

Developments in Porous, Biological and Geopolymer Ceramics

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Developments in Porous, Biological and Geopolymer Ceramics

*A Collection of Papers Presented at the
31st International Conference on Advanced
Ceramics and Composites
January 21-26, 2007
Daytona Beach, Florida*

Editors

Manuel Brito
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Developments in Porous, Biological and Geopolymer Ceramics

Preface

This issue contains a collection of 32 papers on the topics of porous ceramics, bioceramics and geopolymers that were presented during the 31st International Conference on Advanced Ceramics and Composites, Daytona Beach, FL, January 21–26, 2007. Specifically, papers were submitted from the following 3 Symposia/Focused Sessions:

Symposium on Porous Ceramics: Novel Developments and Applications

This volume of the Ceramic Engineering and Science Proceedings incorporates papers presented at the Symposium on Porous Ceramics: Novel Developments and Applications within the 31st International Conference and Exposition on Advanced Ceramics and Composites, held in Daytona Beach, Florida, January 21–26, 2007. The previous symposia or focused sessions in this series were held in Cocoa Beach, Florida since 2000, which demonstrate a continuous interest in this matter. The objective of this symposia series is to provide an international forum for discussion of research results and developments related to a wide variety of ceramic materials with different applications from the perspective of porosity -its structure, control and functionality- as the important parameter.

The papers in this issue cover several aspects of porosity in ceramics, including development of systems, materials for different applications and their processing and performance. These papers taken together provide up-to-date comprehensive information on the status of the technology and related research activities for the community.

The editor thanks Professor Paolo Colombo, University of Padova and Dr. Gary Crosbie, Ford Motor Company, for their assistance in organizing and managing the symposium, to the authors and reviewers for their hard-work, and to The American Ceramic Society staff for their assistance in publishing this issue.

MANUEL BRITO

National Institute of Advanced Industrial Science and Technology (AIST)

Bioceramics and Biocomposites

The bioceramics papers included in this CESP issue represent a cross-section of current bioceramics research. For example, the use of enzymes to control the synthesis and properties of biomaterials is included along with a discussion of techniques for the rapid formation of apatitic calcium phosphate from calcium carbonate precursors. Also, two papers deal with bioceramic/polymeric composites, including organic/inorganic hybrid bioactive materials and techniques to aid in the attachment of polymeric ligament replacement materials to bone. Another two papers address research in bioactive glasses including the conversion of borate glass to biocompatible phosphates and the engineering of the porosity in bulk phosphosilicate glasses. The links between pores size distributions and biomedical/mechanical properties are also explored as well as the formation of oxide overcoats on geometrically complex bioforms. Thus, this issue highlights some of the most recent advances in the critical areas of bioceramic synthesis, ceramic/polymer hybrids, bioactive glasses, the function of porosity in engineered bone tissue, and biotemplated replicas.

ELDON CASE

Michigan State University

Geopolymers

The 31st International Conference on Advanced Ceramics and Composites is the fourth occasion in which a focused session on geopolymers was hosted by The American Ceramic Society (ACerS). The first took place in 2003 during the ACerS 105th Annual Meeting in Nashville, Tennessee; the second session was part of ACerS 106th Annual Meeting in Indianapolis, and the third was part of the ACerS 107th Annual meeting held in Baltimore, MD, 2005. Papers from these geopolymer focused sessions were published in the ACerS Ceramic Transactions Series, Volumes 153, 165 and 175.

In 2007 the Geopolymer Focused Session was moved to the 31st International Conference and Exposition on Advanced Ceramics and Composites, leading to the papers published in this issue of the Ceramic Engineering and Science Proceedings. In this focused session there were 22 speakers during the one and a half day focused session and the resulting 13 papers are published in this issue. This relatively large, international gathering of geopolymer researchers was directly due to the generous financial support for speakers, provided by the US Air Force Office of Scientific Research through Dr. Joan Fuller, Program Director of Ceramic and Non-Metallic Materials, Directorate of Aerospace and Materials Science.

