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Comparison of Slip Cast to Hot Pressed Boron Carbide

by T. Sano, E. S.C. Chin, B. Paliwal, and M. W. Chen

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14. ABSTRACT
To meet the possible increase in future demand for armor materials, an increase in the throughput during manufacturing is necessary. One possibility is the use of the slip casting and sintering technique to form ceramic armor compacts as an alternative to current hot pressing techniques. Dynamic uniaxial compression tests with the Kolsky bar were conducted on two types of slip cast boron carbide, and compared with results from the standard hot pressed boron carbide. One type was slip cast, sintered, and hot isostatically pressed, while the other was only slip cast and sintered. Microstructural characterization by transmission electron microscopy showed graphite inclusions and more annealing twins than in the hot pressed boron carbide material. Examination of fragments recovered from the compression tests determined that the fracture mode of both slip cast materials was brittle transgranular cleavage. The compression test results show comparable compressive strengths between the sip cast and hot pressed boron carbide despite higher density of graphite in the slip cast material.

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ABSTRACT

To meet the possible increase in future demand for armor materials, an increase in the throughput during manufacturing is necessary. One possibility is the use of the slip casting and sintering technique to form ceramic armor compacts as an alternative to current hot pressing techniques. Dynamic uniaxial compression tests with the Kolsky bar were conducted on two types of slip cast boron carbide, and compared with results from the standard hot pressed boron carbide. One type was slip cast, sintered, and hot isostatically pressed, while the other was only slip cast and sintered. Microstructural characterization by transmission electron microscopy showed graphite inclusions and more annealing twins than in the hot pressed boron carbide material. Examination of fragments recovered from the compression tests determined that the fracture mode of both slip cast materials was brittle transgranular cleavage. The compression test results show comparable compressive strengths between the sip cast and hot pressed boron carbide despite higher density of graphite in the slip cast material.

INTRODUCTION

Hot pressing boron carbide (B_4C) powder is the commercial technique used to form personnel armor plates and components for various applications. B_4C is used widely in abrasive, wear resistant components, and armor applications due to its high hardness and low density properties. It is possible for B_4C components formed by the hot pressing technique to reach nearly full theoretical density and achieve high mechanical performance. Compared to sintering processes, hot pressing requires less additives for better densification and strength. These additives could however also form precipitates or secondary phases at the grain boundaries and be detrimental to the mechanical performance. The limitation of the hot pressing technique is the high operation cost per batch and only plates or cylindrical shapes of a limited size can be produced. Also, in addition to a larger die, to achieve the same pressure applied to a smaller specimen, a much larger hot press machine size is required for larger specimens.

Recently, an alternative technique of forming B_4C compacts was described by Matsumoto et al.[1]. In this technique, B_4C powder was slip cast, sintered, and hot isostatically pressed (HIPed). The mechanical properties of these slip cast and HIPed B_4C materials were reported[2] to be as good as the hot pressed B_4C materials. The aim here is to evaluate the dynamic mechanical performance of slip cast–sintered-HIPed, and slip cast-sintered B_4C and compare them with hot pressed B_4C. In addition to the mechanical testing, microstructural characterization of the HIPed and SCS samples was conducted to better characterize the material pre and post compression tests.

EXPERIMENTAL

Two types of B_4C samples were obtained. One was slip cast and sintered, and the other was slip cast, sintered, and HIPed. The slip cast and sintered samples will be referred to as the "SCS" B_4C samples, and when identifying the samples that were also HIPed, described as "HIPed" to distinguishing between the two types. The third sample compared in this study is hot pressed B_4C. This B_4C sample is the armor grade reference benchmark material and will be referred to as the hot pressed sample. This hot pressed B_4C was analyzed in a previous work[3] and the data is taken from the paper on the prior analysis.

