Ceramic Engineering and Science Proceedings
Rajan Tandon

Fracture and Deformation

HIGH-VELOCITY IMPACT RESISTANCE OF ZrB_2-SiC

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ABSTRACT

The high-velocity impact resistance of hot-pressed zirconum 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 (ZrB_2-SiC and HfB_2-SiC) are candidate materials for the leading edges of hypervelocity atmospheric re-entry vehicles due to their moderate strength[1], high melting temperature and oxidation characteristics[2]. 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 ZrB_2-SiC and HfB_2-SiC ceramics manufactured during the SHARP B1 and B2 flight experiments.[3] 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 ZrB_2-SiC materials prepared at the University of Missouri - Rolla.[1] These ceramics have heterogeneous microstructures and fracture toughness values greater than 2.5-3 $MPa{\cdot}m^{1/2}$.[1] The present work focuses on the strength degradation of this ZrB_2-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.[4] When the projectile makes contact with the specimen, a small contact patch is formed between the surface of the projectile and specimen. Ring 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.

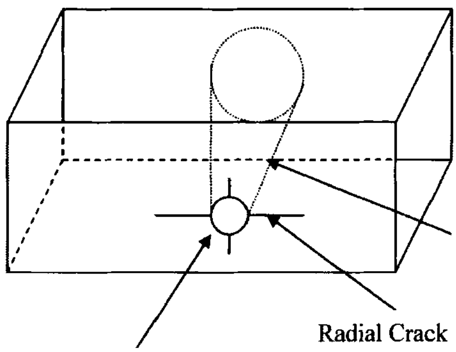

Figure 1: Schematic representation of impact damage in brittle materials. Note that the drawing is not to scale.

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.[4] A variety of studies have demonstrated that quasi-plastic deformation increases at the expense of cone-crack formation as the microstructure coarsens.[4-9] 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.[7-9]

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 B_2O_3 from the surface of the ZrB_2 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.

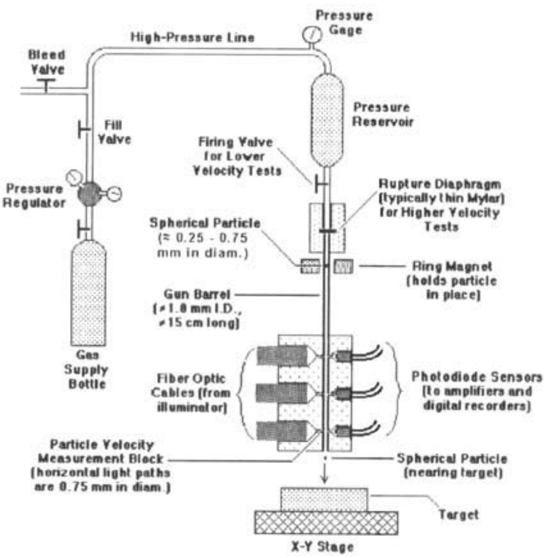

Figure 2: Schematic diagram of the SRI gas gun impact facility.

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).

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 I: Physical properties of tungsten carbide spheres used for impact testing.[a]

Type	WC 44A
Composition (mass%)	6% Co – balance WC
ρ (g/cm^3)	14.95
ν	0.26
E (GPa)	690
H_R (Rockwell)	A91

[a] 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[1]. 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 ~5 μm.

Table II: The physical and mechanical properties of as-prepared ZrB_2-SiC.

Material	**Density (g/cm^3)**	**Relative Density**	**Hardness (GPa)**	**E (GPa)**	σ_f **(MPa)**
ZrB_2-30 vol. % SiC	5.41	~100 %	24±1	485 ± 11	1026 ± 32

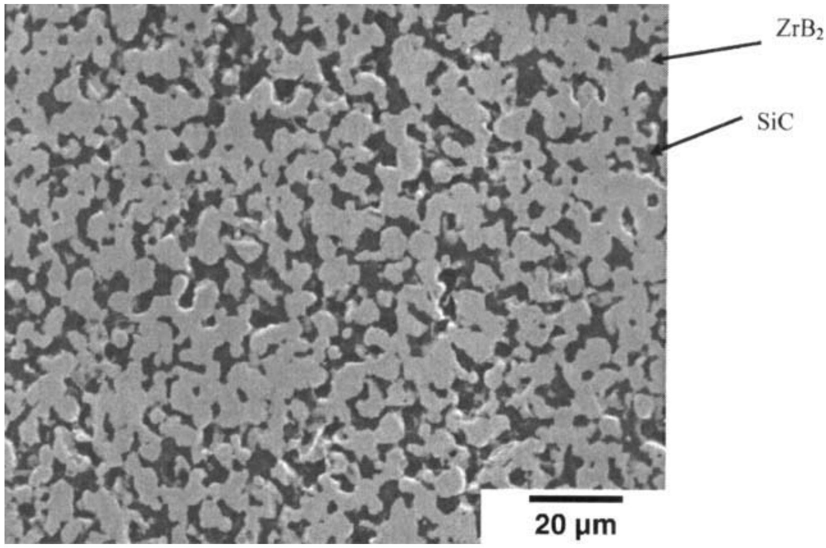

Figure 3: SEM micrograph showing the typical microstructure of as-prepared ZrB_2-SiC.

