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The microstructure and mechanical properties of boron carbide (B_4C) samples processed by slip casting were compared with those processed by the conventional hot-pressing technique. Although the quasi-static and dynamic mechanical experiments showed comparable results between the slip-cast and hot-pressed B_4C , scanning electron microscopy and electron back-scattered diffraction of the B_4C samples revealed significant differences in the microstructure. A notable difference was the numerous growth twins in the slip-cast samples. The twin planes were determined to be $\{10\bar{1}1\}$ and consisted of 35 pct of the boundary population in the slip-cast B_4C but only 1 pct of the boundary population in the hot-pressed B_4C . It was hypothesized that the presence of twins will cause a different failure mechanism. The effect of the twins on the stress state was examined by finite-element simulations and will also be discussed.

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I. INTRODUCTION

B_4C is used widely in abrasives, wear-resistant components, and armor applications because of its high hardness and low density. Hot pressing boron carbide (B_4C) powder is the commercial technique used to form personnel armor plates and components for various applications. It is possible for B_4C components formed by the hot-pressing technique to reach nearly full theoretical density and achieve high mechanical performance. The alternative technique of forming B_4C compacts, as described by Matsumoto *et al.*,^[1] is by slip casting, sintering, and hot isostatically pressing (HIP) the powder. The mechanical properties of these slip-cast and HIP B_4C materials were reported^[2] to be as good as the hot-pressed B_4C materials. However, the different processing routes could affect the microstructure, and the effect of microstructure on the mechanical and ballistic response is an important and common question when characterizing armor materials. Studies investigating the effect of the processing route on the mechanical properties of alumina used for ballistic applications have been conducted by Badmos *et al.*^[3] In their research, it was found that alumina with the same composition but produced by dry pressing had higher hardness and lower toughness than those produced by slip casting.

The aim here is to evaluate the mechanical properties and microstructure of the slip cast, sintered, and HIP B_4C (from now referred to as slip cast B_4C) and compare these properties with those of hot-pressed B_4C . In an effort to determine the effect of microstructure on

mechanical performance, simulations comparing the elastic response of structures (with and without the presence of growth twins) to quasi-static loading will be used to evaluate the differences between the two B_4C types.

II. EXPERIMENT

A commercially available, hot-pressed B_4C and a research grade slip-cast B_4C were obtained. The hot-pressed B_4C sample is the armor-grade reference benchmark material and has a density of 2.49 g/cm^3 , or 98.8 pct of the theoretical density. The slip-cast B_4C was measured by the Archimedes method to have a density of 2.50 g/cm^3 , or 99.2 pct of the theoretical density. The hot-pressed and slip-cast B_4C were also analyzed in a previous work. Chen *et al.*^[4] analyzed the microstructure and intergranular precipitates of the equivalent hot-pressed B_4C , and Sano *et al.*^[5] evaluated the high-strain-rate response of the slip-cast B_4C .

The samples were polished on the Struers Rotopol automatic polisher with diamond slurries of decreasing diamond abrasive size at each polishing step until reaching $0.25 \text{ }\mu\text{m}$. The samples were then polished with $0.02 \text{ }\mu\text{m}$ colloidal silica. The polished, hot-pressed and slip-cast samples were imaged on the FEI Nova Nano SEM 600 scanning electron microscope (SEM) equipped with a field emission gun. To characterize the orientation distribution and any texture differences, electron backscattered diffraction (EBSD) patterns of both B_4C types were collected (EDAX-TSL Pegasus XM4 EBSD system) at 15 kV accelerating voltage.

The Knoop hardness of the slip-cast B_4C sample was measured with a microhardness tester (Wilson Instruments Tukon 2100B) at loads from 3 N to 98 N (0.3 Kg to 10 Kg) and compared with the hardness of the hot-pressed B_4C .^[6] The dynamic uniaxial compression response of seven slip-cast samples and six hot-pressed

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samples were characterized using the Kolsky bar technique. The impact was imaged with a high-speed camera and the stress and strain values were recorded. Details of the dynamic impact test results are found in a previous publication.^[5]

III. SIMULATION

Three different finite-element (FE) studies were considered to compare the effect of the presence of twins on the response of the material to a hydrostatic compressive load. For each study, two simulations were conducted on identically meshed and identically loaded geometries: One simulation contained crystals without twins whereas the second simulation contained crystals with twins. The average von Mises stress was then calculated for each crystal boundary and compared between the corresponding simulations. Therefore, for this preliminary work, there are three studies and six simulations, and in the future, additional FE studies will be conducted to provide statistically significant results using the techniques discussed in this analysis.

