

Ceramic Engineering & Science Proceedings

Issue 4, 1996

Proceedings of the
20th Annual Conference on
Composites, Advanced Ceramics,
Materials, and Structures—B

January 7-11, 1996
Cocoa Beach, FL

Victor Greenhut
Program Chair

Published by
The American Ceramic Society
735 Ceramic Place
Westerville, OH 43081-6136
Copyright © 1996
The American Ceramic Society

ISSN 0196-6219

Ceramic Engineering & Science Proceedings

Issue 4, 1996

Proceedings of the
20th Annual Conference on
Composites, Advanced Ceramics,
Materials, and Structures—B

January 7-11, 1996
Cocoa Beach, FL

Victor Greenhut
Program Chair

Published by
The American Ceramic Society
735 Ceramic Place
Westerville, OH 43081-6136
Copyright © 1996
The American Ceramic Society

ISSN 0196-6219

W. Paul Holbrook, Executive Director
John B. Wachtman Jr., Editor
Mark Mecklenborg, Director of Publications
Lori A. Kozey, Product Manager
Sarah Godby, Production Assistant

Committee on Publications: Marina R. Pascucci, *chair*; ; Man F. Yan; Richard Haber; Terrence E. Mitchell; Steven Freiman, *ex officio*; Prabhat Gupta, *ex officio*; Richard M. Spriggs, *ex officio*; Timothy M. Robinson, *ex officio*; John B. Wachtman Jr., *ex officio*; W. Paul Holbrook, *ex officio*; Mark Mecklenborg, *ex officio*.

Editorial and Subscription Offices: P.O. Box 6136, Westerville, OH, 43086-6136. Telephone (614) 890-4700; Telex TWX 7101109409; and Telefax (614) 899-6109. Annual subscription rate is \$70 per year member, \$85 per year nonmember; single copies \$32 member, \$40 nonmember (postage outside U.S. \$20 additional for surface delivery, \$50 additional for air delivery). Libraries may call for package pricing. Published five times a year. Printed in the United States of America. POSTMASTER: Please send address changes to *Ceramic Engineering and Science Proceedings*, P.O. Box 6136, Westerville, OH, 43086-6136. Second-class postage paid at Westerville, OH, and additional mailing offices. Allow six weeks for address changes.

CESPDK

Vol. 17, No. 4, 1996

The American Ceramic Society assumes no responsibility for the statements and opinions advanced by the contributors to its publications, or by the speakers at its programs.

Copyright © 1996 by the American Ceramic Society. Permission to photocopy for personal or internal use beyond the limits of Sections 107 and 108 of the U.S. Copyright Law is granted by the American Ceramic Society, provided that the base fee of US\$5.00 per copy, plus US\$.50 per page, is paid directly to the Copyright Clearance Center, 222 Rosewood Dr., Danvers MA 01923, USA. The fee code for users of the Transactional Reporting Service for *Ceramic Engineering and Science Proceedings* is 0196-6219/96 \$5.00+\$.50. This consent does not extend to other kinds of copying, such as copying for general distribution, for advertising or promotional purposes, or for creating new collective works. Requests for special photocopying permission and reprint requests should be addressed to the Director of Publications, The American Ceramic Society, P.O. Box 6136, Westerville, OH 43086-6136.

Each issue of *Ceramic Engineering and Science Proceedings* includes a collection of technical articles in a general area of interest. These articles are of practical value for the ceramic industries and the general public. The issues are based on the proceedings of a conference. Both American Ceramic Society and non-Society conferences provide these technical articles. Each issue is organized by an editor who selects and edits material from the conference proceedings. The opinions expressed are entirely those of the presentors. There is no other review prior to publication.