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.

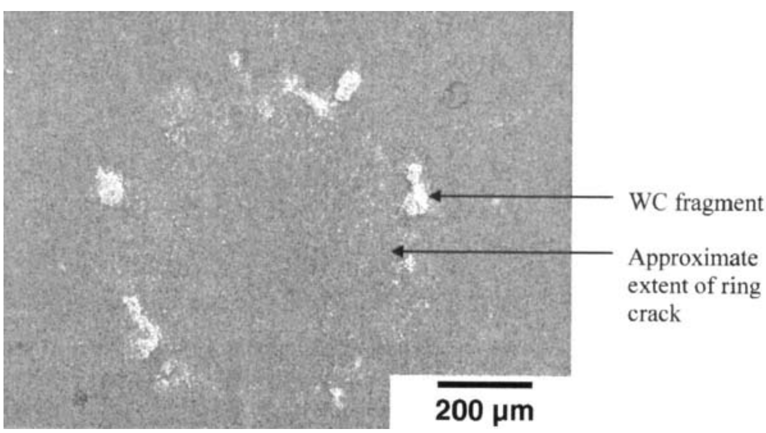

Figure 4: SEM micrograph of a ZrB_2-SiC surface after impact of a WC projectile at 318 m/s.

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 ZrB_2 and SiC grains near the impact site and begin to deflect around the SiC grains outside ~3.5 ring crack diameters from the initiation.

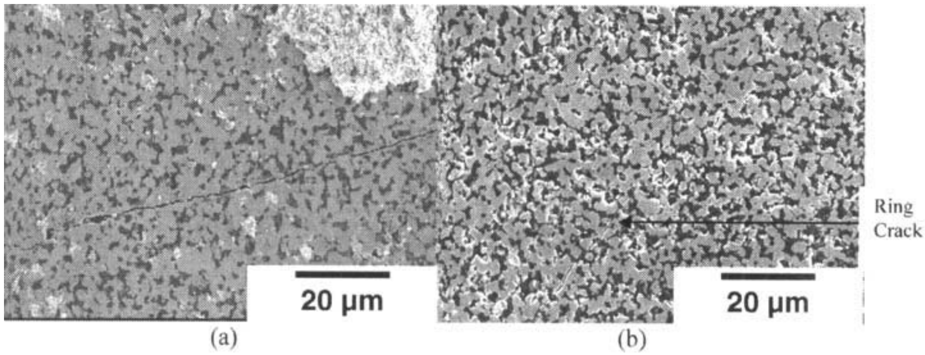

Figure 5: SEM micrographs showing (a) a radial crack and (b) a ring crack after impact of a WC projectile at 304 m/s with the surface of ZrB_2-SiC.

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 ZrB_2-SiC over the impact velocity range.

Impact Velocity (m/s)	**Elastic Modulus (E,GPa)**	**Poisson's Ratio (ν)**
0	485 ± 11	0.146
100-200	483 ± 11	0.146
200-250	483 ± 12	0.146
250-320	483 ± 12	0.146

Prior to testing the ZrB_2-SiC disks, a series of alumina (Al_2O_3) 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 ~250 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 ~290 m/s. Strength decreased from ~1026 MPa to ~600 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 ~400 MPa and failure was thought to initiate at cone cracks. The ZrB_2-SiC samples had no measurable retained strength at velocities above ~320 m/s.

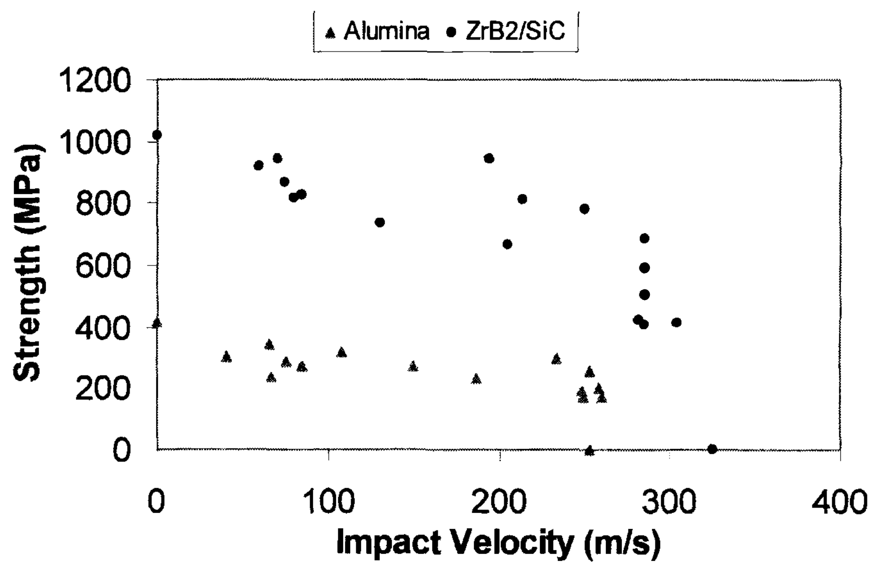

Figure 6: Retained biaxial flexural strength as a function of impact velocity for Al_2O_3 and ZrB_2-SiC.

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 ~290 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 ~290 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.

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