] and laminar composites.[10] Superposition of interface stresses and applied external loading stress may give rise for 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.[11]

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.[12] Non-contact, non-destructive photoelastic measurements are widely used for stress analysis in glass products even of complex shape,[13] in prosthetics,[14] as well as for analysis of fracture processes.[15] Illuminated by polarized light fringe patterns that are related to the difference in principal stresses in a plane normal to the light propagation direction (isochromatics)[16] 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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Rapid communication

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™) from Si/SiC/dextrin powder blends. After printing the C/Si/SiC preforms were infiltrated with a liquid silicone resin for transient shape stabilization. The green bodies were pyrolyzed at 1000 °C in nitrogen atmosphere resulting in a residue with a porosity of ~41%. 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.

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 $m^{0.5}$ 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 (LSI-process) offers the possibility to near net-shape manufacturing of dense SiSiC composites at relative low temperatures and reasonable costs [23]. According to the LSI-process, the REFEL-process and the SILCOMP-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 of Si during solidification may lead to residual microstresses in the SiSiC composites [4]. 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].

The carbonaceous preform is commonly prepared by slip casting or cold pressing using either graphite or a mixture of carbon and α-SiC powder [14]. Macro-structures with complex geometry were fabricated by Solid-Free Form (SFF) technologies from Computer-Aided Design (CAD) data. In three-dimensional printing (3DP™) process solid objects are fabricated by layered printing, in which the sliced 2D profile of a CAD model is printed on a fresh layer of powder via deposition of a suitable binder [15]. Moon et al. [16] fabricated porous carbonaceous preforms using glassy carbon powders of 45–105 μm sizes and an acetone-based furfuryl resin binder as a printing solution. After pressureless reactive infiltration at 1450 °C in nitrogen atmosphere, a SiSiC composite with a coarse-SiC grain structure was formed [16]. Travitzky et al. [18] produced SiSiC composites from a mixture of SiC and starch–cellulose powders by 3D-printing. Dense SiSiC was obtained by subsequent pyrolysis and pressureless liquid silicon melt infiltration [18]. Yin et al. [19] built $TiAl_3/Al_2O_3$ composites by reactive infiltration of an Al melt into a porous TiO_2 preform which was prepared by indirect three-dimensional printing. Nan et al. [20] reported the near-net-shape manufacturing of Ti_3SiC_2-based ceramics by three-dimensional printing combined with liquid silicon infiltration. Schlier et al. [3] created a macrocellular lattice structure of SiSiC ceramics suitable for air–fuel mixture formation as used in automotive applications. Unbonded precursor powder served as support for ligaments with a thickness of 1.5 mm and a spacing distance exceeding 4.4 mm.

The purpose of the present work was to study the effect of Si-content on the microstructure and mechanical properties of Si–SiC composites containing a high Si fraction > 50 vol% fabricated

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Processing of preceramic paper and ceramic green tape derived multilayer structures

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Multilayer laminates with gradients in material composition and porosity were fabricated by the combination of ZrO_2, Al_2O_3–ZrO_2 and Al_2O_3–$MgAl_2O_4$ preceramic papers with MgO–$MgAl_2O_4$, Al_2O_3 and $MgAl_2O_4$ ceramic green tapes. A ZrO_2–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 5 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, slags, fluxes and corrosive atmospheres.[1–3] Degradation of refractories during service in harsh environment is a complex phenomenon and the overall wear rate of refractories is dependent on the contributions of all degradation mechanisms involved.[1,4] Though carbon bonded refractories exhibit superior degradation stability,[1,3] 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 thermal shock resistant 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 lamination 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 65%.[5–8] 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%.[9,10] The tape sintering shrinkage and microstructure formation can be tailored in a wide range by applying multimodal powder mixtures.[11,12] 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 electroceramic components like capacitors, inductors, high integrated circuits and actuators.[10,13–16] 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-plasticiser system and the particles of two 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.[17] Mismatch in the layers' shrinkage behaviour, however, can result in constrained sintering and give rise to an increased residual porosity.[18,19] 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.[20–22]