Table of Contents

20th Annual Conference on Composites, Advanced Ceramics, Materials, and Structures — B

Introduction

CMCs: Research in Europe and the Future Potential of CMCs in Industry	3
Marcel H. Van de Voorde and Martin R. Nedele	

Fibers and Tows

Stability of Polycrystalline Nextel 720 Fiber	25
Gopal Das	

Mechanical and Structural Analysis of Silicon Carbide Fiber Hi-Nicalon Types	35
M. Takeda, J. Sakamoto, A. Saeki, and H. Ichikawa	

Fiber Strength with Coatings from Sols and Solutions	43
Randall S. Hay, Dennis Petry, and Emmanuel Boakye	

Porous Aluminum Oxide and Lanthanum Phosphate Fiber Coatings	53
E. Boakye, M.D. Petry, and R.S. Hay	

Thermomechanical Behavior of Advanced SiC Fiber Multifilament Tows	61
Hee Mann Yun and James A. DiCarlo	

Characterization of Fiber Materials Using Metallographic and Image Analysis Techniques	68
Matthias Hoffmann and David J. Diaz	

Processing

Impregnation Molding of Particle-Filled Preceramic Polymers into Fiber Preforms	79
Merve Erdal and Selçuk Güçeri	

Method for Reinforcing Threads in Multilayer Composite Tubes and Cylindrical Structures	90
G.R. Romanoski and T.D. Burchell	
Influence of Fiber Lay-Up Sequence on Mechanical Properties of SiC(f)/SiC Composites.....	98
Dileep Singh, Jitendra P. Singh, and Manish Sutaria	
Processing, Properties, and Microstructure of HVR Nicalon Fiber Cloth-Reinforced Zirconium Phosphate Composites.....	110
Barry A. Bender, Roy J. Rayne, Todd L. Jessen, and Scott Browning	
Fabrication and Properties of Dense Silicon Carbide Matrix Composites	118
S. Suyama, T. Kameda, and N. Amiji	
Microstructure and Interface Characteristics of Alumina-Zirconia Composites	125
R.F. Yttergren, Z.-K. Liu, and D.J. Rowcliffe	
Fabrication and Mechanical Properties of Si₃N₄-Based Composites with Layer Structure.	131
Yasuhiro Shigegaki, Manuel E. Brito, Kiyoshi Hirao, and Motohiro Toriyama	
The Multi-Fracture Response of Cross-Ply Ceramic Composites. . .	139
D.L. Erdman and Y.J. Weitsman	
Modeling of Flexural Behavior of Continuous-Fiber Ceramic Composites	147
S. Raghuraman, E. Lara-Curzio, and M.K. Ferber	
Low-Cycle Tensile Fatigue Behavior of an SiC/SiC Composite.	157
Ö. Ünal	
Laminated Matrix Composites: A New Class of Materials	166
W. Jack Lackey, Sundar Vaidyaraman, and Karren L. More	
Low-Cost, Near-Net-Shape Ceramic Composites Using Resin Transfer Molding and Pyrolysis (RTMP)	174
W.J. Sherwood, C.K. Whitmarsh, J.M. Jacobs, and L.V. Interrante	
Improvement of Si-Ti(Zr)-C-O Fiber and a Precursor Polymer for High-Temperature CMC.....	184
T. Yamamura, S. Masaki, T. Ishikawa, M. Sato, M. Shibuya, and K. Kumagawa	

Interfaces and Coatings

B₄C Coated Carbon Fiber Reinforced Si₃N₄	195
P.M. Bronsveld, I. Gideonse, A. Van der Heide, S. Guder, J.Th.M. De Hosson, E. Sabouret, J.B. Veyret, and E. Bullock	
Damage Evolution Due to Thermal Shock in a 2-D Woven Fiber-Reinforced CVI SiC Composite	203
James E. Webb and Raj N. Singh	
Carbon-Carbon Composites Joining	211
Liang A. Xue	
Residual Stress and Poisson Expansion Effects on the Fiber-Matrix Interfacial Properties Measured in Fiber Push Tests	225
A.M. Daniel, M.R. Elizalde, J.M. Martínez-Esnaola, and J. Janczak	
Microstructure and Mechanical Response of Lanthanum Phosphate/Yttrium Aluminate and Yttrium Phosphate/Yttrium Aluminate Systems.....	233
Dong-Hau Kuo and Waltraud M. Kriven	
Sol-Gel Coating of Nicalon Fiber Cloths	241
Michael K. Cinibulk	
Rb β-Alumina as an Interface Coating in Oxide CMCs.....	250
S. Sambasivan, J.A. Morris, and W.T. Petuskey	
Interfacial Bonding of Carbon-Coated Glass Fiber Reinforced Cement	258
Chao M. Huang, D. Zhu, C.X. Dong, W.M. Kriven, R. Loh, and J. Huang	
Investigation of Fiber/Matrix Interfacial Mechanical Behavior in Ceramic Matrix Composites by Cyclic Fiber Push-In Testing ...	266
J.I. Eldridge, R.T. Bhatt, N.P. Bansal, and F.A. Olmstead	
Interface Modification During Oxidation of a Glass-Ceramic Matrix/SiC Fiber Composite.....	280
A.M. Daniel, A. Martín Meizoso, K.P. Plucknett, and D.N. Braski	