The surfaces of the as received slip cast B_4C (SCS and HIPed) samples were imaged with a scanning electron microscope (SEM) equipped with a field emission gun. The samples were polished on the Struers automatic polisher with diamond slurries of decreasing diamond abrasive size at each polishing step, until reaching 0.25 μm. The samples were then polished on a vibramet polisher with 0.02 μm colloidal silica. The post-polished slip cast samples were imaged on the SEM. The Knoop hardness of the two samples was measured with a microhardness tester, at loads from 3 N to 98 N (0.3 Kg to 10 Kg). To determine the existence of any impurities and different B-C phases, x–ray diffraction was conducted. The impurity phases were verified, and grain boundaries examined with transmission electron microscopy.

The slip cast samples were also machined into the 4.0 mm x 5.2 mm x 3.0 mm geometry for dynamic mechanical testing with the Kolsky bar. Each sample was loaded onto the Kolsky bar ends with lithium grease, which also acts to minimize the friction between the sample and the titanium alloy platens. The full Kolsky bar setup is described in the work by Ramesh and Narasimhan[4]. Each sample was subjected to dynamic compression at the rate between 150 and 160 MPa/μsec. The stress and strain rate throughout the compression test cycle was captured by a high speed camera at 2 or 3 μs intervals with 300 to 700 ns exposure time.

The sample area was enclosed in a clean polycarbonate box with a clean sheet of paper lining the bottom of the box to collect the fragments after the compression experiment. After each experiment, the polycarbonate box was cleaned and a new sheet of paper was installed to minimize sample contamination and to collect the fragments from the next experiment. The collected fragments were labeled with the B_4C processing type and experiment number then examined with the SEM and energy dispersive spectrometry, or EDS. The fracture surfaces of the HIPed B_4C samples were compared with the SCS B_4C as well as with the hot pressed B_4C samples.

RESULTS

The surfaces of the as machined SCS and HIPed B_4C samples were examined in the SEM and EDS. Samples were also prepared and examined with TEM. The various microscopy techniques revealed dark and pore-like areas to be graphite inclusions. Compared to the hot pressed B_4C, the slip cast B_4C appeared to have more, though smaller sized, graphite inclusions.

The as received slip cast B_4C samples were x-rayed to determine the phases and identify any impurities. The diffraction peaks for both samples were consistent with each other and can be inferred that both samples have the same phase and impurities. When the diffraction peaks in both samples were identified, B_4C, $B_{13}C_2$, and carbon (graphite) were determined to be the phases present. Figure 1 shows the peak identification of the HIPed sample. The same phases were identified in the SCS sample.

The volume percents of the graphite flakes for both slip cast B_4C were measured from SEM images and calculated to be 10%. Even with these graphite inclusions, the density of the HIPed B_4C, calculated by the Archimedes method, was 2.50 g/cm^3, or 99.2 % of the theoretical density, and 2.45 g/cm^3, or 97.2 % of the theoretical density for the SCS B_4C. Assuming from the lack of porosity, determined by visual examination of SEM micrographs, that the HIPed sample is fully dense (i.e. no porosity), the remaining 0.8 % or 0.02 g of the density was comprised of graphite. With this assumption, the vol % of graphite was calculated to be 9 vol %. Assuming the same amount of graphite was in the SCS sample, the remaining 2 % of the theoretical density is therefore due to pores. The total volume of the machined sample was measured to be 0.06 cm^3. From the difference in the density between the HIPed and SCS samples, the approximate volume percent of pores in the SCS sample was calculated to be 2.0 vol%.

Neither volume percent of pores nor graphite was calculated for the hot pressed B_4C, but the density was determined previously by Chen et al.[3] to be 2.49 g/cm^3, or 98.8 % of the theoretical density.

