Mechanical Properties and Performance of Engineering Ceramics and Composites VII

Ceramic Engineering and Science Proceedings

Volume 33, Issue 2, 2012

Edited by

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Jonathan Salem

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WILEY

The American Ceramic Society

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A Collection of Papers Presented at the 36th International Conference on Advanced Ceramics and Composites
January 22-27, 2012
Daytona Beach, Florida

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A John Wiley & Sons, Inc., Publication

Published by John Wiley & Sons, Inc., Hoboken, New Jersey.
Published simultaneously in Canada.

Library of Congress Cataloging-in-Publication Data is available.

ISBN: 978-1-118-20588-4
ISSN: 0196-6219

Printed in the United States of America.

10 9 8 7 6 5 4 3 2 1

Mechanical Properties and Performance of Engineering Ceramics and Composites VII

Preface

This volume is a compilation of papers presented in the Mechanical Behavior and Performance of Ceramics & Composites symposium during the 36th International Conference & Exposition on Advanced Ceramics and Composites (ICACC) held January 22–27, 2012, in Daytona Beach, Florida.

This long-standing symposium received presentations on a wide variety of topics providing the opportunity for researchers in different areas of related fields to interact. This volume emphasizes some practical aspects of real-world engineering applications of ceramics such as oxidation, fatigue, wear, nondestructive evaluation, and mechanical behavior as associated with systems ranging from diamond reinforced silicon carbide to rare earth pyrosilicates. Symposium topics included:

- Composites: Fibers, Matrices, Interfaces and Applications
- Fracture Mechanics, Modeling, and Mechanical Testing
- Nondestructive Evaluation
- Processing-Microstructure-Properties Correlations
- Tribological Properties of Ceramics and Composites

Significant time and effort is required to organize a symposium and publish a proceeding volume. We would like to extend our sincere thanks and appreciation to the symposium organizers, invited speakers, session chairs, presenters, manuscript reviewers, and conference attendees for their enthusiastic participation and contributions. Finally, credit also goes to the dedicated, tireless and courteous staff at The American Ceramic Society for making this symposium a huge success.

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Introduction

This issue of the Ceramic Engineering and Science Proceedings (CESP) is one of nine issues that has been published based on content presented during the 36th International Conference on Advanced Ceramics and Composites (ICACC), held January 22–27, 2012 in Daytona Beach, Florida. ICACC is the most prominent international meeting in the area of advanced structural, functional, and nanoscopic ceramics, composites, and other emerging ceramic materials and technologies. This prestigious conference has been organized by The American Ceramic Society's (ACerS) Engineering Ceramics Division (ECD) since 1977.

The 36th ICACC hosted more than 1,000 attendees from 38 countries and had over 780 presentations. The topics ranged from ceramic nanomaterials to structural reliability of ceramic components which demonstrated the linkage between materials science developments at the atomic level and macro level structural applications. Papers addressed material, model, and component development and investigated the interrelations between the processing, properties, and microstructure of ceramic materials.

The conference was organized into the following symposia and focused sessions:

Symposium 1	Mechanical Behavior and Performance of Ceramics and Composites
Symposium 2	Advanced Ceramic Coatings for Structural, Environmental, and Functional Applications
Symposium 3	9th International Symposium on Solid Oxide Fuel Cells (SOFC): Materials, Science, and Technology
Symposium 4	Armor Ceramics
Symposium 5	Next Generation Bioceramics

Symposium 6	International Symposium on Ceramics for Electric Energy Generation, Storage, and Distribution
Symposium 7	6th International Symposium on Nanostructured Materials and Nanocomposites: Development and Applications
Symposium 8	6th International Symposium on Advanced Processing & Manufacturing Technologies (APMT) for Structural & Multifunctional Materials and Systems
Symposium 9	Porous Ceramics: Novel Developments and Applications
Symposium 10	Thermal Management Materials and Technologies
Symposium 11	Nanomaterials for Sensing Applications: From Fundamentals to Device Integration
Symposium 12	Materials for Extreme Environments: Ultrahigh Temperature Ceramics (UHTCs) and Nanolaminated Ternary Carbides and Nitrides (MAX Phases)
Symposium 13	Advanced Ceramics and Composites for Nuclear Applications
Symposium 14	Advanced Materials and Technologies for Rechargeable Batteries
Focused Session 1	Geopolymers, Inorganic Polymers, Hybrid Organic-Inorganic Polymer Materials
Focused Session 2	Computational Design, Modeling, Simulation and Characterization of Ceramics and Composites
Focused Session 3	Next Generation Technologies for Innovative Surface Coatings
Focused Session 4	Advanced (Ceramic) Materials and Processing for Photonics and Energy
Special Session	European Union – USA Engineering Ceramics Summit
Special Session	Global Young Investigators Forum