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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 cellulosic fibers and organics, porous preceramic paper-derived alumina substrates were post-treated via two different coating routes using silica suspension or methylphenylvinylhydrogen polysiloxane. Sintering of the alumina-filled preceramic papers in air at 1600 °C for 2 h resulted in a non-uniform distributed open porosity ranging from 23 to 26%. After coating and infiltration, all samples were additionally heat treated up to at 1500 °C for 2 h. Thermal analysis (DTA/TG) was applied to determine the pyrolysis temperature of polysiloxane. 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 polysiloxane coated samples the mechanical strength steadily increases after repeated impregnation steps, reaching ~350 MPa.

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 pulp fibers 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 a body and allowing the mix on a relatively fine scale is the infiltration of powder compacts with suitable liquid media. After this process, the sample can subsequently be heated to obtain a dense multiphase component.

Lan and Xiao (2009) used the infiltration process for coating metal substrates. During this process, a porous substrate can be infiltrated by a particle suspension under an applied pressure. Thereafter, infiltrated particles can consolidate inside the porous structure in order to fill the open pores and decrease the porosity.

Lee et al. (2011) developed an infiltration technique to densify a SiC matrix in a high performance SiC_f/SiC composite. The slurry infiltration process was also used by Liu and Miao (2005) to produce highly porous MgO-doped alumina ceramics. In this case, well-dispersed alumina slurry was employed to infiltrate the pore space in polystyrene bead compacts.

Kern 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

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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 process window of the printer used 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 semiconducting layer and the gate electrode as well as the dielectric layer is formed by a thermally oxidized silicon wafer. The electron device assembled shows the typical FET characteristic 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 electron devices, the direct printing of electronic bonds on circuits or the manufacture of "gene 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 piezo 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 the understanding of 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_{Re} number and the Weber number N_{We} of the ink:

$$N_{Re} = \frac{va\rho}{\eta} \tag{1}$$

$$N_{We} = \frac{v^2 a\rho}{\sigma} \tag{2}$$

where v is the velocity, a is a characteristic dimension, i.e., the radius of the printing orifice, and ρ, η, and γ 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 Ohnesorge number Oh [10, 11].

$$Z = Oh^{-1} = \frac{N_{Re}}{\sqrt{N_{We}}} = \frac{\sqrt{a \cdot \rho \cdot \gamma}}{\eta} \tag{3}$$

For inkjet inks, the Z-parameter should lie in between 1 and 14 [10, 11]. If the ink fulfills this condition, then a drop

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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 piezo-electrical 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 behaviour 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.

Keywords: PZT; Sintering; Multilayer; Microstructure-final; 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.[1–3] The application-specific adjustment of the material properties is achieved by the ratio of zirconate to titanate and by doping.[4] Depending on the size of the incorporated ions, a soft or hard PZT is obtained by doping.[5] 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.[4,6]

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.[7,8] 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.[2]

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.[9] For such ceramics the overall sintering process can be described by the liquid phase sintering theory.[10,11] 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.[8] 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.[12] found reactions between Pb and Pd in the temperature range up to 800 °C, which lead to the formation of a thin $PdPbO_2$ layer between the electrode and the ceramic. In contrast, Zuo

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COMMUNICATION

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Dense YSZ Laminates Obtained by Aqueous Tape Casting and Calendering**

By *Veronica Moreno,* Dachamir Hotza, Peter Greil* and *Nahum Travitzky*

Solid oxide fuel cells (SOFC) are high temperature devices aimed to stationary heat and power generation.[1–3] Compared to other fuel cells, SOFC present the highest efficiency, being flexible in relation to fuel characteristics and operation temperature range (700–1000 °C). SOFC devices consist of a dense electrolyte and two porous electrodes.[4–10] The efficiency of the cells is determined by the performance of the electrolyte, which depends in its 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 750 °C comparable to that of YSZ at 1000 °C. However, high cost and instability of ScSZ at temperatures below 600 °C may limit the wide of this material. Doped cerium oxides, like gadolinia-doped ceria (GDC) and samaria-doped ceria (SDC), have a high ionic conductivity, however, they tend to be chemically instable in reducing atmospheres.[15–17]

