Anisotropy of mechanical properties in a hot-pressed boron carbide

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Abstract

Effects of microstructure and material properties on the mechanical behavior of hot-pressed boron carbide are presented. The microstructure and intrinsic microstructural inhomogeneities have been characterized using scanning electron microscopy characterization techniques (SEM/EDS/EBSD). In-situ mechanical characterizations of the boron carbide microstructure and its larger inhomogeneities have been performed by nanoindentation. Macroscopic dynamic and quasi-static compressive responses have been studied in two characteristic orientations (parallel and perpendicular to the hot-pressing direction) using a modified compression Kolsky bar setup (strain rates of 10^2 – 10^3 s^{-1}) and standard MTS test machine (strain rates of 10^{-4} – 10^{-3} s^{-1}). The microstructure characterization showed that boron carbide has a fine-grained microstructure with a complex superposition of non-metallic inclusions, such as free carbon, AlN and BN. Nanoindentation tests conducted in three principal planes of the plate revealed an anisotropy of the mechanical properties. The compression tests revealed that the strength of this hot-pressed boron carbide is orientation-dependent. Detailed SEM analysis indicated transgranular fracture and micro-

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cracking originating at large carbon inclusions. Influences of microstructural anisotropy on the mechanical response of the material is discussed.

Keywords: Boron carbide, Microstructural characterizations, Nanoindentation, Kolsky bar technique, Brittle failure

1. Introduction

Boron carbide (BC) is a material that has gained much attention for use as an abrasive material, neutron absorber, and its potential in defense applications, because of superior hardness, low density and potential for significant improvements [1, 2]. Over the past several years, several shock and impact studies on BC ceramics have been performed, which exhibited significant variability in the reported results [3, 4, 5, 6, 7]. In most cases, the discussion of the mechanisms associated with dynamic and shock events were not supported by post-test microstructural observations. Additional quantitative and qualitative studies of the BC microstructures are needed to fully understand the dynamic behavior of this class of materials, and to establish the processing-microstructure-properties relationships.

A commercial hot-pressed BC is manufactured by applying heat and pressure to a powder. This process eliminates most of the internal voids; however, it is difficult to reach full densification under these conditions. Thus, it has been common practice to add sintering additives to enhance densification of BC powder [1, 8, 9, 10]. Non-oxide additives, such as free carbon, also improve this process [10, 11, 12]. However, the more additives in the powder, the larger the probability that secondary phases and/or precipitates will form at the grain boundaries or within the grains [12, 13]. If the properties of these inhomogeneities are very different from those of BC, inhomogeneous stress fields will likely develop in the material in response to loading. Consequently, cracks can originate from these highly stressed regions and lead to massive failure [14, 15].

The purpose of this study is to illustrate the relationship between the processing, microstructure characteristics, and dynamic response of BC to high

rate loading. Using a commercially available hot-pressed BC, this paper focuses on experimental studies regarding the relation between the properties and the complexity of the microstructure. We characterize the mechanical properties of BC by probing the material using the nanoindentation technique, and relate this to the dynamic behavior and the failure mechanisms using post-test microstructural observations.

2. Experimental procedure

The studied material was a plate of hot-pressed BC (CoorsTek Vista Operations) that was 8 mm thick with a density of 2.51 g/cm^3. Microstructural characterization was performed on a prismatic sample with dimensions of 10×10×8 mm. The sample was cut in accordance with the principal directions ($X_1 \times X_2 \times X_3$) of the BC plate, where X_3 is the hot-pressing direction (and the thickness of the plate), while X_1 and X_2 are principal directions lying in the hot-pressed surface, as illustrated in Fig. 1. The sample was prepared in two steps. First, the three different faces of the sample, which represent three principal planes of the BC plate, were polished down to 0.5 µm using diamond-lapping films. Subsequently, in order to remove damaged surface layers caused by the mechanical polishing and reduce the roughness of sample surfaces, the sample was ion-milled using a Fischione 1060 SEM Mill ion-milling system at 4.5 keV ion beam energy for 15 min. with a 2° specimen tilt angle.

