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TITLE STRUCTURE-PROPERTY CHARACTERIZATION OF A SHOCK-LOADED BORON CARBIDE-ALUMINUM

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Structure - Property Characterization of a Shock-Loaded Boron Carbide-Aluminum Cermet

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Shock-recovery experiments were performed on a 65 vol% B_4C-Al cermet as a function of shock pressure. Samples were recovered largely intact due to the use of confinement and "soft-recovery" techniques. Post-shock examination by optical and transmission electron microscopy (TEM) showed the B_4C structure to be unchanged up to 5 GPa, while the Al phase exhibited a high density of randomly distributed dislocations. After a shock of 10.6 GPa some of the B_4C grains displayed dislocation debris, but the majority showed no evidence of plastic deformation. Uniaxial compression testing of the unshocked cermet showed a 24% increase in strength from 1.94 GPa to 2.4 GPa with an increase in strain rate from 10^{-3} s^{-1} to 10^{3} s^{-1}.

1. INTRODUCTION

The propagation of shock waves through metals is known to induce changes in the microstructure even at relatively low stresses. Consequently, the mechanical response of metals (i.e. hardness and yield strength) is highly dependent on their shock histories via the density of dislocations, twins, point defects, and phase transformation products which are induced during shock deformation.

Under quasi-static loading conditions most ceramics and cermets initially respond elastically and subsequently fail at a critical stress with little or no macroscopic yielding. The mechanical response is not controlled by the behavior of dislocations, twins, or point defects, but by the propagation of one or more microstructural flaws.

Under shock loading conditions the mechanical and microstructural response of ceramics and cermets is largely unknown. The majority of shock-recovery studies on brittle materials have concentrated on minerals (Syono 1977, Grady 1977, Jeanloz 1980) and monolithic ceramics (Brusso 1988, Vanderwalker 1988, Louro 1988). Microstructural studies on shock recovered minerals (quartz, anorthite, and periclase) have revealed planar features, "shock lamallae", implying inhomogeneous local plastic flow above the Hugoniot elastic limit (HEL) as suggested by Grady (1977). Conversely, TEM examination of shock-recovered olivine by Jeanloz (1980) found no evidence of shear bands or zones of high local temperatures. Louro (1988) showed that grain boundary flaws and cracking can lead to fragmentation of polycrystalline ceramics at compressive shock pressures below the HEL and Brusso (1988) observed considerable fragmentation in alumina at a pressure of 20 GPa. Impact loading of alumina by Brusso (1988) and titanium diboride (TiB_2) by Vanderwalker (1988) has also shown that a high density of dislocations, and in the case of TiB_2 a large number of point defects, are produced as a result of shock loading. In view of the fact that fragmentation is a serious limitation to the impact performance of monolithic ceramics, the development of high fracture toughness cermets is of considerable interest.

The objective of this study was to: a) develop shock-recovery techniques for cermets and ceramics which reduce the fragmentation of the microstructure by reflected tensile stress waves enabling intact recovery of the shock-loaded samples and b) investigate changes in the microstructure and mechanical properties of a high fracture toughness aluminum-boron carbide cermet as a function of peak shock pressure. The investigation was comprised of quasi-static, dynamic (Hopkinson Bar) compression, and "soft" shock recovery experiments.

2. MATERIAL

A nominally 35 vol% aluminum - 65 vol% boron carbide cermet fabricated at the University of Washington under the direction of Professor Ilhan Aksay was studied. The material is made by infiltrating pure liquid aluminum into a pre-sintered porous boron carbide skeleton at 1175 C. The aluminum wets the boron carbide well under these conditions and yields a dense cermet with interconnected phases and with less than 1% porosity. Quantitative metallography on polished sections has shown the volume fraction of boron carbide to be as high as 0.736 and the average boron carbide phase size to be 7 microns. Table 1 summarizes the quasi-static properties measured by the University of Washington and Los Alamos National Laboratory. TEM characterization of the as-received material revealed the substructure of the cermet to consist of predominantly defect-free B_4C grains interspersed with the aluminum regions. The Al phase was found to contain a low density of dislocations and was well bonded to the boron carbide phase. The boron carbide grains contained no twins and only occasional stacking faults.