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.[16] In order to produce 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.[18,19] An alternative technique was reported[20–24] 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 8YSZ 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 (8YSZ, 8 mol% Y_2O_3-stabilized ZrO_2, Sigma–Aldrich) was deagglomerated in deionized water with 1 wt% dispersant (Darvan 821A, Vanderbilt) using ball milling for 24 h. After deagglomeration, binder (Mowilith LDM-6138, 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, Mistler) with a Mylar carrier coated with a silicon layer (G10JRM, Mistler). 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 cm^2 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, Bürkle) was carried out in a steel die. Pressure varying between 16 and 19 MPa was applied for 5 min. The lamination by calendering (CA5, Sumet) was performed at 10–15 MPa with a roll speed of 10 m s^{-1}. Tapes were either 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, Nabertherm) 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 1600 °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}.

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ORIGINAL ARTICLE

Synthesis and characterization of laminated Si/SiC composites

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Abstract Laminated Si/SiC ceramics were synthesized from porous preforms of biogenous 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 laminar structure with successive Si/SiC layers. The produced composites show weight gain of ≈5% after heat treatment in air at 1300 °C for 50 h. The produced bodies could be used as high temperature gas filters as indicated from the permeability results.

Introduction

Porous SiC ceramics 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-Al_2O_3 interfaces between Al_2O_3 bars showed markedly improved fracture resistance for these composites as compared with monolithic Al_2O_3 [5].

Clegg et al. [7] have produced laminated SiC with graphite interface layers. These multilayer 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].

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Robocasting of alumina hollow filament lattice structures

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Abstract

Robotic controlled deposition (robocasting) of an aqueous colloidal α-Al_2O_3 gel for manufacturing of cellular ceramics with periodical lattice structure was investigated. The colloidal gel was loaded with 50 vol% α-Al_2O_3 and exhibits shear-thinning behavior, a shear modulus of 288 kPa and a yield-stress of ~700 Pa. Tubular 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 tubular filaments for long distances (~650 mm). Critical conditions to avoid capillary collapse were discussed by considering bulging stress and pressure distribution within the hollow filament. At short filament length oil infiltration into the capillary driven by capillary suction supports the tubular filament whereas oil flow driven by movement of the tool nozzle causes pressure difference to increase linearly with increasing filament length.

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.[1] 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.[2] Aqueous colloidal gels were applied to manufacture three dimensional space filling monoliths as well as lattice structures even with free spanning filaments.[2,3] 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.[4] Ceramic gels based on silica,[1] alumina,[3,5–8] mullite,[9] lead zirconate titanate,[10] tricalciumphosphate,[11] hydroxyapatite,[12] lead magnesium niobate (PMN),[13] porcelain[14] and barium titanate[15] were successfully applied to robocasting. Applications of the ceramic robocasting process include for example highly porous grid structures for bone restoration,[11,12] catalyst carriers and meshes for filters.[3]

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, $\tau_y > 100$ Pa, $\eta > 10$ Pa s[3]) and shear thinning behavior to enable continuous deposition of filaments with feature sizes down to 100 μm as well as free spanning structures.[5] Gelation of powder suspensions with solids volume loading fractions ranging from 0.3 to 0.6 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 syneresis cellulose derivatives (Hydroxypropylmethylcellulose[5], Ethylhydroxyethylcellulose[3]) were added which give rise for appreciable yield-stress of the particle suspension.[16] 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

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COMMUNICATION

ADVANCED ENGINEERING MATERIALS

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PLLA/HA Composite Laminates**