The microstructure analysis was performed using a TESCAN MIRA3 field emission Scanning Electron Microscope (SEM) equipped with a fully automated electron backscatter diffraction (EBSD) analysis system and Energy Dispersive Spectroscopy (EDS) capabilities. The TEAM$^{\text{TM}}$ software (EDAX, Inc.) was used for chemical analysis of the sample. The resulting EBSD maps were analyzed with the OIM$^{\text{TM}}$ software from TexSem Laboratories (TSL). The BC material in the OIM software was defined using a rhombohedral lattice system, $R\bar{3}m$ symmetry space group, and lattice constants (a = 0.5653 nm and c = 1.215 nm), as reported in [16]. In addition, a validation procedure using single

crystal BCs of known crystallographic orientations was used to ensure that the crystallographic orientation of grains is indexed properly. The inhomogeneities (i.e., possible secondary phases, inclusions or grain boundary films) were not the subjects of interest in the EBSD analysis. The mapping was performed in the X_1–X_3 plane with a step size of 0.4 µm. The chemical analysis of the sample was carried out with an operating voltage of 15 keV and minimum spot size of 0.5 µm. The characteristics of inclusions were also quantified by processing optical micrographs with an image analysis package in Matlab software (MathWorks, Inc.). The micrographs were obtained using a Zeiss optical microscope with an AxioCam MRC camera. A detailed description of the quantification method for inclusions is provided in [14, 15].

Nanoindentation tests were performed on an MTS Nanoindenter XP system using a Berkovich diamond pyramid tip at room temperature in air. The load versus indentation displacement normal to the exposed surface was measured during each experiment, and the mechanical properties were extracted using the approach proposed by Oliver and Pharr [17]. Hence, the hardness (H) is defined as

$$H = \frac{P}{A_c}$$

where P is the peak load and A_c is the projected contact area between the indenter tip and the specimen at the peak load. For a Berkovich indenter, A_c is determined from the relationship

$$A_c(h_c) = 25.4h_c^2$$

where h_c is the contact indentation depth determined from the load–displacement curve.

Assuming that the elastic properties of the sample are independent of indentation depth, the modulus of the sample, E, is calculated from the following formula

$$\frac{1}{E_r} = \frac{1-v^2}{E} + \frac{1-v_i^2}{E_i}$$

where E_r is the reduced modulus, ν is the Poisson's ratio of the sample, and E_i and ν_i are Young's modulus and Poisson's ratio of the indenter (E_i=1141

GPa and ν_i=0.07), respectively. The reduced modulus is calculated from the unloading data as

$$E_r = \frac{\sqrt{\pi}}{2\beta} + \frac{S}{\sqrt{A_c}}$$

where S is the contact stiffness, and β is a constant (β=1.034 for a Berkovich indenter).

Both E and H were continuously measured as a function of the indentation depth using the Continuous Stiffness Measurement (CSM) technique. The procedure involved the following steps: (1) choosing the position of the indentation in such way to avoid interaction with inclusions visible on the surface of the sample; (2) impressing the indenter in 5 cycles of loading/unloading (over a period of 10 s under each cycle) until the pre-specified value of the maximum load was attained; (3) holding the indenter in this position for 5 s; and (4) smoothly withdrawing the indenter from the specimen. The sample was indented in two sets of experiments to a maximum load of 200 mN and 400 mN, respectively. At least twenty-five indentation tests were performed under each experimental condition. The nanoindentation technique was also used to study the mechanical properties of the inclusions. However, these tests were only conducted in the X_1–X_2 plane due to the limited area of the inclusions for a valid indentation test in the X_1–X_3 and X_2–X_3 planes. The procedure involved 10 cycles of loading/unloading until the maximum load of 200 mN was reached.

Prior to testing, the instrument was calibrated on a standard specimen of fused silica in order to provide assurance of the indentation system for property measurement. This process consisted of two steps: the tip calibration (also called the area-function calibration) and validation of the system through the nanoindentation tests (the indenter tip was driven 1000 nm into the specimen). Also, note that the key assumption of the nanoindentation technique is that Poisson's ratio of the material being tested is known a priori. Here, it is assumed for the moment that Poisson's ratio of BC material is the same in three principal directions of the specimen and equal to 0.17. The same value is assumed for investigated inclusions. The instrument, once calibrated, was finally

validated on the investigated BC sample. That is to say, 5 nanoindentation tests were performed to a maximum load of 500 mN in the X_1–X_2 plane to measure the projected contact area of nanoindentation by the SEM technique. These measurements were in good agreement with the theoretical values. Post-experiment, the SEM was also used to examine the quality of indents.