TABLE 1

PROPERTY	VALUE
Four Point Bend Strength (MPa)	624 $\pm$ 25
Fracture Toughness (MPa $m^{1/2}$)	9.5 $\pm$ 0.5
Density (g/cc)	2.570 $\pm$ 0.003
Longitudinal Sound Speed (km/s)	10.63 $\pm$ 0.01
Transverse Sound Speed (km/s)	6.24 $\pm$ 0.01
Young's Modulus, w/sound speed, (GPa)	247 $\pm$ 1.5
Young's Modulus, w/compression test, (GPa)	151 $\pm$ 2.5
Compressive Strength, @ 10^{-3} s^{-1}, (MPa)	1935 $\pm$ 96

3. EXPERIMENTAL PROCEDURES

3.1 Hopkinson Bar \ Quasi Static Compression

Uniaxial compression tests were performed on cermet samples at nominal strain rates of 0.0015 s^{-1} and 1500 s^{-1}. Dumb bell shaped specimens having over all dimensions of 13 mm x 4.5 mm with a gage length of 5 mm and a gage diameter of 2.2 mm were loaded using tungsten carbide loading platens to minimize axial splitting. A constant loading rate machine was used for quasi-static compression testing. Two 12.7-mm dia x 0.625 meter Vascomax 350C maraging steel bars were used in the split Hopkinson pressure bar dynamic tests. Specimen load was determined from a load cell during quasi-static testing and from a centrally located transmitted bar strain gage during Hopkinson bar testing. Specimen strain and strain rate was determined by two or three independent strain gages attached around the specimen gage section. A 19 mm long maraging steel striker bar was used to obtain 8 microsecond pulses during the Hopkinson bar tests.

3.2 Shock Recovery

Shock recovery experiments were performed using an 80-mm single-stage gas gun. Samples 18 mm dia x 3.8 mm thick were sandwiched by two 1.5 mm cover plates made from the cermet and were placed in the axially bored hole of a 38 mm dia x 19 mm thick Ti-6Al-4V container. The sample sandwich was placed 16.5 mm deep in the container which was closed from the rear with a threaded plug machined with 18UNF-3A threads. The sample was confined under pressure in an attempt to reduce fracturing by the application of 27 N-m torque to the plug. In the high velocity (10.6 GPa) test the sample was further protected from tensile release waves by a split cermet trapping ring with an outer diameter of 25 mm. This ring was placed around the circumference of the sample and cover plates before placement within the Ti-6Al-4V container. The sample container was backed by a 38 mm dia x 6 mm thick spall plate and surrounded by two concentric momentum trapping rings with outer diameters of 70 mm and 80 mm, respectively. All of the shock-recovery assembly components were made from Ti-6Al-4V to prevent impedance mismatching.

Sample containers were "soft" recovered to minimize incidental impact damage by decelerating the sample in a water catch chamber behind the impact chamber. Previous experiments by Gray *et al* (1988) describe this technique and show that residual strain in shock loaded copper can be maintained at 1.5% or less. Samples were impacted at 508 m/s and 1023 m/s with a 2.8 mm and a 3.0 mm thick Ti-6Al-4V alloy flyer, respectively, to generate a 1 microsecond pulse. The constitutive equation for the cermet:

$$U_s = 6.1 + 1.402\ U_p \tag{1}$$

was calculated based on a rule of mixtures for 65 vol% B_4C and 35 vol% Al.

Impact velocities of 508 m/s and 1023 m/s were estimated to produced peak shock amplitudes of 5 GPa and 10.6 GPa, respectively, in the cermet.

3.3 Sample Preparation / Examination

The as-received and shock-loaded samples were sectioned for TEM examination using a low-speed diamond saw into wafers nominally 250 microns thick. Disks with diameters of 3 mm were ultrasonically cut from the wafers. The disks were mechanically dimpled using 3 micron diamond paste to a center thickness of 25 microns and then ion thinned at approximately 150 C using a 6 kV ion source at a grazing angle of 10 to 15 degrees. Foils were observed at 200 kV using a JEOL 2000FX equipped with a double tilt stage.