The proceedings papers from this conference will appear in nine issues of the 2012 Ceramic Engineering & Science Proceedings (CESP); Volume 33, Issues 2-10, 2012 as listed below.

- Mechanical Properties and Performance of Engineering Ceramics and Composites VII, CESP Volume 33, Issue 2 (includes papers from Symposium 1)
- Advanced Ceramic Coatings and Materials for Extreme Environments II, CESP Volume 33, Issue 3 (includes papers from Symposia 2 and 12 and Focused Session 3)
- Advances in Solid Oxide Fuel Cells VIII, CESP Volume 33, Issue 4 (includes papers from Symposium 3)
- Advances in Ceramic Armor VIII, CESP Volume 33, Issue 5 (includes papers from Symposium 4)

- Advances in Bioceramics and Porous Ceramics V, CESP Volume 33, Issue 6 (includes papers from Symposia 5 and 9)
- Nanostructured Materials and Nanotechnology VI, CESP Volume 33, Issue 7 (includes papers from Symposium 7)
- Advanced Processing and Manufacturing Technologies for Structural and Multifunctional Materials VI, CESP Volume 33, Issue 8 (includes papers from Symposium 8)
- Ceramic Materials for Energy Applications II, CESP Volume 33, Issue 9 (includes papers from Symposia 6, 13, and 14)
- Developments in Strategic Materials and Computational Design III, CESP Volume 33, Issue 10 (includes papers from Symposium 10 and from Focused Sessions 1, 2, and 4)

The organization of the Daytona Beach meeting and the publication of these proceedings were possible thanks to the professional staff of ACerS and the tireless dedication of many ECD 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 37th International Conference on Advanced Ceramics and Composites (http://www.ceramics.org/daytona2013) January 27 to February 1, 2013 in Daytona Beach, Florida.

MICHAEL HALBIG AND SANJAY MATHUR
Volume Editors
July 2012

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DAMAGE SENSITIVITY AND ACOUSTIC EMISSION OF SIC/SIC COMPOSITE DURING TENSILE TEST AND STATIC FATIGUE AT INTERMEDIATE TEMPERATURE AFTER IMPACT DAMAGE

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ABSTRACT

This paper discusses the tensile resistance of an impact-damaged SiC/SiC based ceramic composite. As-received and impact-damaged specimens were subjected to static fatigue tests at 650°C and 450°C and to monotonous tensile tests at room temperature. Damage induced during the tensile and fatigue tests was characterized by the evolution of linear density of acoustic emission events (AE) during unloading-reloading cycles. Results of tensile tests at room temperature show the material impact insensitivity and the analysis of AE signals has demonstrated efficiency for proper investigation of damage evolution in the impacted specimens. During fatigue at 450°C, 650°C and under 80 MPa, both impact-damaged and as-fabricated samples survived 1000 hours. Residual strengths indicated insensitivity to impact damage. At 650°C, damage evolution seemed to slow down from matrix self-healing.

INTRODUCTION

Due to low weight/mechanical properties ratios and high temperature strength, ceramic matrix composites (CMCs) are very attractive candidates for civil aircrafts applications. For these applications, resistance to foreign object damage (FOD) is a key issue to insure structural reliability in service. In the literature, a few authors have investigated the FOD response of 2D woven CMC[1-5]. They have shown that low energy impact was equivalent to quasi-static indentation and that a conical damage zone was created.