By Steferson Luiz Stares,* Márcio Celso Fredel, Aguedo 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.[1] 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 surgeries. The medical requirements are mainly related to the biocompatibility and biodegradability of the material.[2,3] In order to attend mechanical requirements, has to be observed: (a) a high initial strength to withstand the stresses during the surgical proceedings of implantation and support the external and physiological loads during the early stage of tissue cicatrization; (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.[4,5] The aim of the present work is to investigate some properties of novel PLLA/HA multilayer composites with the future goal of produce devices for use in bone fracture 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 ($Ca_{10}(PO_4)_6(OH)_2$—Merck, Darmstadt, Germany) as reinforcement. PLLA with an M_w of ≈151 kDa and density of 1.23 g cm^{-3} was supplied in the form of granules (Ø2 × 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., Waltham, 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 20-10-5-0-0-5-10-20 (denoted as "20-0-20") and the second group was formed by a sequence 0-5-10-20-20-10-5-0 (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 200T – Servitec Maschinenservice GmbH, Wustermark, Germany).

1.2. Characterization

Samples were freeze-fractured 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 (ESEM, Quanta 200, FEI, Czech Republic).

Bending strength and elastic modulus 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.

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Paper-derived β-TCP

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ABSTRACT

Porous β-TCP structures were manufactured via novel preceramic paper process. The paper sheets were sintered at 1200 °C for 2 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 compressive strength and compressive modulus of sintered specimens vary between 6.55 and 5.62 MPa and 0.33–0.39 GPa, respectively.

1. Introduction

β-TCP (tricalcium phosphate) is a synthetic bioceramic 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]. 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 porosities for a broad field of applications [3–5]. Preceramic paper is made up of inorganic fibers and loaded with inorganic powders [6]. The processing approach used for the deposition of fibers or their 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 preceramic paper derived β-TCP 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 compressive strength and compressive modulus.

2. Experimental

Preparation of preceramic paper: Preceramic papers loaded with β-TCP spheres and pulp fibers were prepared from dilute aqueous suspensions. β-TCP ($Ca_3(PO_4)_2$) spheres were obtained from Sigma-Aldrich, Steinheim—Germany with a mean particle size of 3 μm and density of 3.14 g cm^{-3}. The aqueous suspension of a pulp mixture containing 0.60 wt% non-refined softwood pulp (Celbi PP, Celulose Beira Industrial (Celbi) 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 4.0 vol% anionic starch ester (Fibraffin A5, Südstärke GmbH, Schrobenhausen, Germany). Preceramic paper sheets were formed on a Rapid Köthen sheet forming device (Haage Laborblattbildner BBS-2, Estanit GmbH, Mühlheim an der Ruhr, Germany). Circular sheets with a diameter of 200 mm were obtained after dewatering under mild vacuum ($<10^4$ Pa). The as-filtrated specimens were dried at 93 °C for 15 min resulting in preceramic paper sheets denoted series A, B, and C. Table 1 summarizes the feedstock composition excluding water. The preceramic paper sheets were sintered in air atmosphere in an electrically heated furnace (HT 16/17, Nabertherm, Lilienthal, Germany). A single step annealing sequence was applied where the temperature was raised with a constant heating rate of 5 °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 600 °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 samples (ρ_{ap}) was determined from weight and volume measurements. The thickness of the samples was measured with a digital dial indicator. Skeletal density ($\rho_{skeletal}$) of the samples was measured by He-Pycnometry (AccuPyc 1330, Micromeritics Instrument Corporation, Norcross, GA, USA). Total porosity of

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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 filler. Pressure loading was applied in order to increase the packing density in the paper sheets. The paper 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 balls test (B3B test) and elastic modulus of sintered specimens vary between 18 and 28 MPa and 0.65–1.53 GPa, respectively.

Keywords: Bioceramic; 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 micro-scopic porosities 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 ($Ca_{10}(PO_4)_6(OH)_2$) 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 (Celbi PP, Celulose Beira Industrial (Celbi) 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 feed-stock suspension, induced by addition of 8.0 vol% anionic starch ester (Fibraffin A5, Sildstärke GmbH, Schroben-hausen, Germany).

Preceramic paper sheets were formed on a Rapid Köthen sheet forming device (Haage Laborblattbildner

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Paper-Derived Bioactive Glass Tape**

By Steferson Luiz Stares,* Alina Kirilenko, Márcio Celso Fredel, Peter Greil, Lothar Wondraczek and Nahum Travitzky

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 33 MPa and 0.30–0.85 GPa, respectively.