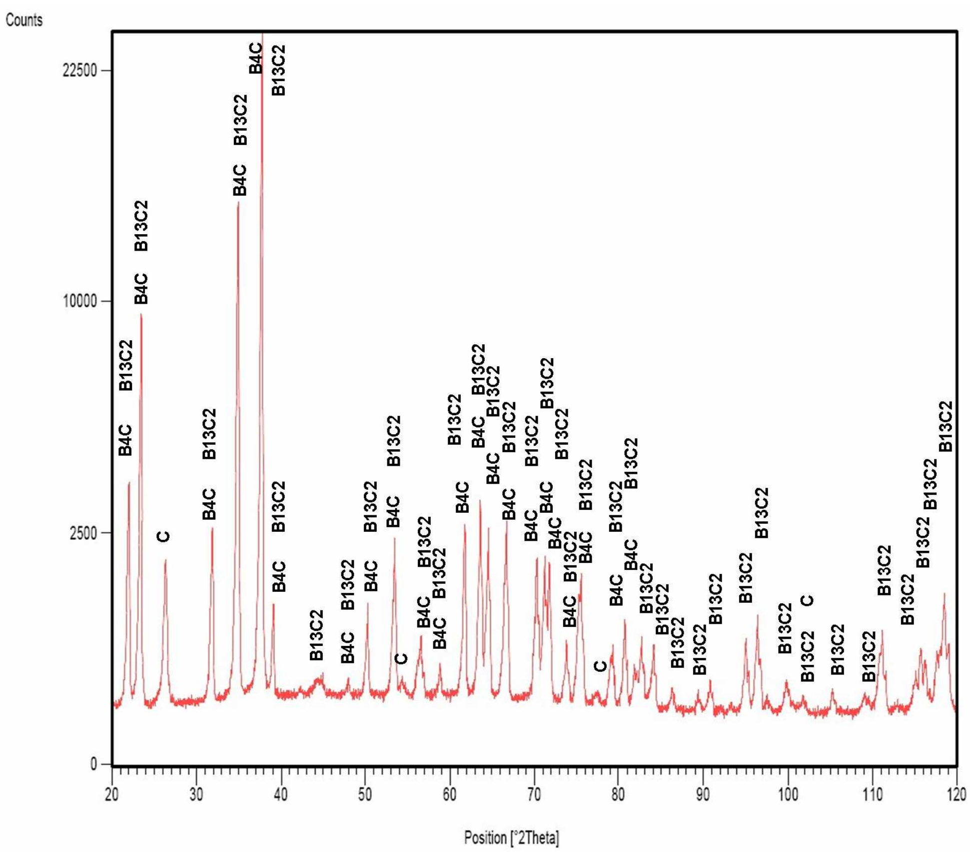


Fig. 1. The HIP diffraction pattern with B_4C, graphite, and $B_{13}C_2$ peaks labeled.

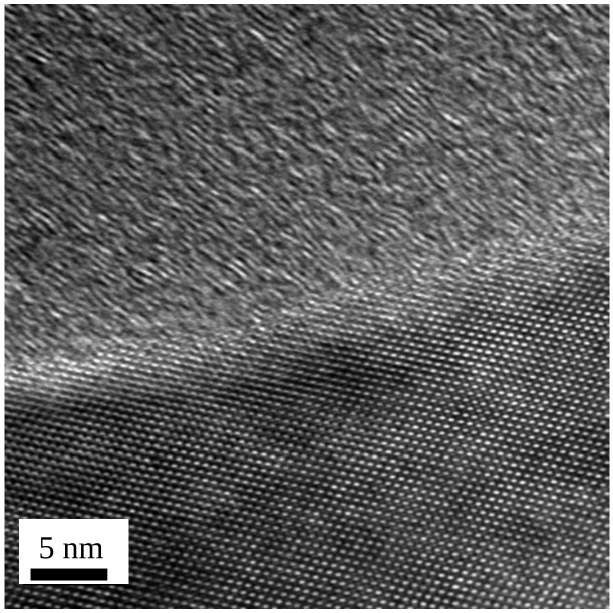


Fig. 2. Amorphous interface boundary between graphite and a B_4C grain.

Compared to the hot pressed B_4C, which had possible precipitates and distinct chemical compound at the triple junctions[3], the slip cast B_4C had fewer impurities. SEM and TEM analysis of both slip cast samples show only graphite inclusions, some trapped within the grain and others at triple junctions. The interface between the graphite inclusion and B_4C grains were determined by TEM to be amorphous as shown in Fig. 2. Another observation by SEM and TEM was the numerous twins; more than in the hot pressed B_4C samples, as shown in Fig. 3. This is in agreement with previous work by Schwetz et al[5]. Examination of the 100 nm to 1 μm grain size B_4C powder used in the slip cast samples did not show any twins. However the polished surfaces of slip cast samples that did not undergo compression tests, show many grains with twins. Hence the twins observed in the slip cast samples are not deformation twins and are growth twins formed during the processing steps.