The mechanical response of this BC was studied under dynamic loading conditions using a modified compression Kolsky bar setup and prismatic (rectangular cross-section) specimens at strain rates of $10^2 - 10^3$ s^{-1}. The specimens were cut from a plate in two principal directions of the plate (i.e., parallel and perpendicular to the hot-pressing direction). The dimensions of the specimens were nominally 3.5×4×5.3 mm. A more detailed description of the Kolsky bar experimental setup and testing procedure for ceramic materials is provided in [18]. A standard MTS test machine was used to apply compressive loads to specimens (of the same dimensions as in the previous case) under quasi-static conditions (strain rates of $10^{-4} - 10^{-3}$ s^{-1}). Post-experiment, the fragments of the specimens were collected and investigated using the SEM technique to identify failure mechanisms.

3. Experimental results

3.1. Microstructure

A schematic three-dimensional representation of the microstructure reconstructed from optical micrographs with labelled coordinate system is presented in Fig. 1. The microstructure is densely populated with inhomogeneities (identified later as free-carbon-rich inclusions, AlN, BN and pores). Most of them have rather irregular shapes. However, the larger inhomogeneities (mostly free carbon), can be described as having a flake-like geometry. As seen in the figure, there is a significant number fraction of small-size inclusions. The fraction of large inclusions (i.e., larger than 10µm) is small but important (it is likely that first failures initiate at locations with the highest stress concentrations, such as large inclusions). These large carbon inclusions have a strong preference in the

orientation of the major dimension with respect to the X_3 axis (the major axis is oriented almost perpendicular to the hot-pressing direction). However, this is not the case for the second population, which has a much weaker orientation dependence of inclusions. This distinction arises because of the processing route. The average number-weighted size of inclusion is estimated to be ∼2 µm, while the average area-weighted size is ∼7 µm (determined based on image analysis of optical micrographs in the X_1–X_3 plane). The characteristics of the inclusions are described in detail in our previous study [15].

Figures 2(a–d) show a combined SEM/EBSD/EDS analysis of BC in the X_1–X_3 plane. A large population of inclusions has been examined on the polished surfaces on the sample (Fig. 2(a)). Many of them are larger than the average size of inclusion discussed earlier. The following EBSD investigation reveals the crystallographic structure of the material (Fig. 2(b)). This structure consists of fine grains with a rather weak crystallographic preferred orientation. Note that the local texture characteristics might not correspond to the macroscopic texture. The grain boundary misorientation angles are typically larger than 15°. The measured average area-weighted grain size is approximately 5 µm. However, several larger grains (> 25 µm) can be seen on the map. Figures 2(c–d) present the EDS analysis for the same area, where the distribution of Boron, and an overlay of Carbon, Aluminum and Nitrogen elements are shown in Fig. 2(c) and Fig. 2(d), respectively. Three different characteristic inclusions can be distinguished from the maps: (i) carbon-rich inclusions, (ii) AlN and (iii) BN. Carbon in BC materials can be present in different kinds of structures, including amorphous-like grain boundaries [19] or crystalline forms at triple-junction points [12, 20]. In this BC, graphite-like inclusions were found to be dominant for fracture initiation among the whole population of inclusions and their influence on the dynamic mechanical behavior must be taken into account.

3.2. Nanoindentation

Figure 3 shows typical load–displacement curves of the BC (matrix) material and large flake-like carbon inclusions (within the matrix material) collected from

experiments performed in the hot-pressing direction and up to 200 mN peak load. In the case of the polycrystalline matrix material, most of the collected loading and unloading curves were continuous and smooth without remarkable pop-in and pop-out observations. This might suggest that the material experiences elastic–plastic deformation without significant fracturing. However, small hysteresis loops between unloading/reloading cycles were frequently observed for 100 mN peak loads and higher. See, for example, the load cycle of the BC matrix material indicated by an arrow in Fig. 3. Such hysteresis loops are present if a considerable change in volume take place during the nanoindentation test. In BC materials, these changes in volume would most likely originate from nanofracture or amorphization. The missing loops for the first 2 load cycles and the gradual increase of the loop area for higher loads suggest the first scenario, i.e. nanofracturing. Indeed, the post-experiment SEM investigations reveal well developed indentation cracks. It seems that these cracks form relatively quickly, and gradually grow during each loading cycle. A complementary experimental investigation showed that the first radial cracks, which propagated from the corners of the indents, can develop at the peak load of 100 mN. Subsequently, the crack lengths are observed to gradually increase with increasing indentation load. A micrograph of the indentation imprint with a well-developed radial cracks is also present in Fig. 3. The SEM image was taken for the nanoindentation test performed for the validation of the Oliver and Pharr method (see the *Experimental procedure* section for more details).