For the current work, the following orthotropic elastic material constants were assumed based on the Voigt average methodology^[7]: $E_x = E_y = 482$ GPa, $E_z = 450$ GPa, $G_{xy} = 98$ GPa, $G_{yz} = G_{zx} = 90.5$ GPa, $\nu_{xy} = 0.213$, $\nu_{yz} = \nu_{zx} = 0.187$. These nonunique values were chosen to give a shear modulus of 190 GPa and a bulk modulus of 260 GPa based on the authors' familiarity with high-rate constitutive modeling (the bulk modulus was taken as the average of the bulk moduli for the two phases as discussed by Holmquist and Johnson^[8]). In future work, orthotropic elastic constants measured using resonant ultrasound spectroscopy (McClellan *et al.*^[9]) will be used instead. For the first FE study, orientations obtained from the EBSD inverse pole figure (IPF) map were used to specify the

orientation of each crystal in the model. For the remaining two studies, eight representative orientations were distributed throughout the hexagonal structure based on patterns observed from the EBSD IPF map.

For the first FE study, an IPF map from an EBSD dataset of a slip-cast specimen (heavily twinned) was used to generate a 40- μm by 40- μm mesh in ANSYS of a region containing two twinned grains surrounded by 32 grains, 3 of which are also twinned (Figure 1(a)). For the remaining two FE studies (identified later as hex cases 1 and 2), two different idealized 15- μm by 13- μm hexagonal structures with randomly oriented grains were generated; an example is shown in the inset image in Figure 1(b). The bold hexagon in the inset image corresponds to the bold hexagon in the resulting FE mesh of the compressed simulation. Eventually, a large number of these structures will be generated randomly to produce statistically meaningful results. For both cases, a single layer of eight-node linear hexahedral elements was used; the element length was approximately 0.15 μm . To avoid changes in the mesh and in keeping with the idealized hexagonal crystal representation, it was assumed that all twins would occur in one of three possible orientations. In this way, the same mesh was used for a grain without a twin as was used for the same grain with any of the three possible twin orientations. Because the mesh was identical for both cases, the same elements were considered when calculating the resulting stress states for cases with and without twins.

Identical compressive hydrostatic boundary conditions were obtained for each study by applying uniform displacement boundary conditions on each side of the model that resulted in equal net pressures applied to each face. In other words, using an iterative procedure, displacement boundary conditions were applied and the normal forces acting on all face elements were calculated. The sum of these normal forces was divided by