After polishing, hardness measurements were conducted on the slip cast B_4C samples. Vickers indentation was initially attempted. However due to the high hardness and low toughness properties of B_4C, the indentation marks were not measurable. Hence Knoop indentations were performed with varying load. Table I shows the comparison of the Knoop hardness measurements at a load of 19.6 N (HK(2)), in accordance to ASTM C1326.

Each sample that underwent dynamic uniaxial compression test with the Kolsky bar was imaged with a high speed camera and the stress and strain values recorded. Figure 4 (a) shows camera frame shots of the SCS sample #1 during the compression test and (b) is the plot of the stress and strain rate over time. In Fig. 4 (a), the sample exhibited cracking from the edges inward. The other SCS samples, as well as the HIPed samples, regardless of the compressive strength, showed similar trends in the stress and strain profiles. All samples displayed through-sample cracking at the time interval just past the maximum stress peak, and destruction of the sample shortly thereafter. The comparison among the SCS, HIPed, and the hot pressed B_4C compressive strengths are shown in Fig. 5.

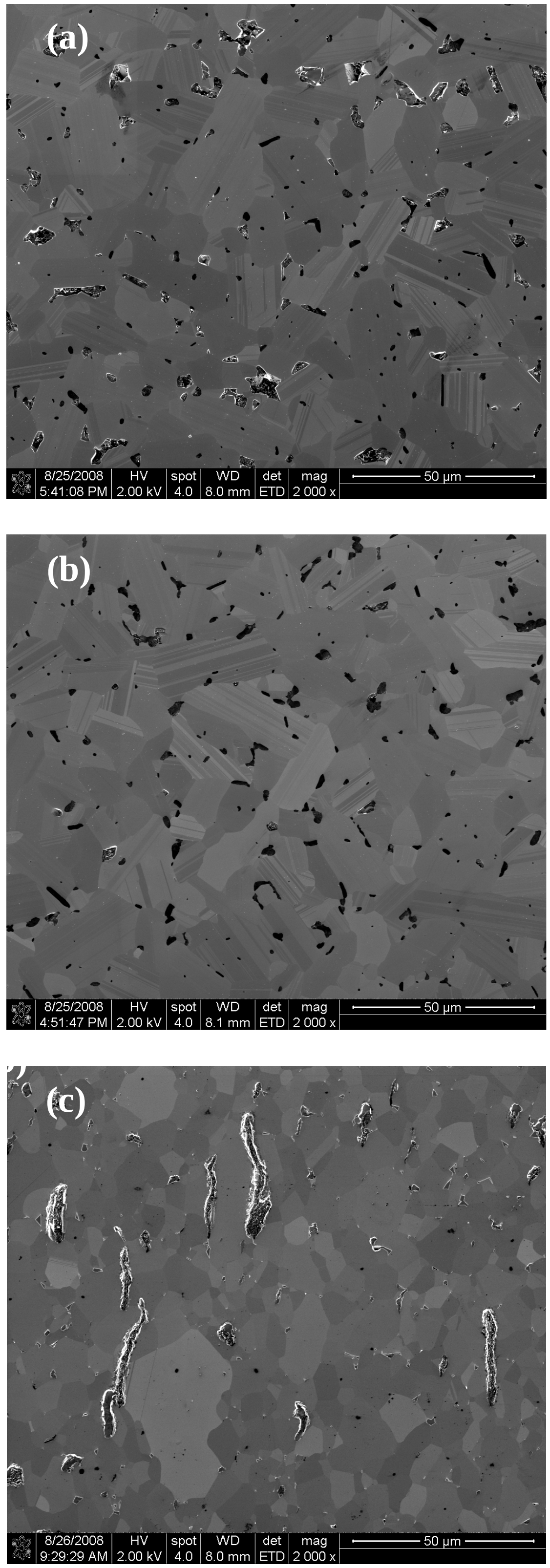
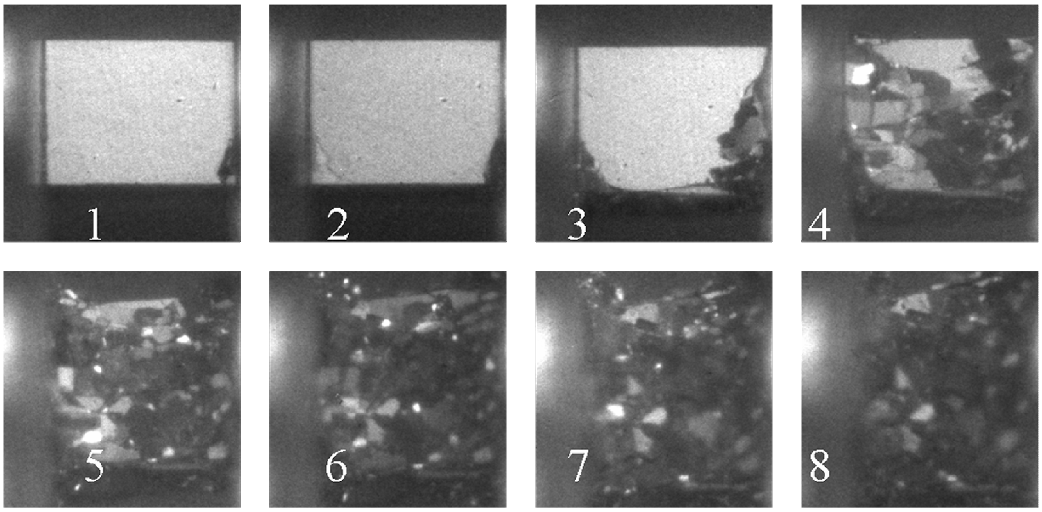