Optical microscopy was conducted on polished sections of the shock loaded samples which were mounted to observe the microstructure along the loading axis. Polished sections were prepared by hand using 15 micron alumina in a water slurry followed by 6 and 1 micron diamond paste on a flat glass plate. Due to the extreme differences in abrasive removal rates between the boron carbide and aluminum phases only details of the boron carbide phase can be properly discerned from the polished sections.

4. RESULTS

4.1 Hopkinson Bar \ Quasi-Static Compression

Figure 1 is a typical stress versus strain plot for both Hopkinson bar and quasi-static strain rates. Peak strengths of between 2280 MPa and 2500 MPa were measured during three Hopkinson bar tests-to-failure. The average peak strength was found to be 2390 MPa with a standard deviation of 110 MPa. The total strain measured at peak strength averaged 1.3%. Typically, another 1.2% strain appears to accumulates after peak strength and prior to complete failure. The elastic loading modulus averaged 190 GPa for the Hopkinson bar tests.

Quasi-static testing was performed to failure on four specimens. The three independent strain gages typically indicated a notable difference in strain during loading due to misalignment in the loading axis or due to poor gage attachment. Hence, the quasi-static modulus at 1% total strain appears to vary between 140 GPa and 160 GPa and the strain at peak strength varies by up to 30%. Yield in the quasi-static tests appears to occur as early as 0.1% strain and becomes notable at a total strain of about 1.0% to 1.2% which corresponds to the Hopkinson bar peak strength condition. Permanent strains of between 0.4% and 0.7% were observed after complete unloading due to failure in the quasi-static tests. These values are approximately 1/3 the amounts measured in the Hopkinson bar tests. Post-test examination of the samples shows that fracture orientations of 45 degrees predominate for both strain rates indicating shear failure on planes of maximum shear stress. Failure of the Hopkinson bar samples appears to be the result of the coalescence of a network of microcracks. A network of microcracks intersecting the shear fracture plane of a Hopkinson bar test sample is shown in Figure 2.

4.2 Shock Recovery

Low magnification observations of the cermet after shock loading to 10.6 GPa and 5 GPa (Figures 3 A&B, respectively) shows largely intact samples and reveals similar radial and circumferential cracking. The cracking patterns are considered to arise from tensile waves generated by reflections of the compressive shock wave. Optical microscopy of polished sections of the shocked samples parallel and perpendicular to the load axis indicated occasional cracks aligned with those shown in Figure 3. No localized shear damage or uniform micro-cracking through the section thicknesses were observed.

TEM characterization of the microstructure following shock loading revealed that the cermet appears to have responded predominantly elastically to both the 5 and 10.6 GPa shocks. The boron carbide substructure displayed <u>no</u> evidence of plastic flow, i.e. dislocation activity, after the 5 GPa shock. The Al phase contained a uniform distribution of dislocation debris.

The overall microstructure of the cermet after the 10.6 GPa shock (Figure 4) was similar to the 5 GPa shocked sample. However, higher magnification TEM revealed that some of the B_4C grains contain evidence of local plastic