Meanwhile, investigations by Ogi *et al.*[6] and Herb *et al.*[7] on 3D woven CMC have shown that tri-dimensional fibre architectures prevent the material from delamination so that the damaged cone remains limited and well delineated. After indentation (*i.e.* ballistic[1-6] or quasi-static impact tests[7]), residual strengths were measured using tensile or flexural tests at room temperature[1-4,6,7].

Few works on the effect of impact damage on composite lifetime under fatigue at elevated temperature have been reported. Recently, Verrilli *et al.*[5] have performed cyclic fatigue tests on 2D cross ply SiC/SiC composite at 1316°C after impact tests at 1200°C. They have observed that lifetime decreases tremendously with increasing impact energy. After high energy impact damage the average lifetime was 40 times smaller than that obtained after low energy impact tests. However, impact damage evolution during fatigue has not been studied in real time.

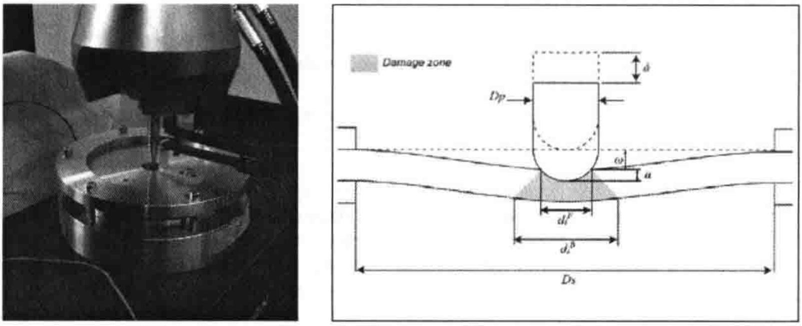
This paper investigates the evolution of impact damage during fatigue at high temperature on a 3D SiC/SiC composite using acoustic emission signals and the sensitivity to impact damage. Acoustic emission data were analyzed using homemade software that determines the spatial distribution of AE events during the tests. As-fabricated and impact-damaged specimens were tested at room and at high temperatures (*i.e.* 450°C and 650°C).

EXPERIMENTAL PROCEDURE

Material and specimen preparation

The SiC/SiC composite investigated (manufactured by Snecma Propulsion Solide - SAFRAN Group (Bordeaux, France)) was made of an interlock preform of plies of 0/90 yarns woven in a 8 HSW pattern[8] with a self-healing [Si-B-C] matrix. The fibres were coated by a PyC layer deposited via chemical vapour infiltration[8]. This 3D fibrous preform (Guipex® preform) improves the through thickness properties[9]. Yarns contain 500 SiC Nicalon fibres. The fibre volume fraction was between 35% and 40% and the porosity volume fraction was around 12%. A barrier coating protected sample surface against oxidation.

Rectangular test specimens were machined out of 1.8 mm thick panels: specimens' dimensions were 24 mm in width and 200 mm in length. Impact damage was generated by quasi-static indentation by Herb *et al.*[7]. A hemispherical steel punch with a 9 mm diameter was used. The specimens were clamped as shown on Figure 1.



(a) (b)

Figure 1. (a) Quasi-static indentation indentation with D_s=18 and D_p= 9 mm. (b) Schematic view of specimen loading (from Herb *et al.*[7]).

Figure 2 shows the cone crater that had been created. Breakage of fibre bundles was observed on back side (Figure 2a), and the impact side (Figure 2b). On the impact side, the sample displayed a neat circular mark. Images of the impact and back sides were post-treated using the Image J software, in order to measure cone area on each side of samples. It was found to be about 33 mm^2 on the front side which is equivalent to a hole with a diameter of 6.5 mm. On the back side, the damaged area was about 85 mm^2 which is equivalent to a hole with a 10.5 mm diameter. The cone crater was sharply delineated, which can be attributed to the 3D fibrous architecture of material and the resulting absence of delamination.

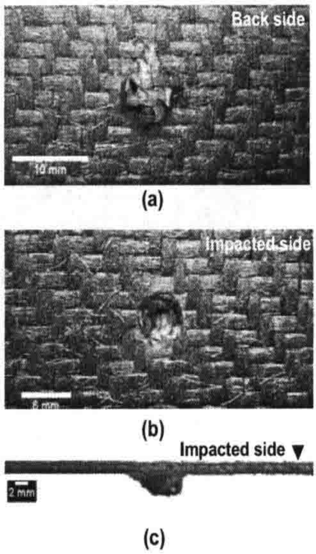


Figure 2. Optical photographs of the SiC/SiC specimens after quasi static indentation: (a) back side (d_i^B= 10.5 mm) (b) impacted side (d_i^F = 6.5 mm) (c) thickness view.