Fig. 3. Polished surfaces of B_4C that was a) SCS, b) HIPed, and c) Hot pressed. Numerous growth twins are visible in a) and b).

Table I. Average Knoop Hardness Values at 19.6 N Load

Material	Ave. (GPa)	Std. Dev. (GPa)
HIP	21.1	0.6
SCS	18.6	0.9
Hot Pressed[7]	19.6	1.8



(a) Inter-frame time 3μs, exposure time 500ns

(b)

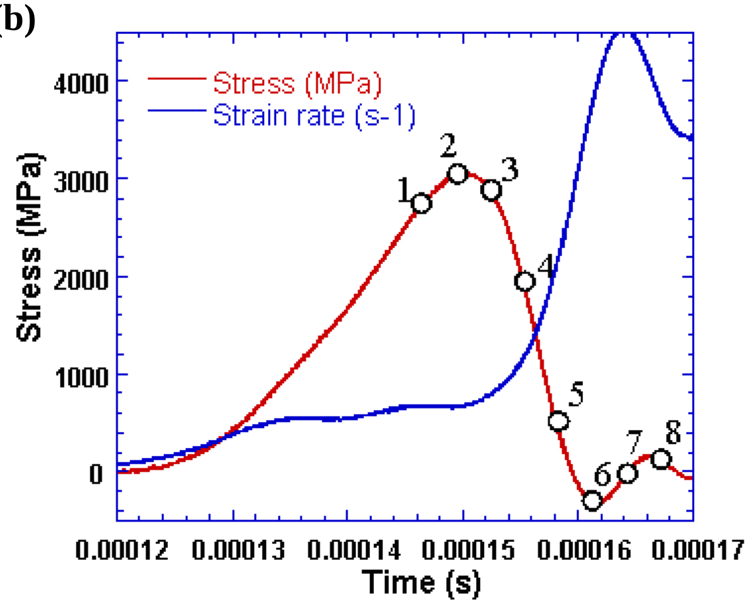


Fig. 4. a) High speed camera images of SCS sample #1 during the Kolsky bar compression test. The image numbers correspond to the stress and time plotted in b).