WALTRAUD M. KRIVEN

University of Illinois at Urbana-Champaign

Introduction

2007 represented another year of growth for the International Conference on Advanced Ceramics and Composites, held in Daytona Beach, Florida on January 21-26, 2007 and organized by the Engineering Ceramics Division (ECD) in conjunction with the Electronics Division (ED) of The American Ceramic Society (ACerS). This continued growth clearly demonstrates the meetings leadership role as a forum for dissemination and collaboration regarding ceramic materials. 2007 was also the first year that the meeting venue changed from Cocoa Beach, where it was originally held in 1977, to Daytona Beach so that more attendees and exhibitors could be accommodated. Although the thought of changing the venue created considerable angst for many regular attendees, the change was a great success with 1252 attendees from 42 countries. The leadership role in the venue change was played by Edgar Lara-Curzio and the ECD's Executive Committee, and the membership is indebted for their effort in establishing an excellent venue.

The 31st International Conference on Advanced Ceramics and Composites meeting hosted 740 presentations on topics ranging from ceramic nanomaterials to structural reliability of ceramic components, demonstrating the linkage between materials science developments at the atomic level and macro level structural applications. The conference was organized into the following symposia and focused sessions:

- Processing, Properties and Performance of Engineering Ceramics and Composites
- Advanced Ceramic Coatings for Structural, Environmental and Functional Applications
- Solid Oxide Fuel Cells (SOFC): Materials, Science and Technology
- Ceramic Armor
- Bioceramics and Biocomposites
- Thermoelectric Materials for Power Conversion Applications
- Nanostructured Materials and Nanotechnology: Development and Applications
- Advanced Processing and Manufacturing Technologies for Structural and Multifunctional Materials and Systems (APMT)

- Porous Ceramics: Novel Developments and Applications
- Advanced Dielectric, Piezoelectric and Ferroelectric Materials
- Transparent Electronic Ceramics
- Electroceramic Materials for Sensors
- Geopolymers

The papers that were submitted and accepted from the meeting after a peer review process were organized into 8 issues of the 2007 Ceramic Engineering & Science Proceedings (CESP); Volume 28, Issues 2-9, 2007 as outlined below:

- Mechanical Properties and Performance of Engineering Ceramics and Composites III, CESP Volume 28, Issue 2
- Advanced Ceramic Coatings and Interfaces II, CESP, Volume 28, Issue 3
- Advances in Solid Oxide Fuel Cells III, CESP, Volume 28, Issue 4
- Advances in Ceramic Armor III, CESP, Volume 28, Issue 5
- Nanostructured Materials and Nanotechnology, CESP, Volume 28, Issue 6
- Advanced Processing and Manufacturing Technologies for Structural and Multifunctional Materials, CESP, Volume 28, Issue 7
- Advances in Electronic Ceramics, CESP, Volume 28, Issue 8
- Developments in Porous, Biological and Geopolymer Ceramics, CESP, Volume 28, Issue 9

The organization of the Daytona Beach meeting and the publication of these proceedings were possible thanks to the professional staff of The American Ceramic Society and the tireless dedication of many Engineering Ceramics Division and Electronics Division members. We would especially like to express our sincere thanks to the symposia organizers, session chairs, presenters and conference attendees, for their efforts and enthusiastic participation in the vibrant and cutting-edge conference.

ACerS and the ECD invite you to attend the 32nd International Conference on Advanced Ceramics and Composites (<http://www.ceramics.org/meetings/daytona2008>) January 27 - February 1, 2008 in Daytona Beach, Florida.

JONATHAN SALEM AND DONGMING ZHU, Volume Editors
*NASA Glenn Research Center
 Cleveland, Ohio*

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Porous Ceramics

HIERARCHICAL POROSITY CERAMIC COMPONENTS FROM PRECERAMIC POLYMERS

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ABSTRACT

Cellular SiOC ceramic components with a porosity > 70 vol% and cell size dimension ranging from 10 μm to a few mm approximately have been developed using a silicone resin. These cellular materials have been further functionalized by developing a high specific surface area (SSA), which is useful for applications such as absorbers or catalyst supports. Several approaches were followed to achieve this goal, including controlling the heating process to retain the transient porosity, depositing a SiO_2 -based meso-structured coating on the surface of the SiOC cellular material, or etching the foams in HF. The samples were characterized using various techniques, including SEM, high resolution and analytical TEM methods and N_2 adsorption-desorption. The SSA value produced varied according to the processing strategy followed.