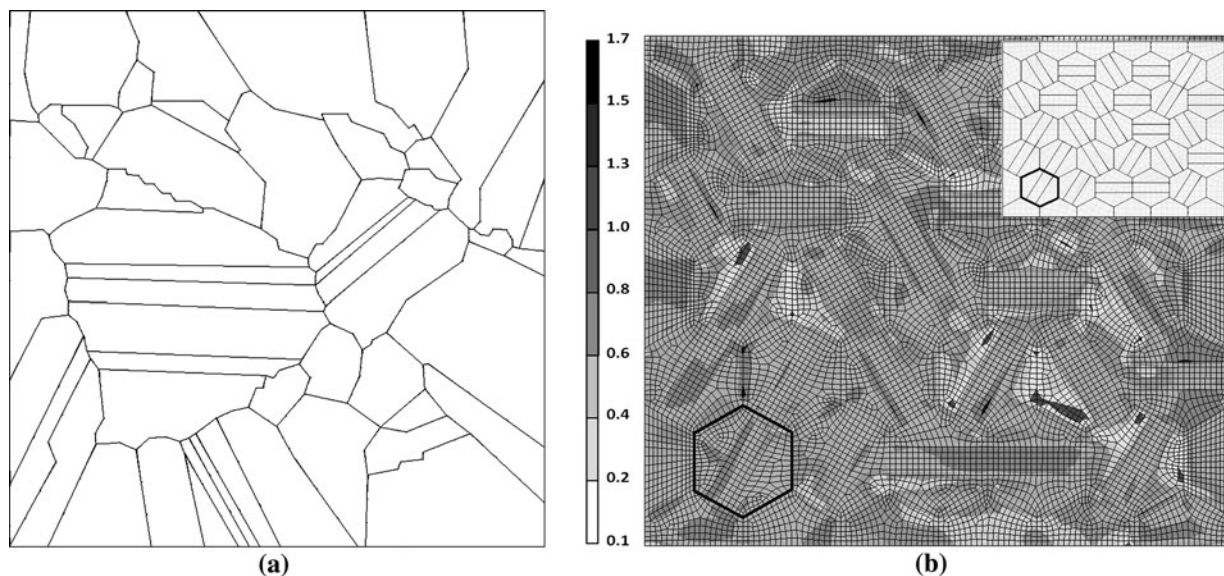


Fig. 1—(a) FE domain for EBSD image. (b) Grayscale of compression FE simulation results with corresponding structure shown in the inset image.

the corresponding face surface area, resulting in a net pressure. The surface displacements were adjusted continually until the net pressures on the three surfaces equaled the desired value. Being a linear analysis, all results are linearly dependent on the applied boundary conditions. With the final displacement boundary conditions applied, the nodal von Mises stresses from the contiguous elements forming the boundary of each grain were calculated using the built-in post-processing features of ANSYS. The average stress from all grain edge elements was used to describe the stress state of each grain for each simulation; the average stress from all grain edge elements for the simulations *without twins* was used to normalize the grain stress states for each study (*i.e.*, each pair of simulations). An additional analysis was performed on particular grains, as described in Section IV, whereby twins in the vicinity of certain grains were included or removed before

applying the aforementioned hydrostatic boundary conditions.

IV. RESULTS

The Knoop hardness curves for both samples (Figure 2) were comparable. The Knoop hardness values were compared at the 19.6 N load, the load at which the hardness becomes load independent in accordance with ASTM C1326. The slip-cast B_4C had a hardness of 21.2 ± 0.6 GPa and the hot-pressed B_4C had a hardness of 19.6 ± 1.8 GPa.

The dynamic uniaxial compression test of the hot-pressed and slip-cast B_4C with the Kolsky bar also showed comparable results. The compressive strength ranged from 3 GPa to 4.3 GPa for both B_4C types. All samples displayed through-sample cracking at the time interval just past the maximum stress peak and destruction of the sample shortly thereafter.

From the SEM images of the polished microstructures, using the linear intercept method, the grain size of the hot-pressed B_4C was calculated to be $10.2 \mu m$ and $12.4 \mu m$ for the slip-cast B_4C when not counting twins as separate grains. Although the grain sizes were similar, the microstructures were different. The SEM images also revealed numerous growth twins in the slip-cast B_4C but minimally in the hot-pressed samples. The twin plane was determined to be $\{10\bar{1}1\}$. This is in agreement with the results of Oleinik and Ostapchuk,^[10] who observed the presence of $\{10\bar{1}1\}$ growth twins in B_4C sintered or annealed below 2473 K (2200 °C) as well as Ruh *et al.*^[11] who investigated B_4C and boron nitride composites of varying compositions. The EBSD IPF maps confirmed the existence of twins in the slip-cast sample. An example IPF map in grayscale of a slip-cast sample and of a hot-pressed sample is shown in Figure 3.

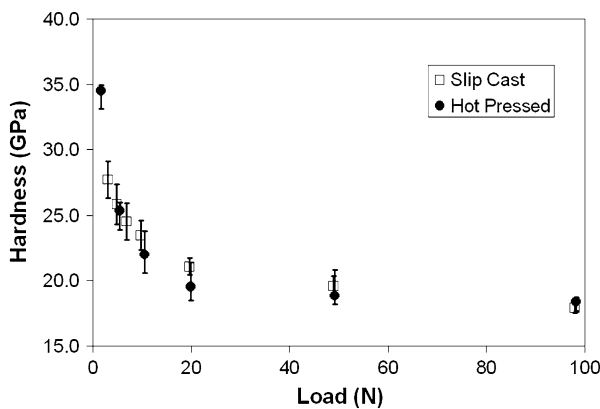


Fig. 2—Knoop Hardness of the slip-cast and hot-pressed B_4C samples.