Mechanical and Elevated Temperature Properties

Hi-Nicalon SiC Fiber Reinforced Glass and Glass-Ceramic

Matrix Composites 291
William K. Tredway

Mullite Fiber Reinforced Reaction Bonded Si_3N_4 Composites 299
T. Saleh, A. Lightfoot, J. Haggerty, and A. Sayir

The Effect of Fiber Loading on the Mechanical Behavior of Unidirectional CFCMCs 307
Todd L. Jessen, Barry A. Bender, and Victor A. Greenhut

Tensile and Interlaminar Shear Evaluation of Du Pont Lanxide CMCs 316
Michael R. Effinger, Dennis S. Tucker, and Terry R. Barnett

Microstructures and Mechanical Properties of Hi-Nicalon Fiber Reinforced Si_3N_4 Matrix Composites 324
K. Nakano, S. Kume, K. Sasaki, and H. Saka

Mechanical Properties and Microstructure of Oxidized SiC/SiC Composites 333
Ö. Ünal, A.J. Eckel, and F.C. Laabs

Mechanisms of Hot Corrosion of a Silicon Carbide Fiber-Reinforced Glass-Ceramic 342
Atul Kumar, Alan G. Fox, and Shaio-Wen Wang

Fracture Energy and Fiber Pullout Process of a Continuous Carbon Fiber Reinforced Silicon Nitride Matrix Composite at Elevated Temperatures 349
T. Miyajima, D. Torikai, and Y. Yamauchi

Elevated Temperature Properties and Performance of Nicalon Reinforced Enhanced SiC Matrix Composites 357
Mehran Elahi, Kenneth Reifsnider, Thomas Dunyak, and Kin Liao

Time Dependence of Oxidation-Induced Microstructural Changes in Nicalon- and Nextel-Reinforced SiC 366
P.F. Tortorelli and K.L. More

ARPA's Low-Cost Ceramic Composites Program

Process Simulation for RTM of Blackglas Matrix/Nextel Fiber Ceramic Composites	377
R. Leek, G. Carpenter, J. Madsen, and T.M. Donnellan	
Curing and Pyrolysis of Blackglas Resins and Composites	386
Roger Y. Leung and Wallace D. Porter	
NMR Study of Redistribution Reactions in Blackglas and Their Influence on Oxidative Study	394
M.A.B. Meador, F.I. Hurwitz, and S.T. Gonczy	
Pyrolysis Behavior of Blackglas Composites	401
J. Annamalai, W.N. Gill, A. Tobin, J. Madsen, and T.M. Donnellan	
Performance of Blackglas Composites in 4000-Hour Oxidation Study	411
S. Campbell, S. Gonczy, M. McNallan, and A. Cox	
Strength of Fabric-Reinforced Blackglas Composites	421
Charles Lei and Frank K. Ko	
Abradability Testing of BN-Nextel/312/Blackglas 3-D Woven Composites and the Effect on Retained Strength	431
Durell Wildman and Pramod Khandelwal	
NDE of Nextel 312 and Nicalon Fiber Reinforced Blackglas Composites	441
Pramod Khandelwal, George Y. Baaklini, Don J. Roth, James R. Bodis, and Richard W. Rauser	
Processing and Performance of SiC/Blackglas CFCCs Using Filament Winding.	449
M.N. Ghasemi Nejhad, M.V. Chandramouli, and A.A. Wereszczak	