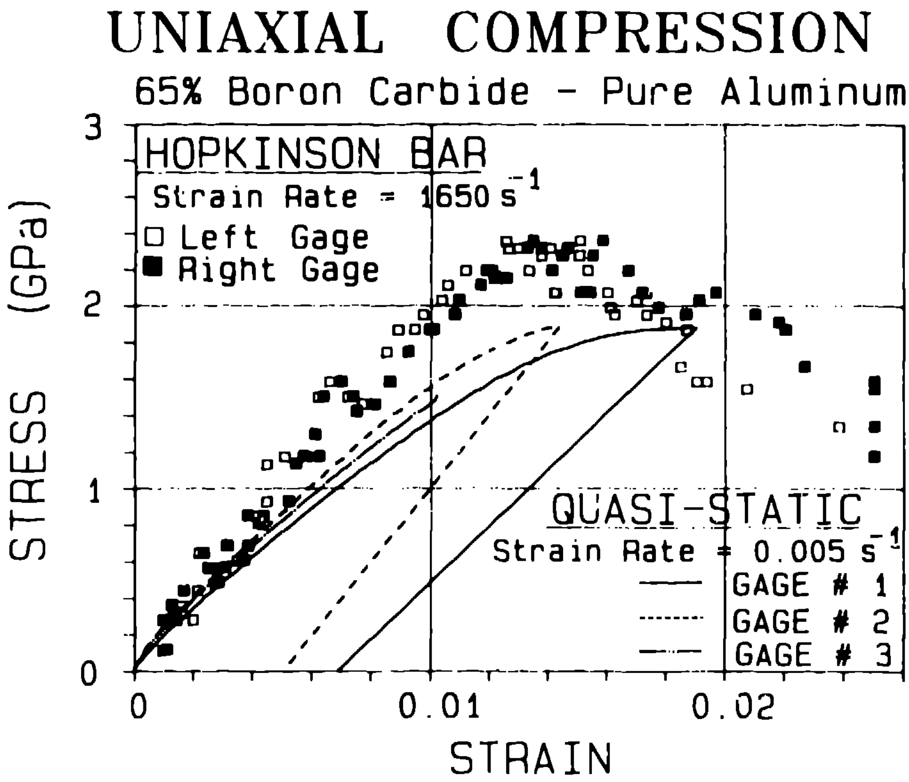

Figure 1: Stress versus strain plot for both quasi-static and dynamic strain rate loading of a 65% B_4C - Al cermet.

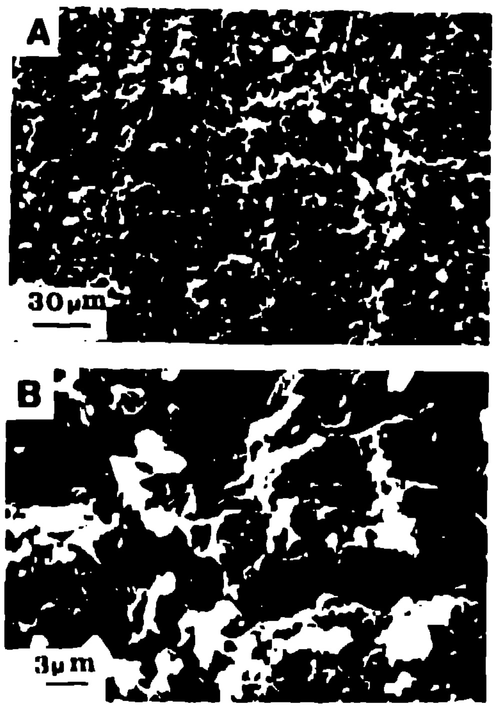

Figure 2: Shear fracture surface of a Hopkinson bar sample revealing a network of microcracks.

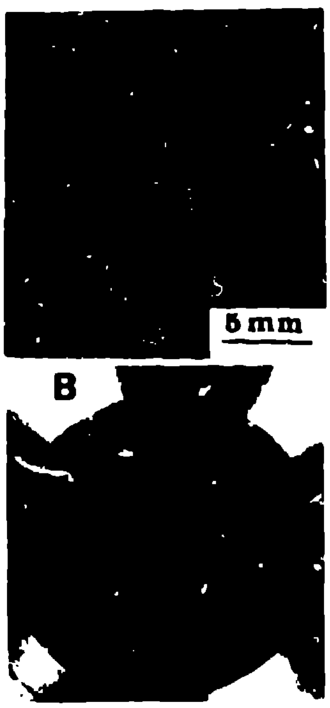

Figure 3: Cermet samples after shock loading to: a) 10.6 GPa and b) 5 GPa.

Figure 4: General microstructure of the cermet following shock loading at 10.6 GPa using TEM.