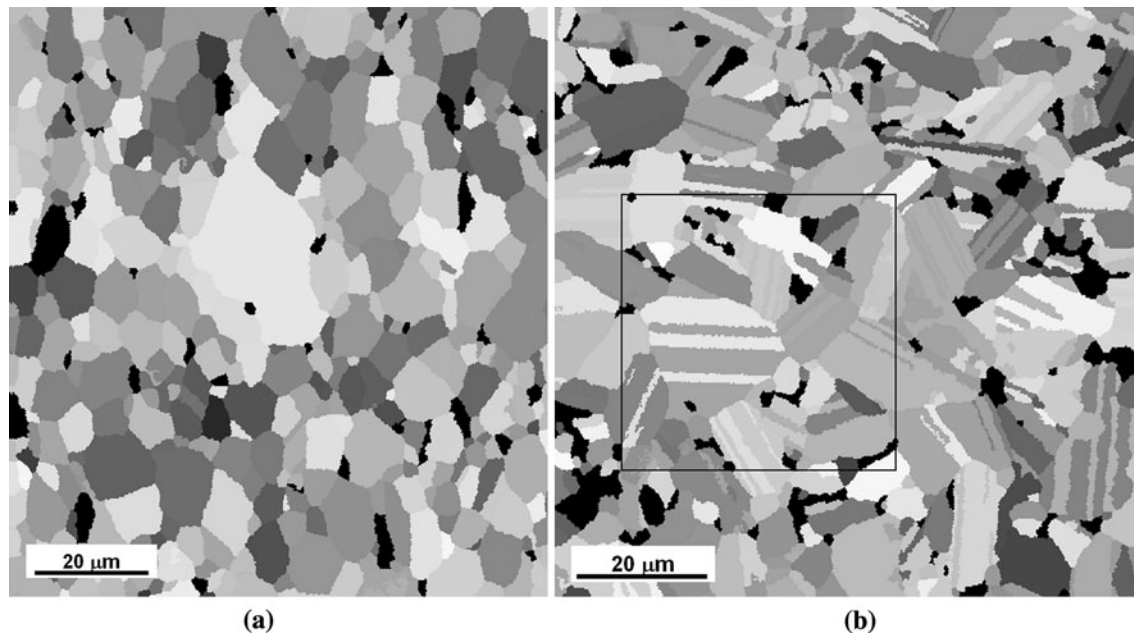
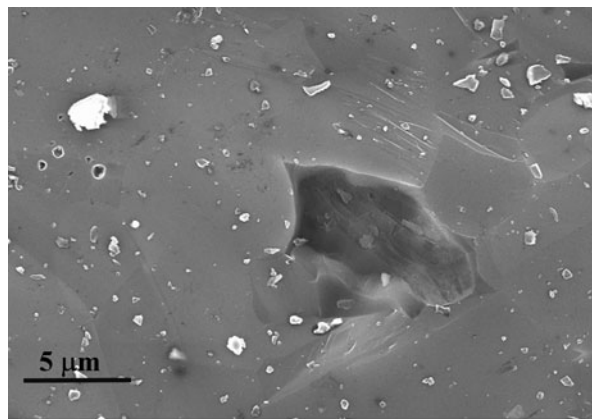
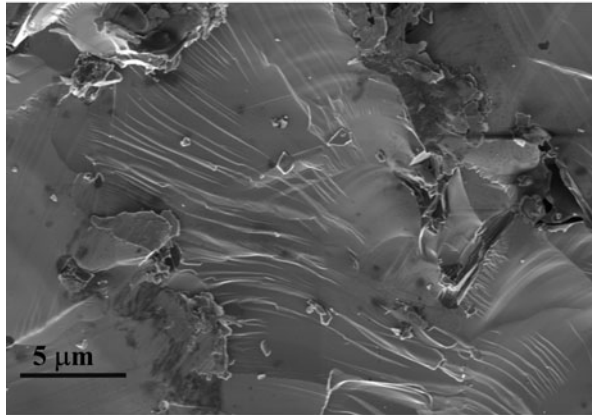


Fig. 3—EBSD IPF map of (a) hot-pressed and (b) slip-cast samples. The boxed region in (b) corresponds to the region shown in Fig. 1(a).



(a)



(b)

Fig. 4—SEM image of a fracture surface of the (a) hot-pressed B₄C and (b) slip-cast B₄C.