The fragments from the Kolsky bar experiments were carefully collected and examined in the SEM. All samples displayed fracture by brittle, transgranular fracture mode, evidenced by the cleavage fracture surfaces. Figure 6 (a) is an example of a fractured surface of a fragment from a SCS sample (Sample #1). Twins were observed on all the fractured surfaces. Graphite inclusions were also frequently observed on fractured surfaces, whether due to them actively influencing the fracture path or due to the high density of the inclusions in the material. Similar to Fig. 6 (a), Fig. 6 (b) is a SEM image of a fragment from a HIPed sample. Compared to the sintered B_4C samples, the hot pressed samples revealed the same cleavage fracture, however with very few fractured surfaces with twins.

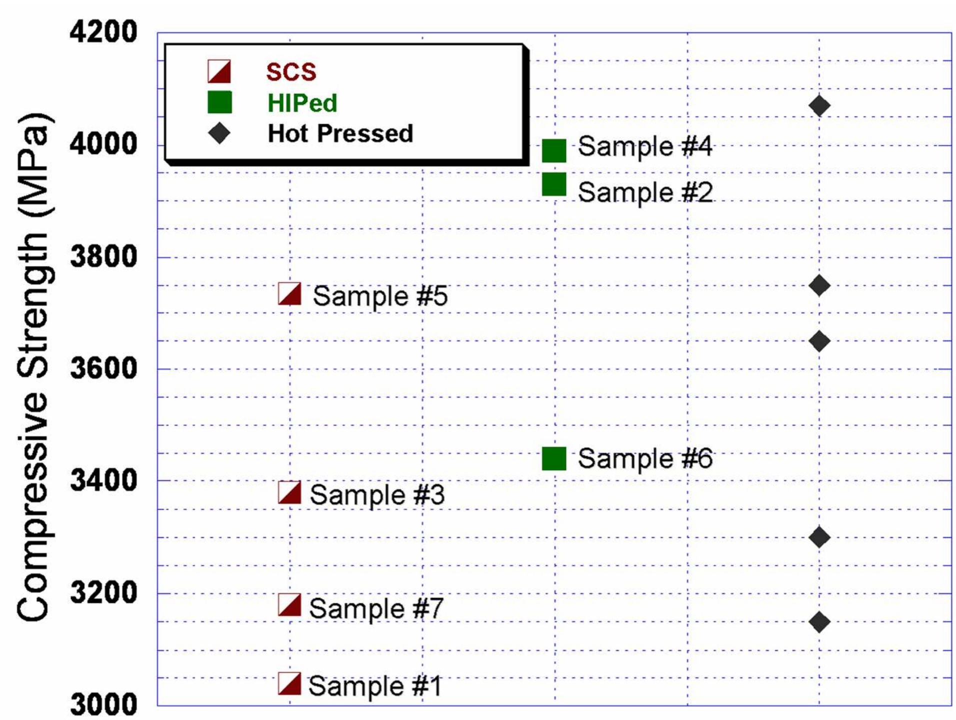


Fig. 5. Compressive strengths of SCS, HIPed, and hot pressed B_4C samples.