Introduction

CMC's Research in Europe and the Future Potential of CMC's in Industry

Marcel H. Van de Voorde
European Union
JRC-Institute for Advanced Materials
P.O. Box 2, 1755 ZG Petten
The Netherlands

Martin R. Nedele
Deutsche Forschungsanstalt für Luft- und Raumfahrt
Institute of Structures and Design
Pfaffenwaldring 38-40, D-70569 Stuttgart
Germany

Abstract

CMC's (Ceramics Matrix Composites) have been developed for high temperature applications in aerospace and military industries. In general, the CMC's should be capable of outperforming the best available superalloys. Great efforts are being given to pinpoint spin-off technologies i.e. applications in modern fossil fuel power plants, gas turbines, petrochemistry etc. In these applications, the CMC's have to operate at temperatures up to 1400°C, in corrosive environments for long durations. These developments will provoke a breakthrough for this new group CMC-materials.

The paper will:

- i) give an overview of the CMC's research in Europe; processing, and characterization of physical, chemical and engineering properties at high temperatures;
- ii) pinpoint the R&D needs to achieve the potential growth;
- iii) review the industrial potentials.

Introduction

Many challenges for improvements and new developments in energy technology and industrial productivity are not met because of the unavailability of structural materials capable of assuring safe and reliable plant operation, at competitive costs. The advanced energy technology requires materials with:

- i) improved erosion - and corrosion resistance,
- ii) better mechanical behaviour,
- iii) higher temperature capability.

A new group of high temperature materials with engineering properties better than the best modern superalloys and metal matrix composites has to be invented. The transport industry has an additional need for light weight materials; with high stiffness and wear resistance. New materials, complementing the use of advanced polymers and carbon composites have to be realized, to meet the severe technical specifications.

Monolithic ceramics offer combined properties of low weight, high temperature strength and

environmental stability. The problem with these ceramic materials is their lack of defect tolerance, leading to the possibility of catastrophic service failure in structural components. This may result in:

- i) air-traffic problems caused by for example; damaged aero engine components and
- ii) in important financial losses due to unforeseen shutdowns in industrial plants.

Improved ceramic processing techniques and toughening of ceramics through the incorporation of particulates, whisker and short fibers result in ceramics with better fracture toughness, but the brittle failure problem still persists.

Long fiber and woven reinforced ceramic composites have the potential to combine chemical resistance, high temperature strength and toughness. CMC's may offer a challenge for the technology of the 21st century in which the modern industry demands a combination of:

- i) high temperature stability for thousands of hours,
- ii) hot and ash corrosion resistance,
- iii) reliable mechanical properties - in the 1200°C to 1600°C temperature range; and
- iv) cost competitiveness, Figs. 1 and 2.

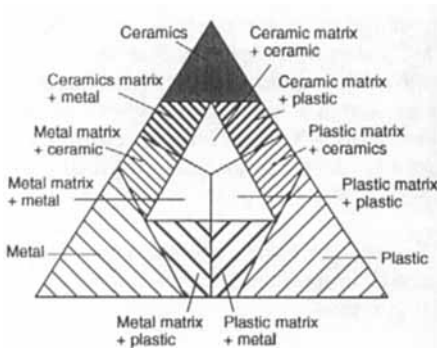


Fig. 1: Different forms of composites

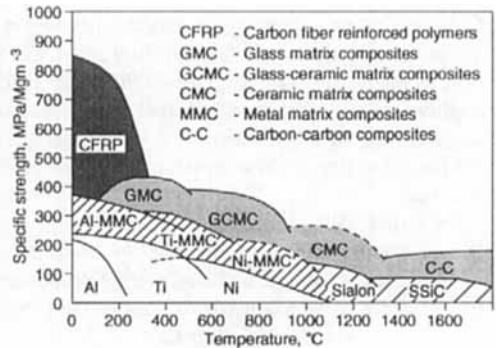


Fig. 2: Strength of structural materials

The ceramic matrix composites "C-SiC" and SiC-SiC" form the leitmotiv through the whole paper, because of the great interest in these materials by industry and the extreme difficulties to introduce them in innovative technologies. Much information available in the paper can easily be used for other ceramic- and carbon-carbon composites.