The twin boundary length populations (measured from more than 1.5 mm² of the carefully polished areas for each B₄C) were 35 pct for the slip-cast sample and 1 pct for the hot-pressed sample. Examining the fracture surfaces of the hot-pressed and slip-cast B₄C revealed differences in the fracture patterns. The slip-cast fracture surfaces had much rougher patterns, with hackles, numerous cleavage planes, and twins, whereas the hot-pressed fracture surfaces were relatively smooth (see Figure 4).

Figure 5 presents a summary of the data obtained from the three different FE studies that were conducted. Each data point corresponds to a single grain from each study. For the EBSD map study, the two larger center grains and eight of the surrounding grains (those not on the model edges) were considered. For the idealized grain structure (hex case 1 and hex case 2), the 23 interior grains were considered. The average von Mises stress of all the grain edge elements was used to normalize the calculated stresses of the individual grain edges, such that data points with a normalized grain boundary stress <1 correspond to average grain boundary stresses below the average, whereas data points with normalized stress >1 correspond to stresses above the average. For each grain, the average grain boundary stress was determined with twins present throughout the modeled region as well as without the presence of twins.

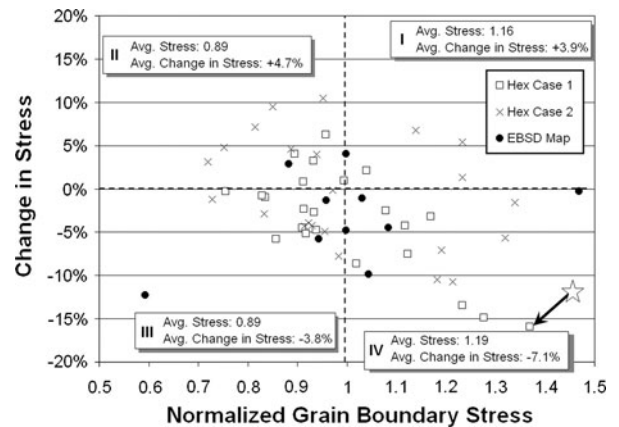


Fig. 5—Plot showing changes in the average grain boundary stress and the corresponding normalized grain boundary stress from the FE simulations.

The effect of adding twins to the model on each grain boundary is shown by the change in stress along the vertical axis. For example, the data point marked with a star corresponds to an average grain boundary stress (without any twins present) roughly 36 pct greater than the average of all grain boundary stresses measured in the simulation. As a result of including twins in each of the grains, the average grain boundary stress of that particular grain dropped by 16 pct.

For the hex case 1 simulation, there does seem to be a trend showing a decrease in the grain boundary stresses because of the presence of twins associated with higher initial grain boundary stresses. On average, a slight overall reduction (~2 pct) occurs in the average stress for each of the three cases because of the presence of twins. The results shown in Figure 5 have been subdivided into four quadrants: (I) higher than average initial stress with an increase in stress due to twins, (II) lower than average initial stress with increasing stress, (III) lower than average initial stress with decreasing stress, and (IV) higher than average initial stress with decreasing stress. The averages for each quadrant are shown in Figure 5 as well. Quadrants I and IV are the most important because these would be associated with the most important reductions in stress (quadrant IV) and the most important increases in stress (quadrant I) as a result of the presence of twins.

Because of the importance of quadrants I and IV, particular grains in these two quadrants from hex cases 1 and 2 were examined in greater detail in an attempt to refine our understanding on the role of growth twin density on the response of B₄C to quasi-static loading. For example, twinned grains associated with significant increases or decreases in stress can be analyzed by adding and removing their twins while leaving the remaining structure unchanged. In two such instances in the current study, the inclusion of a single twin led to a stress reduction in all grains surrounding the twinned grain. But in one instance, the stress in the twinned grain was reduced, whereas in the other instance, the stress was increased. The equivalent isotropic bulk modulus calculated from the various simulations was 257 GPa,

suggesting that the model size was sufficiently large to represent bulk material.^[7]

V. CONCLUSIONS

Because the density, static, and dynamic properties were comparable, it was hypothesized that the microstructure, and more importantly the presence or lack of twins, was the cause of the different fracture mechanisms. To investigate the hypothesis, the orientation data and orientation statistics were applied in finite-element simulations of the microstructure under hydrostatic stress. Although the current work is still ongoing, noticeable reductions in stress occur, which result from the addition of twins. It is anticipated that a larger series of numerical studies will shed more light on the role growth twins may play, and it is the hope of the authors that this study will give insight into possible improvements in B₄C fabrication techniques.

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