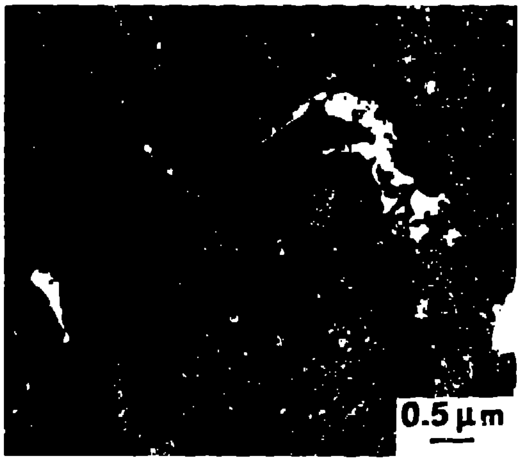

Figure 5: TEM micrograph of dislocations in a B_4C grain after the 10.6 GPa shock.

deformation. Figure 5 shows a TEM bright-field micrograph of dislocations in a B_4C grain after the 10.6 GPa shock. The dislocations appear to be primarily concentrated near the B_4C grain boundaries suggesting that either dislocation activity was higher near the grain boundaries or that dislocations have piled-up against the boundary after traversing the grain interior. No evidence of deformation twinning was observed in either shock-loaded sample.

Ultrasonic characterization of a portion of each shocked samples indicates a steady decay in both the longitudinal and shear velocities (C_l and C_s) with shock pressure. A C_l of 10.01 ± 0.01 km/s and a C_s of 6.01 ± 0.01 km/s were measured for the 5 GPa shocked sample. A C_l of 9.63 ± 0.08 km/s and a C_s of 5.68 ± 0.07 km/s were measured for the 10.6 GPa shocked sample. This represents a decay in C_l of 5.83% and 9.45% for the 5 GPa and 10.6 GPa shocks, respectively. C_s was found to decay by 3.74% and 9.04% for the 5 GPa and 10.6 GPa shocks, respectively. Densities were measured using Archimedes method and found to be 99.9% of the unshocked cermet density for both pressures.

5. DISCUSSION

The present quasi-static compression strengths and strains at failure using the dumb-bell specimen are fully twice those measured during quasi-static compression tests using right circular cylinders with a length-to diameter ratio of 1.27 and teflon tape at the interfaces to reduce frictional effects. Fractures on these cylindrical specimens were predominantly axial splitting due to the development of tensile stresses and stress concentrations at the specimen ends. Hence, the use of dumb-bell-shaped specimens prevents premature failure and provides a proper magnitude for the compression strength during quasi-static compression testing. Compression testing requires precision sample machining, careful sample alignment, and multiple strain gaging of the sample to ensure alignment of the load axis. Precise alignment of the quasi-static samples in the present study was not generally achieved. The best sample alignment produced strain records where the lowest record was 92% of the highest record. A compression strength of 1964 GPa was measured for this sample. The worst alignment occurred when the lowest strain record was only 78% of the highest strain record. The compression strength in this case was the highest recorded at 2057 GPa. Hence, slight mis-alignment of the dumb-bell specimen appears to produce a strengthening effect. The moderate strain variations measured during quasi-static testing were not observed during Hopkinson bar testing using identical specimen geometries and strain gages. Consequently, Hopkinson bar compression strengths are more reproducible and may be more accurate. The Hopkinson bar compression test should be considered as a basic property screening test for materials subject to both quasi-static or dynamic compression loading where retention of the fracture surfaces is desired.

The low strain rate sensitivity in the current study is not in conflict with previous observations by Lankford (1981) for sintered silicon carbide and MgO-doped Al_2O_3. In Lankford's study a sharp increase in the compressive strength of between 30% to 100% is observed for strain rates of between about 10^3 s^{-1} and 10^4 s^{-1}. This behavior is characteristic of some, but not all, of the ceramic materials which have been tested.

Using the present Hopkinson bar configuration, strain rates as low as 3 x 10^2 s^{-1} could be achieved. However, sample strain rates could not practically be driven over 3 x 10^3 s^{-1}. It was apparent that load train component impedance mismatch became a limiting factor and that test accuracy decayed as the strain rate was increased. Therefore, use of the Hopkinson bar as a stand-alone test to investigate material strain rate sensitivity is considered to be of limited value unless the material appears to be moderately strain rate sensitive between a nominal quasi-static strain rate of 10^{-3} s^{-1} and an optimum Hopkinson bar strain rate of about 10^3 s^{-1}.