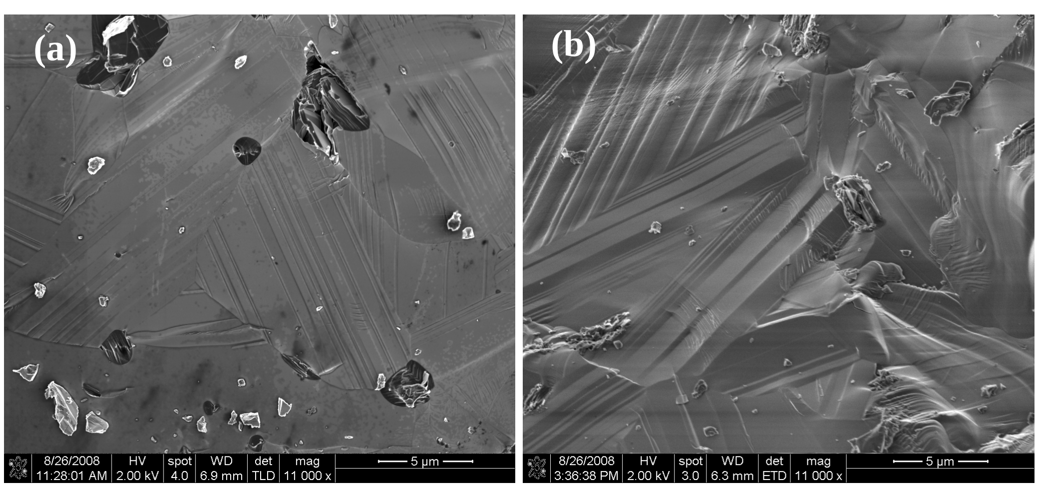


Fig. 6. (a) An SCS sample fragment surface. (b) A HIPed sample fragment surface.

DISCUSSION

All B_4C samples had graphite inclusions, which for the slip cast samples, was found at the grain boundaries, triple junctions, and trapped within the grains. The graphite was already in the initial powder, and was not formed during the processing.

The Vickers hardness values provided by the manufacturer for the HIPed B_4C was the highest at 39 GPa (load unknown) followed by the SCS B_4C with 32 GPa. The Vickers hardness HV(0.3), provided by the manufacturer for the hot pressed B_4C was 26.5 GPa. When Vickers hardness tests were conducted by the authors, severe spalling occurred and the data was deemed unreliable. However Knoop hardness values were successfully obtained. The HK(2) values for the HIPed B_4C was 21.1 GPa with a standard deviation of 0.6 GPa and the SCS B_4C was 18.6 GPa with a standard deviation of 0.9 GPa. These values as well as the tested range of hardness values from HK(0.3) to HK(10) were in agreement with the Knoop hardness results of hot pressed B_4C by Swab[7]. Except for at HK(10), the HIPed B_4C consistently had the highest hardness values, followed by hot pressed, then SCS. This hardness trend follows the density, in which the densest B_4C was the HIPed sample, followed by hot pressed, then the SCS sample.

For the Kolsky bar compression test, having rough surfaces is known to lower the compressive strength, even with lubricant applied along the contact sides of the test specimen[8]. If too much lubricant is used, the lubricant can be detrimental by filling in the pores and other depressions on the rough surfaces and force crevices to open. Since the as machined slip cast B_4C had many scratches on the surface, it can be expected that with a smoother surface finish, the compressive strengths would be higher. The way to minimize the surface finish problem would be to machine cylindrical dog-bone shaped specimens. However the machining cost for such a geometry would be expensive. Nevertheless assuming that the hot pressed B_4C has similar machining difficulties affecting the surface finish as the slip cast material, the compressive stress results should still be comparable.

Even with limited number of data and despite all the processing and microstrucutral differences, all three B_4C samples performed within a standard deviation from each other in the dynamic uniaxial compression test. Provided that the surface finish was similar, this indicates the importance of the material properties on the compressive strength rather than the processing method to achieve that strength. This also shows that impurities and growth twins play limited roles on the compression performance. It can be deduced that as long as the initial B_4C powder is at least 96 wt% pure, the grain size ranges from 5 to 15 μm, and the sample density is above 98.8 % of the theoretical density, the compressive strength can be expected to fall within the range of 3000 to 4100 MPa. It is shown here that slip casting or slip casting with the HIP step can be used to form unconventional shapes of B_4C samples with similar mechanical performance as B_4C samples that are hot pressed. Also, though not realized in this study, the addition of the HIP process increases the density which, based on general trends, should increase the mechanical performance. Hence the HIP process could not only complement B_4C production, but could possibly produce better B_4C compacts.

CONCLUSION

Microstructural characterization and dynamic uniaxial compression tests with the Kolsky bar were conducted on B_4C samples that were SCS and slip cast, sintered, and HIPed. Growth twins were more frequently observed on the surfaces of SCS and HIPed samples than those of hot pressed, benchmark B_4C samples. The compressive strengths results were compared with those of previously tested commercially hot pressed B_4C samples. The compressive strength of the HIPed and non HIPed samples were in the range of compressive strength of the hot pressed B_4C samples. Hence slip casting and sintering B_4C is a feasible technique to obtain unconventionally shaped samples with comparable compressive strength as hot pressed B_4C samples.

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