The ultimate goal in the area of reconstructive medicine is the structural and functional restoration of tissue 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.[1,2] The main advantage is their high biocompatibility.[3–5] Specially 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.[6–8] 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,[9,10] although some load-bearing applications have also been attempted.[11,12] BaG implants were manufactured by different techniques including: injection molding, extrusion, solvent casting, foam replication method, plasma spraying, 3D-printing between other.[13–20]

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 porosities for a broad field of applications.[21–25] Preceramic paper is made up of inorganic fibers and loaded with inorganic powders.[26] The processing approach used for the deposition of fibers or their mixtures with or without the addition of fillers defines the paper properties.[21] The organic fraction of the paper substrate is burned out during firing in air, leaving a porous ceramic residue.[27]

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) calander, on the fabricated samples properties was explored.

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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 shear forces 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, flat substrates at low cost. In today's coating technology, a wide range of methods are applied to apply thin liquid coatings onto sheet-like substrates or pieces [5].

In today's coating technology, mainly self-metering methods (e.g. doctor blade, roll coating) are applied to apply thin liquid coatings onto substrates. Here the achievable thickness of the coating on the substrate is 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 methods (broad slot dies operated 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 powder suspensions to large-area functional coatings in the nano- or micrometre range, the pre-metered broad slot die method boasts key advantages with regard to controllability and reproducibility.

Tab. 1 Selection of influencing factors on the resulting coating accuracy

Influencing Factor	Details	Coating Method
• Gap accuracy	• Between doctor blade and substrate • Between doctor blade and anilox roll	• Doctor blade coating • Anilox roll coating
• Mechanics	• Doctor blade • True running accuracy of the roll • Shape, filling and release rate of the anilox cells • Pressure	• Roller, doctor blade, anilox roll coating
• Viscosity, surface tension • Contact angle	• Surface condition of the coating fluid	• Roller, doctor blade, anilox roll coating

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

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Proceedings

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Slide ring sealing and method of manufacturing

DE 102011083859 (2013) and WO 2013/045614 (2013)

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

Symposium Organizer: EUROMAT 2013 European Congress and Exhibition on Advanced Materials and Processes, "Nano-Powder and Solution Routes: Synthesis to Materials - Additive Manufacturing and other Near Net Shape Techniques", Sevilla, Spain, 8-13 September 2013

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.

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

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

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

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

A. Roosen

Advances in ceramic green tape technology

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

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

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

DKG Symposium of the Technical Committee Process Engineering "Processes for manufacturing ceramic layers", Erlangen, 3 December 2013

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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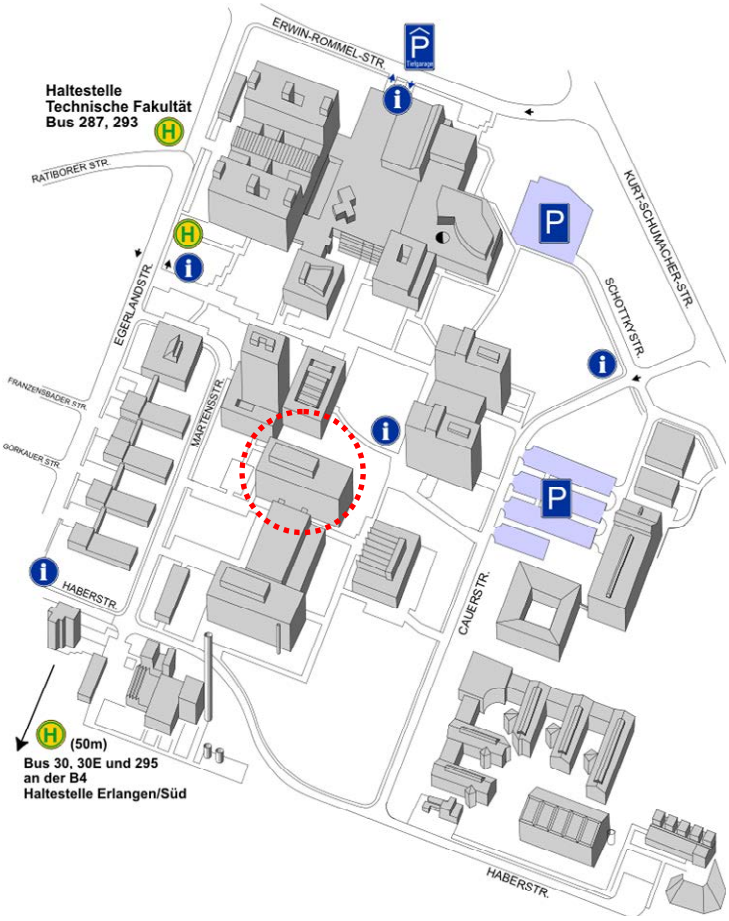
E-mail: ww3@ww.fau.de