The measurable yield and permanent deformation occurring prior to failure in the 35% Al-65 vol% B_4C cermet for strain rates of 10^{-3} s^{-1} and 1650 s^{-1} indicates that the aluminum phase allows microcracking of the boron carbide phase, but that it resists further crack propagation affirming the high quasi-static fracture toughness measurements shown in Table 1. The permanent deformation is <u>not</u> the result of plastic flow in the B_4C grains, but is a combination of microcracking and accommodating plastic flow by the Al phase. The unusual aspect of the Hopkinson bar failure is the extensive "sliding" damage observed on the fracture surfaces and the associated "melt" features (Figure 2) suggesting frictional heating up to at least the melting temperature of aluminum (660 C). Quasi-static fracture surfaces have not been examined at high magnification to corroborate the dynamic observations. Since failure at both strain rates occurs in the maximum shear stress mode, the average shear strength can be calculated as one-half the average compression strength, i.e. 958 MPa for quasi-static loading and 1195 MPa for dynamic loading.

The results of the "soft" recovery shock experiments show that an Al-B_4C cermet does not undergo catastrophic fragmentation upon shock loading, even at a peak pressure likely to be above the HEL of the cermet. This result is in contrast to previous shock recovery work on monolithic ceramics and suggests that either cermets behave drastically different than monolithes during the shock process or that the current shock assembly design circumvents many previous problems related to "soft" shock recovery of brittle solids. The very low HEL and strength of the Al phase means that the peak shock stress is almost entirely supported by the B_4C skeleton. It therefore seems most likely that the successful recovery of the cermet is due to the recovery techniques employed rather than any dynamic performance differences between ceramics and the cermet. The use of internal momentum trapping components made from the same cermet as the sample is considered to be the key to successful recovery in this study.

The implications of the shock recovery portion of this study are that care <u>must</u> be taken when assessing the defect generation in brittle materials prescribed to the shock, especially when the experiment does <u>not</u> yield intact samples below the HEL. The present study shows that when a cermet is shocked below or slightly above the HEL the mechanical response is principally elastic. If this study had yielded highly fragmented samples below the HEL of the cermet, similar to previous studies on monolithic ceramics, then the authors would be reluctant to attribute defects in the fragments to the shock conditions since these defects could be the result of tensile interactions causing the fragmentation.

6. CONCLUSIONS

The following conclusions can be reached from uniaxial compression testing of a 65% B_4C - Al cermet: 1) Use of a dumb-bell-shaped specimen geometry was the key to successful compression testing by preventing premature failure due to axial cracking. 2) A 24% increase in compressive strength occurs as the strain rate is increased from about 10^{-3} s^{-1} to 10^{3} s^{-1}. 3) Yielding and permanent deformation occur at both strain rates due to the development of microcracks in the B_4C phase which are arrested by the ductile aluminum phase.

Based upon "soft" shock recovery experiments the following conclusions can be made: 1) Momentum trapping, protective confinement, and "soft" arrest techniques are critical to successful shock recovery and subsequent microstructural analysis of brittle materials. 2) The predominant mechanical response of the cermet to both the 5 and 10.6 GPa shocks is elastic, although the aluminum phase behaved plastically due to its very low HEL. Only the 10.6 GPa shock produced dislocation activity in a few of the B_4C grains. Dislocation debris from this activity was concentrated near the grain boundaries. 3) No evidence of localized shear damage or uniform microcracking was observed at either 5 GPa or 10.6 GPa.

7. REFERENCES

Brusso J A, Mikkola D E, Flinn J E, and Kelsey P V 1988 *Scripta Metall.* 22 47

Grady D E 1977 *High Pressure Research* ed M H Manghnani and S Akimoto (New York: Academic Press) pp 389-435

Gray G T III, Follansbee P S, and Frantz C E 1988 *Mat. Sci. & Engin.* in press

Jeanloz R 1980 *J. Geophysical Res* 85 3163

Lankford J 1981 *J. Am. Ceram. Soc.* 64 C-33

Louro L H L, Lindfors A and Meyers M A 1988 *DYMAT88 J. De Physique Colloque C3 Supplement #9* 49 333

Syono Y, Goto T, Nakagawa Y and Kitamura M 1977 *High Pressure Research* ed M H Manghnani and Akimoto S (New York: Academic Press) pp 477-489

Vanderwalker D M and Croft W J 1988 *J. Mater. Res.* 3 761

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