INTRODUCTION

Porous ceramic components, in particular cellular ceramics, find use in a very broad range of engineering applications, for instance as filters for molten metal or particulate in gas streams, as catalyst support, as load-bearing lightweight structures or as scaffolds for biomedical applications. Most of these applications have rather strict requirements in terms both of the amount of porosity and of its morphology (cell size, cell window size, interconnectivity between the cells) that can be met by choosing the appropriate fabrication method^{1,2}.

The development of components with hierarchical porosity (micro-, meso- and macro-porosity) is of particular interest because they possess at the same time high permeability and tortuosity of the flow paths (improving mixing and heat transfer), provided by the macro-pores (with a dimension $d > 50 \text{ nm}$), and a high specific surface area (SSA), given by the micro- ($d < 2 \text{ nm}$) and meso-pores ($2 < d < 50 \text{ nm}$). Components with hierarchical porosity can thus be employed in separation, gas storage, removal of pollutants and catalysis applications.

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Preceramic polymers allow the fabrication of cellular ceramics (micro- and macro-cellular foams^{3,4}) as well as of components with high SSA^{5,6}, taking advantage both of the shaping possibilities given by the use of a polymeric material and of the transient porosity that is generated upon pyrolysis. Moreover, functionalization of the porous components is possible, by simply adding suitable fillers to the preceramic polymers, in order to enhance/modify some physical properties (for instance electrical conductivity or magnetic properties)⁷.

In this paper, we briefly discuss the use of several possible approaches for developing a high specific surface area in open-cell macro-porous ceramic components obtained from preceramic polymers.

EXPERIMENTAL

SiOC microcellular foams were prepared using a preceramic polymer (MK Wacker-Chemie GmbH, Germany) and poly-methylmetacrylate (PMMA) microbeads (Altuglas BS, Altuglas International, Arkema Group, Rho (MI), Italy) of nominal size ranging from 10 to 185 μm acting as sacrificial filler⁴. The powders were mixed at a constant weight ratio (20 wt% MK, 80 wt% PMMA) by ball milling for 1 hour and then warm pressed (130 to 180°C, 20 MPa). The warm pressing temperature was adjusted as a function of the PMMA microbeads size, in order to optimize the viscous flow of the molten polymer through the beads and the degree of crosslinking of the preceramic polymer. The green samples were then treated in air at 300°C for 2h (heating rate = 0.5 °C·min⁻¹) in order to burn out the PMMA microbeads and allow for the crosslinking of the preceramic polymer. Macro-cellular foams were produced by direct foaming according to the procedure reported in reference 3. All the foams, in their polymeric stage, were pyrolyzed under nitrogen flow at 1200°C for 2 hours (heating rate = 2 °C·min⁻¹). During pyrolysis, the preceramic polymer samples were subjected to a large volume shrinkage (~50%; about 23% linear shrinkage) due to the polymer-to-ceramic transformation, occurring with the elimination of organic moieties and leading to the formation of an amorphous Si-O-C ceramic.

A mesoporous silica precursor solution was prepared containing tetraethyl orthosilicate (TEOS) as the silica source, and the block copolymer Pluronic F127 (EO₁₀₆-PO₇₀-EO₁₀₆, EO=CH₂CH₂O, PO=CH(CH₃)CH₂O) as the structure-directing agent. A starting solution was prepared by mixing 4 g TEOS and 2.5 g EtOH, and successively adding 0.355 g of a 0.768 M HCl solution. The solution was then stirred for 45 minutes in order to pre-hydrolyze the silane units, and added to a solution containing 12 g EtOH, 1.3 g Pluronic F127 and a 0.057 M HCl solution. This was stirred for further 15 minutes before impregnation into the SiOC foams. The SiOC foams were immersed into the silica solution and withdrawn after 1 minute; excess solution was then removed from the samples with absorbing paper. The impregnated samples were dried in air at ambient temperature for a few days. The impregnated samples were heat treated in order to remove the surfactant from the mesopores and promote further condensation of the silica framework. Calcination was carried out in air at 350°C for 1 hour (heating rate = 1°C·min⁻¹).