Internet: http://www.glass-ceramics.fau.de/



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**".

http://www.glass-ceramics.fau.de/Home/contact.htm

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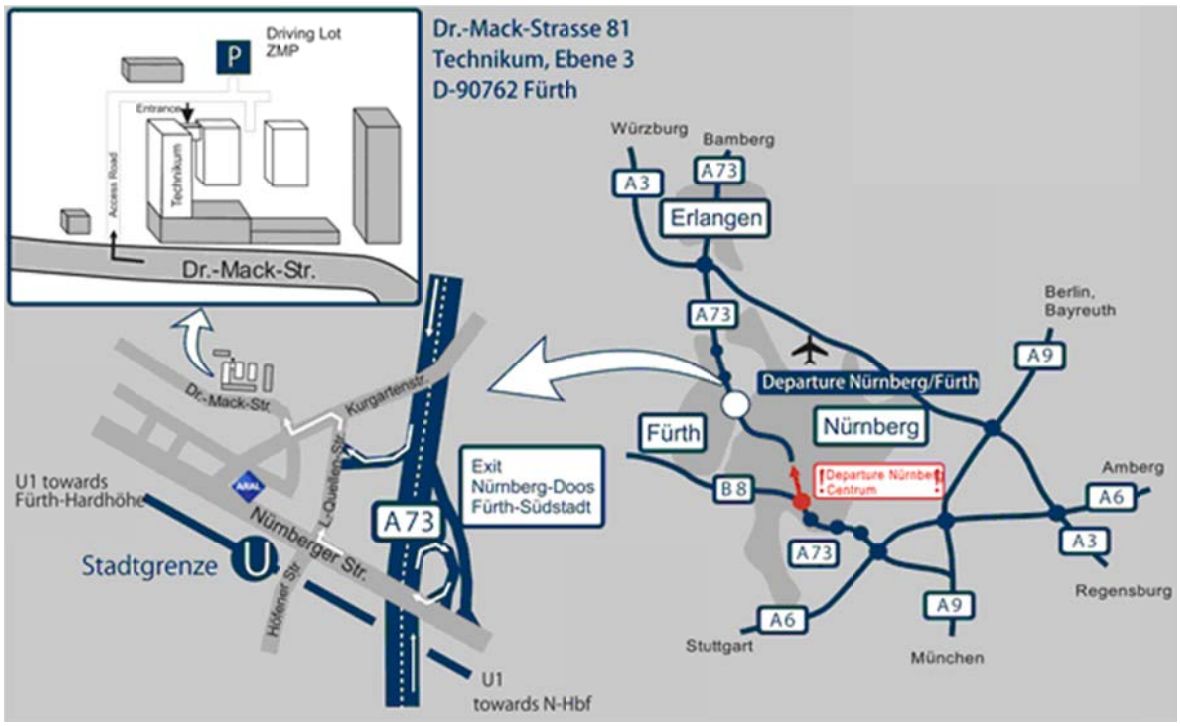

http://www.zmp.fau.de/anfahrt/

6. IMPRESSUM

Prof. Dr. Peter Greil

Dr. Andrea Dakkouri-Baldauf

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