2. MANUFACTURE OF HIGH TEMPERATURE CMC-MATERIALS

A status report is given on the development of:

- i) high temperature ceramic fiber reinforcements,
- ii) ceramic matrices and
- iii) manufacturing methods for industrial CMC's-materials.

2.1. Advanced Ceramic Fiber Reinforcements

The commercial breakthrough of CMC's strongly depends on the future of high temperature technology/industry. Therefore, continuous ceramic fiber reinforcements should have a variety of corresponding 'high temperature' properties: high stiffness at low and high temperatures, high strength, high thermal-mechanical stability, high oxidation - corrosion resistance, small diameter for CMC fabricability, and low cost for commercial viability.

Potential ceramic fibers of commercial interest for high temperature use in European industry and for research are discussed:

2.1.1. Alumina based fibers: A series of multifilament small-diameter fibers were recently made available commercially: the "Nextal 720" from 3M Company; Sumitomo's "Altex" fiber and a high purity α -alumina fiber "Almax" from Mitsui. The main advantage of oxide fibers is their chemical stability. However, internal grain growth, phase transformation and creep of polycrystalline fibers limit their working temperature to a maximum of 1200°C. Superior creep resistance can be obtained by the use of monofilament large - diameter fibers, for example the single crystal Sapphire fibers from Saphikon and the YAG (Yttrium Aluminum Garnet) fiber from General Atomics. They have potential for CMC service above 1200°C, however their large diameter of 140 μm prohibits weaving and low cost fibers are not available.

2.1.2. Silicon carbide based fibers: New multifilament small-diameter fibers are available: Nippon Carbon's Hi-Nicalon and Ube's Tyranno SiC-fibers are typical representatives. The new Hi-Nicalon fiber gives an increase in creep resistance of up to 200°C compared with standard SiC fibers. The small diameter fiber of roughly 10 μm allows weaving into two and three - dimensional fabrics and the availability in large quantities provides industrial production of high quality CMC-materials. Tyranno fibers, with only 13% oxygen also offers better engineering properties.

Above their thermal stability threshold of about 1100°C degradation rapidly occurs primarily by recrystallisation and crystal growth, and additionally through internal oxidation due to the residual oxygen content. Developments focussed on the reduction of both effects have resulted in a series of new fiber types with improved properties. Nippon "Hi-Nicalon S" fiber; powder derived and sintered α -SiC fiber from Carborundum and a polymer derived and sintered β -SiC fiber from Dow Corning. The new fibers have excellent thermal stability - up to 1200°C, with high elastic modulus of 420 GPa, higher temperature creep and corrosion resistance than the commercially available fibers and are potential candidates for the reinforcement of CMC's in the 1200°C to 1400°C temperature range.

Monofilaments "100 to 140 μm " diameter CVD-SiC fibers are produced. They currently offer the best combination of creep and rupture strength and corrosion resistance. Typical examples are the Textron Specialty Materials (TSM) - SCS-6 and the DRA-Sigma fibers. The newly developed TSM-SCS-2 with 50 μm diameter, is especially promising.

2.1.3. Silicon nitride based fibers: Latest developments are focussed on improved products of silicon nitride and derivatives. Examples are the Tonen corporation Si_3N_4 fibers; the Dow Corning silicon nitride carbide fibers and the single phase Si-B-(N,C) fibers of Bayer A.G. The forecast is that continuous ceramic fibers will be produced with outstanding oxidation resistance (up to 1600°C) and with considerably improved mechanical performance at high temperatures (crystallization resistance up to 1800°C). The commercial availability is long term.