SiOC foams were etched using an HF solution (20 vol% in H₂O). Samples were placed in a polypropylene container with the hydrofluoric acid (HF) solution. The weight ratio between the SiOC samples and the HF solution was kept constant to 1:50. The solution was gently stirred at room temperature for 9h, and then the samples were extracted and rinsed with distilled water to remove any residual HF. They were then heated up to 110°C to eliminate the remaining water. Some samples were heat treated in air at 650°C for 2h after pyrolysis (before etching), to remove completely any traces of residual carbon possibly deriving from the incomplete burn out of the PMMA microbeads.

MK silicone resin was mixed (50 wt%) with a liquid poly-dimethyl-siloxane (Rhodorsil RTV 141, Rhodia, France), cast into a mold and, after crosslinking, the sample was pyrolyzed at 1200°C for 2 hours (heating rate = 2 °C·min⁻¹).

The samples were characterized using Scanning Electron Microscopy (SEM, JEOL JSM-6490, Japan), Transmission Electron Microscopy (Philips CM20 FEG microscope, operated at 200 keV), and nitrogen adsorption-desorption (ASAP 2020, Micromeritics; analysis performed at 77 K). Specific Surface Area (SSA) was determined from a BET (Brunauer, Emmet, and Teller) analysis⁸. Pore size distribution was calculated from the adsorption branch of the isotherm through the BJH analysis⁸.

RESULTS AND DISCUSSION

Several approaches can be followed in order to produce porous components using preceramic polymers. Macro-porous cellular components with a large cell size (macro-cellular foams, average cell size from ~250 µm to ~3 mm) or a small cell size (micro-cellular foams, average cell size from ~1 µm to ~200 µm) can be fabricated using different procedures: macro-cellular foams can be obtained from direct foaming or by using polyurethane precursors as expanding agents^{3,9}, while micro-cellular foams are produced with the aid of sacrificial fillers^{4,10}. Additionally, micro- and/or meso-porosity can be developed within cellular ceramics, thus obtaining ceramic components with hierarchical porosity.

Several routes that can be pursued to accomplish this goal are listed in Table I and are addressed in this paper.

Table I. Possible strategies for producing hierarchical porosity ceramic components with high SSA from preceramic polymers, with typical SSA values obtained

| Strategy | SSA (m ² /g) | Reference |
|--|----------------------------|-----------|
| Controlled thermal treatment of preceramic polymers | 400-700* | [5,6] |
| Addition of high SSA fillers | 400-650* | [5,6] |
| Deposition of zeolites | 150-300 | [11] |
| Deposition of meso-porous coatings | 60 | [12] |
| Infiltration of aerogels into foams | 150-220 | [13] |
| Etching | 150-600 | [14] |
| Mixing of preceramic polymers with different characteristics | 20-30 | [15] |

* at 600°C

The first approach listed in Table 1 is the controlled thermal treatment of preceramic polymers. This approach takes advantage of the transient porosity generated upon heat treating a preceramic polymer in the temperature range at which the polymer-to-ceramic conversion occurs (generally 400-800°C)^{16,17}. The build-up of internal pressure in the component, provoked mainly by the decomposition of the organic moieties in the preceramic polymer (with generation typically of CH₄ and H₂ gas) leads to porosity (with pore size typically below 50-100 nm). This (micro)porosity is transient, in that it is eliminated when the pyrolysis temperature leading to the completion of the ceramization process is increased. Figure 1a shows the gas evolution of MK polysiloxane, with its associated weight loss, which is maximum in the polymer-to-ceramic transformation temperature interval. Figure 1b reports the data for the pore volume present in a macro-cellular foam pyrolyzed at various temperatures. It can be seen that with increasing