Post-indentation mechanical testing

Static fatigue and tensile experiments were performed using a 25kN uniaxial pneumatic tensile loading machine with one direction of fibres parallel to the loading direction. Specimen elongation was measured using an extensometer (gauge length = 32 mm).

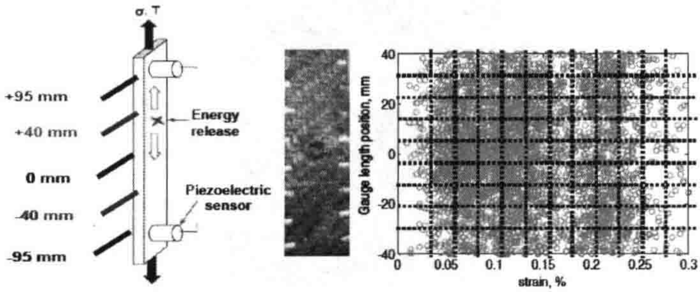
Static fatigue tests were carried out at 450°C and 650°C under air, with uniform temperature in the gauge length (cold grips). Specimens were heated up to the test temperature at a rate of 20°C/min. The load was applied after 2 hours, when the gauge length was expected to be at the test temperature. During the static fatigue tests, specimens were first loaded at a constant rate of 1200 N/min up to the test load corresponding to 80 MPa, close to the elastic limit of composite.

Tensile tests under monotonous loading were performed at room temperature, on impacted specimens (post-impact strength) and on specimens after 1000 hours of static fatigue (residual strength). For all the tests, damage was characterized using periodical unloading-loading cycles (every 12 hours during the static fatigue tests) and also acoustic emission signals. For comparison purposes, a few tests were performed on as-fabricated specimens.

Acoustic emission monitoring

AE was monitored using a MISTRAS 2001 data acquisition system (Euro Physical Acoustics). Two MICRO-80 sensors were positioned directly on the specimen, inside the grips, using vacuum grease with a medium viscosity as a coupling agent. Acquisition parameters were set as follows: pre-amplification 40 dB, threshold 36 dB, peak definition time 50 μs, hit definition time 100 μs, hit lockout time 1000 μs[10,11]. AE signal parameters (amplitude, energy, duration, counts, location...) as well as time, load and strain were measured in real time by the data acquisition system.

As depicted by Figure 3, the zone of interest for the AE events was 80 mm long and located on each side of the mid-plane. Since two sensors only were used, the longitudinal positions of AE origins were determined (see Figure 3a).



(a) (b)

Figure 3. (a) Specimen and Acoustic Emission setting (b) Locations of AE sources in a tensile specimen versus strain. Also shown is the grid defined in order to determine the linear density of counts of acoustic events along specimen axis during tensile tests or static fatigue tests.

AE sources locations were derived using AE wave velocity, that was measured using a pencil lead break procedure: 9100 m/s for both the as-fabricated and the impact-damaged specimens. Morscher *et al.*[12] showed that in ceramic matrix composites wave velocity decreases with increasing stress-induced damage. Wave velocity value was corrected using the attenuation coefficient

$$\gamma = (E/E_0)^{0.5} \qquad (1)$$

E is the secant elastic modulus determined from unloading-reloading hysteresis loops and E_0 is the initial elastic modulus.

AE data were post-treated using dedicated software, which provides linear density along specimen axis of acoustic events during the tests[13]. This analysis of acoustic emission is non-trivial. It is useful to identify the most active zones during the tests.

RESULTS AND DISCUSSION

Effect of impact-damage on tensile behaviour

During the tensile tests, the impacted specimens failed from the mid-plane whereas the as-fabricated samples failed from the upper or lower parts of the gauge length. Figure 4a shows typical tensile curves. For both the as-fabricated and the impact-damaged specimens, the stresses were determined from the net-section of samples: