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This proceedings contain a collection of 72 papers from the Mechanical Properties and Performance of Engineering Ceramics and Composites symposium that was held during the 30th International Conference & Exposition on Advanced Ceramics & Composites, January 22-27, 2006 in Cocoa Beach, Florida.

The papers in this volume address a wide range of topics, presented by an international group of scientists and engineers from government, industry and academia. Technical areas include processing and manufacturing; tribological behavior; carbon-carbon composites, mechanical behavior; ceramic-metal systems; non destructive evaluation and novel characterization techniques; joining and brazing; reliability and analysis; wear and erosion; synthesis, characterization and properties of nitrides, carbides, and borides; fracture and deformation; and design.

I would like to thank and express appreciation for the many volunteers and the staff of The American Ceramic Society, who make this meeting possible. In particular I am indebted to those who attended and participated in the meeting, to the session chairs and organizers, and to those who helped in the review of the manuscripts contained in this volume.

RAJAN TANDON
Introduction

This book is one of seven issues that comprise Volume 27 of the Ceramic Engineering & Science Proceedings (CESP). This volume contains manuscripts that were presented at the 30th International Conference on Advanced Ceramic and Composites (ICACC) held in Cocoa Beach, Florida January 22–27, 2006. This meeting, which has become the premier international forum for the dissemination of information pertaining to the processing, properties and behavior of structural and multifunctional ceramics and composites, emerging ceramic technologies and applications of engineering ceramics, was organized by the Engineering Ceramics Division (ECD) of The American Ceramic Society (ACerS) in collaboration with ACerS Nuclear and Environmental Technology Division (NETD).

The 30th ICACC attracted more than 900 scientists and engineers from 27 countries and was organized into the following seven symposia:

- Mechanical Properties and Performance of Engineering Ceramics and Composites
- Advanced Ceramic Coatings for Structural, Environmental and Functional Applications
- 3rd International Symposium for Solid Oxide Fuel Cells
- Ceramics in Nuclear and Alternative Energy Applications
- Bioceramics and Biocomposites
- Topics in Ceramic Armor
- Synthesis and Processing of Nanostructured Materials

The organization of the Cocoa Beach meeting and the publication of these pro-
ceedings were possible thanks to the tireless dedication of many ECD and NETD
volunteers and the professional staff of The American Ceramic Society.

ANDREW A. WERESZCZAK
EDGAR LARA-CURZIO
General Editors

Oak Ridge, TN (July 2006)
Fracture and Deformation
HIGH-VELOCITY IMPACT RESISTANCE OF ZrB₂-SiC

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Rolla, MO 65409

Jochen Marschall
Molecular Physics Laboratory
SRI International
Menlo Park, CA 94025

ABSTRACT
The high-velocity impact resistance of hot-pressed zirconium diboride with 30 volume percent silicon carbide was studied using a combined experimental and computational approach. Test specimens in the form of 2 mm thick polished disks were impacted with ~0.8 mm diameter tungsten carbide spheres at velocities up to 320 m/s. The intrinsic flexure strength of the specimens was ~1000 MPa. The flexure strength retained by impacted specimens decreased linearly with increasing impact velocity, falling to ~600 MPa at ~290 m/s. Above this threshold velocity, the retained flexure strength fell rapidly, with no measurable retained strength for samples impacted at 320 m/s. The experimental results suggest gradual strength degradation is associated with the formation of shear and sliding faults under the impact zone at moderate impact velocities. The abrupt decrease in strength above 290 m/s is due to cone-crack propagation. Finite element modeling supports the failure mechanism for impact velocities above 290 m/s, but fails to provide insight as to the failure mechanism below this velocity.

INTRODUCTION
Ceramics based on zirconium and hafnium diborides with silicon carbide additions (ZrB₂-SiC and HfB₂-SiC) are candidate materials for the leading edges of hypervelocity atmospheric re-entry vehicles due to their moderate strength, high melting temperature and oxidation characteristics. It is critical to understand the evolution of impact damage in these materials for possible encounters with debris during launch, orbit, or re-entry. A previous impact study was performed on ZrB₂-SiC and HfB₂-SiC ceramics manufactured during the SHARP B1 and B2 flight experiments. The reported strength of the materials from this era was less than ~400 MPa. Recent improvements in processing have lead to intrinsic flexure strengths in excess of 1000 MPa for ZrB₂-SiC materials prepared at the University of Missouri - Rolla. These ceramics have heterogeneous microstructures and fracture toughness values greater than 2.5-3 MPa-m⁰.5 The present work focuses on the strength degradation of this ZrB₂-SiC material as a function of impact velocity with WC projectiles.

Figure 1 is a schematic of the damage expected for a hard sphere impact on a ceramic surface, where the ceramic responds in a classic brittle manner to a predominately elastic stress field. When the projectile makes contact with the specimen, a small contact patch is formed between the surface of the projectile and specimen. Ring
High-Velocity Impact Resistance of ZrB$_2$-SiC

Cracks are formed as concentric circles on the impact surface outside of the contact patch, driven by tensile stresses during loading. Cone cracks start at the surface as ring cracks and penetrate into the substrate when a critical impact load is exceeded. Radial cracks initiate from the contact patch and extend outwards through the ring cracks.

![Schematic representation of impact damage in brittle materials](image)

**Figure 1:** Schematic representation of impact damage in brittle materials. Note that the drawing is not to scale.

In addition to these classic cracking patterns, a quasi-plastic zone can form in the region of high compressive and shear stresses beneath the contact patch, by intergranular microcracking, sliding, and shear fault deformation. A variety of studies have demonstrated that quasi-plastic deformation increases at the expense of cone-crack formation as the microstructure coarsens. In ceramics where ring and cone crack formation dominates the impact damage, failure during flexure testing is caused by radial or cone cracks propagating through the ring crack system. In materials where quasi-plastic deformation dominates impact damage, failure during flexure testing occurs by propagation of shear or sliding faults within the contact patch.

The purpose of this paper is to report the effect of high velocity impact on the retained flexure strength of ZrB$_2$-SiC. The flexure strength of ZrB$_2$-SiC was tested after impacts with velocities up to 320 m/s. The observed fracture modes were compared to the behavior of alumina and to a finite element model.

**EXPERIMENTAL PROCEDURE**

Specimens contained 70 vol. % ZrB$_2$ (Grade A, H.C. Starck, Newton, MA) and 30 vol. % SiC (UF-10, H.C. Starck, Newton, MA). Precursor powders were mixed using an attrition mill (Union Process, Akron, OH). Cobalt-bonded WC media and spindle were used to mill the powders in hexane for 2 hours. After milling, the hexane was removed by rotary evaporation (Model Rotavapor R-124, Buchi, Flawil, Germany) at a temperature of 70 °C, a vacuum of 200 Torr, and a rotation speed of 150 rpm.
The milled powder was hot-pressed (Model HP-3060, Thermal Technology, Santa Rosa, CA) in graphite dies lined with boron nitride-coated graphite foil. The powder charge was heated to 1450 °C under vacuum. An isothermal hold at 1450 °C was used to promote the removal of B2O3 from the surface of the ZrB2 particles by vaporization. The length of the hold was determined by the time required for the vacuum in the furnace chamber to return to the nominal vacuum of ~150 mTorr. The powder was then heated to 1650 °C and held until the vacuum again reached ~150 mTorr. Then, the hot-press was back-filled with argon and the temperature was increased to 1900 °C at a rate of 10°C/min. When the die temperature reached ~1800 °C, a uniaxial load of 32 MPa was applied. Billets were pressed for 45 min once the temperature reached 1900°C. After 45 minutes, the hot-press was cooled at ~20 °C/minute to room temperature. The load was removed when the temperature reached ~1700 °C.

The billets were, on average, ~50 mm in height and 32 mm in diameter. The billets were sliced into ~2 mm thick disks and the disks were polished to a surface finish of 1 μm roughness. Each billet yielded around 17 disks. The bulk density of the individual disks was measured according ASTM C373 (Archimedes' technique) using water as the immersing medium. The strength was determined according to ASTM C1499 (ring-on-ring biaxial flexure) while the elastic constants were measured according to ASTM C1259 (impulse excitation). Microstructure was characterized by examining polished cross sections using optical and scanning electron microscopy (SEM).
High-Velocity Impact Resistance of ZrB$_2$-SiC

Impact testing was performed in a gas gun facility at SRI International using tungsten carbide spheres with a diameter of approximately 0.8 mm (1/32 inch). A schematic of the facility is shown in Figure 2 and the physical properties of the WC spheres are given in Table I. Impacts were performed at velocities ranging from 70 to 320 m/s at room temperature. The corresponding kinetic energy of the projectiles ranged from about 0.01 to 0.2 Joules. While numerous shots were made at a variety of impact energies, not all of the tests could be correlated to surface damage. A fraction of the low-velocity impacts created damage regions that were undetectable by either optical or scanning electron microscopy.

<table>
<thead>
<tr>
<th>Type</th>
<th>WC 44A</th>
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<tr>
<td>Composition (mass%)</td>
<td>6% Co – balance WC</td>
</tr>
<tr>
<td>$\rho$ (g/cm$^3$)</td>
<td>14.95</td>
</tr>
<tr>
<td>$v$</td>
<td>0.26</td>
</tr>
<tr>
<td>$E$ (GPa)</td>
<td>690</td>
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<tr>
<td>$H_k$ (Rockwell)</td>
<td>A91</td>
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Manufacturer data; New England Miniature Ball Corporation (www.nemb.com)

RESULTS AND DISCUSSION

Microstructure and Properties of As-Prepared ZrB$_2$-SiC

The mechanical properties and microstructure of over 30 as-prepared disks were examined. The measured properties are summarized in Table II. The average strength of the as-prepared specimens was over 1000 MPa, which is consistent with previous reports using a similar processing route. The measured density and modulus both indicate that the ZrB$_2$-SiC reached nearly theoretical density during hot pressing. This was confirmed by the lack of porosity observed in polished cross sections revealing the typical microstructure of the ceramic (Figure 3). The average grain size was $\sim 5 \mu$m.

<table>
<thead>
<tr>
<th>Material</th>
<th>Density (g/cm$^3$)</th>
<th>Relative Density %</th>
<th>Hardness (GPa)</th>
<th>$E$ (GPa)</th>
<th>$\sigma_f$ (MPa)</th>
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<tr>
<td>ZrB$_2$-30 vol. % SiC</td>
<td>5.41</td>
<td>$\sim 100$ %</td>
<td>24±1</td>
<td>485 ± 11</td>
<td>1026 ± 32</td>
</tr>
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High-Velocity Impact Resistance of ZrB$_2$-SiC

Figure 3: SEM micrograph showing the typical microstructure of as-prepared ZrB$_2$-SiC.

Observed Impact Damage

Figure 4 shows an impact site produced by a WC projectile with a velocity of 318 m/s. The largest ring crack observed for impacts above 300 m/s was approximately 484 µm in diameter. Analysis by energy dispersive x-ray spectroscopy confirmed that fragments of the WC projectile were implanted into the ceramic near the impact site. No evidence of spallation or removal of ZrB$_2$-SiC was observed as a result of impact testing.

Figure 4: SEM micrograph of a ZrB$_2$-SiC surface after impact of a WC projectile at 318 m/s.
High-Velocity Impact Resistance of ZrB₂-SiC

A number of radial cracks (Figure 5a) and ring cracks (Figure 5b) were observed on the impacted surfaces. Even though the cracks are similar in appearance, the radial cracks and ring cracks are caused by different mechanisms and they initiate at different impact velocities. Rings cracks are initiated on the surface of the sample when the critical radial tensile stress is reached while the exact origin of the radial cracks is undetermined. The initiation of ring cracks was found at much lower impact velocities than the radial cracks. Once formed, the radial cracks appeared to propagate through both ZrB₂ and SiC grains near the impact site and begin to deflect around the SiC grains outside ~3.5 ring crack diameters from the initiation.

![Image of radial and ring cracks](image)

**Figure 5:** SEM micrographs showing (a) a radial crack and (b) a ring crack after impact of a WC projectile at 304 m/s with the surface of ZrB₂-SiC.

Mechanical Testing

The Young’s modulus, Poisson’s ratio, and biaxial flexure strength were measured for impacted specimens. The results showed that neither Young’s modulus nor Poisson’s ratio varied as a function of impact velocity for velocities up to 320 m/s (Table III).

**Table III:** Elastic modulus and Poisson’s ratio for ZrB₂-SiC over the impact velocity range.

<table>
<thead>
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<th>Impact Velocity (m/s)</th>
<th>Elastic Modulus (E,GPa)</th>
<th>Poisson’s Ratio (ν)</th>
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<tr>
<td>0</td>
<td>485 ± 11</td>
<td>0.146</td>
</tr>
<tr>
<td>100-200</td>
<td>483 ± 11</td>
<td>0.146</td>
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<tr>
<td>200-250</td>
<td>483 ± 12</td>
<td>0.146</td>
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<tr>
<td>250-320</td>
<td>483 ± 12</td>
<td>0.146</td>
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Prior to testing the ZrB₂-SiC disks, a series of alumina (Al₂O₃) disks were impacted and their strength was measured as a function of impact velocity (Figure 6). The alumina was found to have an as-prepared strength of 416 MPa. The alumina showed ~100 MPa decrease for all impact velocities increased up to ~250 m/s. Above impact
velocities of \( \sim 250 \text{ m/s} \), the alumina disks failed upon impact (retained strength < 1-2 MPa). The failure mechanism in flexure appeared to be a mixed fault failure with no observations of cone cracking in the alumina over the velocity range in this experiment.

Compared to alumina, ZrB\(_2\)-SiC showed a larger drop in strength with increasing impact velocities below the critical threshold of \( \sim 290 \text{ m/s} \). Strength decreased from \( \sim 1026 \text{ MPa} \) to \( \sim 600 \text{ MPa} \) as the impact velocity approached the critical value. At higher impact velocities, the behavior was different than alumina. Between 290 and 320 m/s the retained strength fell rapidly to \( \sim 400 \text{ MPa} \) and failure was thought to initiate at cone cracks. The ZrB\(_2\)-SiC samples had no measurable retained strength at velocities above \( \sim 320 \text{ m/s} \).

![Graph](image)

**Figure 6:** Retained biaxial flexural strength as a function of impact velocity for Al\(_2\)O\(_3\) and ZrB\(_2\)-SiC.

The sharp decrease in the flexure strength of ZrB\(_2\)-SiC at velocities exceeding 290 m/s may be caused by a change in the primary failure mechanism from a possible mixture of shear and sliding faults to failure by cone crack propagation. Initiation of failure at cone cracks was verified for samples above the \( \sim 290 \text{ m/s} \) threshold by optical microscopy.

An FEM model was developed for this impact study. The results of the study support the cone cracking failure mode that is associated with the large strength reduction at velocities greater than \( \sim 290 \text{ m/s} \). These results will be discussed in future manuscripts.
Summary

The effect of high-velocity impacts on the strength of hot-pressed ZrB$_2$-SiC ceramics was studied. The retained flexure strength decreased monotonically as impact velocity increased up to a threshold velocity near 290 m/s. At this threshold, the retained strength dropped rapidly to ~40% of the as-prepared ZrB$_2$-SiC strength (1026 MPa). This rapid decrease in strength is attributed to severe cone crack propagation. Near the impact site, crack deflection wasn’t sufficiently active to retain a higher level of strength; therefore the addition of a third phase with high-energy crack deflection properties may improve the high energy impact resistance of ZrB$_2$-SiC.

References

HIGH TEMPERATURE FATIGUE BEHAVIOR OF MULLITE/ SiC MULTI-COMPOSITE
CRACK-HEALED

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ABSTRACT
Static fatigue behavior of mullite/ 15vol% SiC whisker/ 10vol% SiC particle multi-
composite was investigated at temperatures from 1273 K to 1473 K. Mullite/ 15vol% SiC
whisker/ 10vol% SiC particle multi-composite is good candidate material for use ceramic spring,
because of its low Young's modulus as well as its reliability guaranteed by excellent crack-
healing ability. However, before estimating the usefulness of the composite as ceramic spring at
high temperature, we must know the fatigue limit at high temperature. Mullite/ 15vol% SiC
whisker/ 10vol% SiC particle multi-composite test specimens were pre-cracked indentation
(surface length of 100 μm) and crack-healed at 1573 K for 2 h in air. The times to failure were
investigated when the crack-healed test specimens were applied the elevated tensile stress in air
at temperatures from 1273 K to 1473 K. The fatigue limit was determined from the test results.
Below 1273 K, the fatigue limit of the crack-healed composite was found to be almost equal to
the corresponding flexure strength. Therefore, the composite was not susceptible to fatigue due
to crack-healing. In contrast, the fatigue limit of the crack-healed composite was found to be less
than the corresponding flexure strength above 1373 K, because the matrix and the crack-healed
region fatigue above 1373 K and 1473 K, respectively.

INTRODUCTION
Ceramic springs are expected to be useful in various fields because they can operate at
higher temperatures than metal springs. However, it is necessary to overcome certain inherent
weaknesses of ceramics, e.g. low mechanical reliability, before ceramic springs can be realized.
Ceramics cannot resist rapid crack growth since the fracture toughness of the crack-healed
composites is quite low. Therefore, the strength of ceramics is determined by the size of flaws,
which limits mechanical reliability. If these flaws were to be completely repaired or eliminated,
 improvement in mechanical reliability would be anticipated. In particular, for ceramic springs in
which the maximum tensile stress is applied to the surface, the elimination of surface flaw is of
the greater importance.

Crack-healing1-3 is a useful technique to repair or eliminate the surface flaws. Because
ceramics include countless surface cracks and flaws, we cannot detect and repair them all and
therefore repairing them all individually. Thus, endowing structural ceramic with self-crack-
healing ability is an attractive and effective way. The present authors succeeded in producing
silicon nitride4-6, alumina7-10 and mullite11-13 materials with self-crack-healing ability by
High Temperature Fatigue Behavior of Mullite/SiC Multi-Composite Crack-Healed admixing SiC particles. When these materials were kept at high temperature in air, only SiC located on crack surfaces were oxidized. The oxidized material filled surface cracks, and bonded strongly with the base materials. As a result, all surface cracks were eliminated.

Mullite/ SiC whisker/ SiC particle multi-composites have potential application in ceramic springs because of excellent crack-healing ability and the excellent mechanical properties. In particular, mullite/ 15 vol.% SiC whisker/ 10 vol.% SiC particle multi-composite, used in this study and abbreviated MS15W10P, is superior to other mullite/ SiC multi-composites. MS15W10P has twice the high fracture strength and 1.7 times the fracture toughness compared to monolithic mullite. Moreover, its crack-healed MS15W10P has high strength up to 1573 K. Thus, MS15W10P is the best candidate material for ceramic springs used in high temperatures.

However, since ceramic springs are subjected to continuous loading, it is important to know the fatigue limits of spring materials at high temperatures. In general, fatigue behavior has been well known to be time dependent rather than cyclic dependent. Therefore, the aim of this study was to investigate static fatigue behavior of MS15W10P composites at elevated temperature. From the test results, the static fatigue limits were obtained from 1273 K to 1473 K.

EXPERIMENTAL

MS15W10P composites were prepared in this study. The mullite powder (KM 101, Kioritz Co. Ltd., Japan) had an average particle size of 0.2 μm and an Al2O3 content of 71.8 %. The SiC whiskers (SCW, Tateho Chemical Industry Co. Ltd., Japan) had diameters between 0.8 μm and 1.0 μm and lengths between 30 μm and 100 μm. The SiC powder (Ultrafine grade, Ibiden Co. Ltd., Japan) had a mean particle size of 0.27 μm. Specific weights of mullite powder, SiC powder and SiC whiskers were mixed well in alcohol via alumina balls and a mill. SiC powder and mullite powder were blended first for 24 h. Then SiC whiskers were added to the mixture and the mixtures of mullite/ SiC whisker/ SiC particle were blended for an additional 12 h. Square plates of 50 mm × 50 mm × 9 mm in size were hot pressed in Ar at 1973 K and 40 MPa pressure for 1 h. All sintered plates had relative densities greater than 98%. The sintered plates were cut into 3 mm × 4 mm × 22 mm rectangular bar test specimens. The test specimens were polished to mirror finish on one face, and the edges of test specimens were beveled 45°, as shown in Fig. 1, to reduce the likelihood of edge initiated failures.

Figure 1 Dimensions of three point bending and the bar specimens
A semi-elliptical surface crack of 100 μm in surface length was made at the center of the tensile surface of the specimens with a Vickers indenter, using a load of 19.6 N. The introduced crack, as shown in Fig. 1, is termed a pre-crack in this paper. The ratio of the depth (d) to half the surface length (c) of the crack (aspect ratio d/c) was 0.9. The tested specimens were subjected to crack-healing treatment at 1573 K for 2 h in air, where the crack-healing conditions were determined by reference to the previous study. These test specimens were called as crack-healed specimens.

All strength tests were conducted with a SiC three-point flexure having a span of 16 mm. The span used in this study was short compared to that of the JIS standard R-1601, because the flexural strength of crack-healed region was measured. Monotonic flexure test were performed from room temperature to 1773 K. The crack-healed specimens were subjected to the elevated static stress at temperatures from 1273 K to 1473 K. The time to failure was measured. From these results, the static fatigue behavior at elevated temperature was investigated. The static fatigue test was finished after 100 h by reference to the JIS standard R-1632. Furthermore, the test specimens that survived the fatigue testing were fractured by three point flexure at the same temperature as the fatigue test had been carried out. All fracture origins were detected by SEM observation.

RESULTS AND DISCUSSIONS

Figures 2, 3 and 4 show the static fatigue results at 1273 K, 1373 K and 1473 K, respectively. The closed triangles attached with the center-line indicate the test specimens fractured from the crack-healed region. This suggests that the weakest region in the test specimen be the crack-healed region. Moreover, the left and right columns show the flexural strength of the crack-healed specimen before and after fatigue testing, respectively. At 1273 K, all crack-healed MS1510P test specimens survived for 100 h under static stresses 50 MPa less than the lower bound of the flexural strength at the same temperature. Alternatively, the crack-healed MS15W10P test specimens fractured at the less than 100 s under stresses corresponding to the lower bound of the flexural strength. This failure is not fatigue but rather rapid fracture. Therefore, it is confirmed that the crack-healed MS15W10P composite is not degraded by the static fatigue at 1273 K.

At 1373 K, the crack-healed MS15W10P test specimens fractured immediately at 450 MPa, corresponding to a stress 50 MPa less than the lower bound of the flexural strength at 1373 K. The time to failure increased as the applied stress decreased. All crack-healed MS15W10P test specimens survived for 100 h under static stresses less than 300 MPa. Thus, the present authors defined the fatigue limit as a maximum stress, under which the specimens never fracture during fatigue test, thereby determining the static fatigue limit of crack-healed MS15W10P at 1373 K to be 300 MPa. Moreover, all fracture origins of the test specimens that fractured during the static fatigue test were not crack-healed region. This suggested that the static fatigue of the crack-healed MS15W10P at 1373 K resulted from the degradation of the base material rather than that of the crack-healed region.

From the relation between the applied stress and the time to failure, the static fatigue limit at 1473 K was determined to be 200 MPa. This value is 280 MPa less than the lower bound of the flexural strength at 1473 K. Moreover, test specimens fractured during the static fatigue test had fracture origins in the crack-healed region. Thus, it was found that the degradation of the crack-healed region occurred at 1473 K.