In the case of the large carbon inclusions, the load–displacement curve has two different stages. In the first stage, a slight increase in load is accompanied by a significant indentation displacement. In the later stage, the shape of the slope is very similar to that in the BC material. The reason for this is likely that the flake-like carbon inclusion has a finite thickness in the hot-pressing direction (Fig. 1 and Fig. 2). If the penetration depth exceeds the thickness of the inclusions, then the measured mechanical properties are a combination of both the inclusion and BC material. This makes the analysis of the measurements more complicated. Note that the thickness of the inclusion will probably be

different for each indentation test, and the reported E and H values correspond to different indentation depths (and load cycles) in each case. Therefore, the very last load cycle that most likely correspond to the inclusion response is discussed later in the paper (see Fig. 3 and the load cycle indicated by an arrow). Up to this point, the pop-in and pop-out observations were very common for the inclusions, which causes an additional scatter of the measured mechanical properties. Wide hysteresis loops were also observed during these nanoindentation tests. The most likely reason for this behavior is the severe delamination of the inclusions. Note that the carbon inclusions have mostly inhomogeneous graphite-like layer structure. Therefore, when the inclusion is penetrated by the indenter tip, cracks can easily form under the indenter tip and propagate across the interfaces between different layers.

The elastic modulus and hardness of this BC matrix material as a function of indentation load are presented in Figs. 4(a–b) in each of the three principal directions of the specimen. The results show a definite mechanical property anisotropy linked to the hot-pressing direction. Indentation in this direction clearly shows a statistically significant lower E than in both in-plane directions of the plate. However, the differences for H are not clearly statistically different, but the trends do indicate a similar difference. Consequently, the indentation depth is also larger in the hot-pressing direction (i.e. X_3) as compared to both perpendicular directions. The average measured values of the indentation depth at 400 mN peak load were 839±6 nm, 838±6 nm and 904±12 nm for X_1, X_2 and X_3 directions, respectively. Note that the measured H and E are consistent for X_1 and X_2 directions. These results all suggest that BC is stiffer in both in-plane directions of the plate than in the plate thickness direction. The results also show that the indentation loads have a significant influence on the measured mechanical properties, which are higher at lower peak loads. The related standard deviations are also higher at lower loads, decreasing with increasing peak load.

Such data variations at very low loads are usually due to the so-called load-size effects, and have been observed in BC single crystals [21] and many other

brittle materials [22, 23, 24, 25, 26, 27]. Domnich *et al.* [21] suggested that a load of 100 mN or higher is required for the proper measurement of BC single crystal properties. The scatter of the measured values was attributed to a combination of the surface roughness and imperfections of the indenter's side angles and tip sharpness. The indenter tip shape encumbered with geometric errors will lead to the uncertainty in measurements when using the Oliver and Pharr method. In this study, the relationship between the indentation load and E for X_1 and X_2 directions is of the same nature as those presented in [21]. Therefore, it is also assumed that one has to be careful when interpreting the results of BC materials performed at load less than 100 nN. The results for the X_3 direction show, however, more decay than two other directions. There are two possible explanations for this difference. First, the more decay in the X_3 direction might suggest a larger probability of interactions with the neighboring inclusions under the indenter tip. This is because of the larger area of the flake-like carbon inclusions in the X_1–X_2 plane. In that case, the measurements most likely correspond to the *composite* response rather than to the BC material. Further experimental efforts, such as the focussed ion beam (FIB) lift-out method, could be performed to support this hypothesis. The second possibility is that there is a preferential orientation of the BC grains with respect to the hot-pressing direction. Note that the standard deviation of the measured properties at 400 mN are of the same magnitude for each direction of the BC sample. Although the EBSD investigation has not revealed any evidence of a strong crystallographic texture, the strong ($\sim$10:1) anisotropy of the BC crystal implies that minor textures can lead to anisotropy of the polycrystalline material.

The scatter in the indentation results also suggest that the crystallographic orientation of the grains in the specimen may have had a statistically important influence on the measured values. Note that the indenter tip is most likely probing a single grain at the very small load. This might result in variability of the measured properties since the Young's modulus of BC is strongly orientation-dependent on the prismatic and the pyramidal planes [28]. This variability likely

gradually decreases at each loading cycle because the indentation area gradually increases, and the measured properties at large indentation depths likely corresponds to 4–5 grains (based on the ∼6 µm edges of the indented polygon at 400 mN, as measured by SEM. Other sources of variability include surface roughness, material inhomogeneities under the indenter tip and temperature fluctuations. Whether it is the anisotropy of BC crystals or the *composite* response, the nature of this anisotropy remains unclear and needs to be explored in future studies.

Figure 5 shows hardness and elastic modulus for carbon inclusions as a function of indentation load in the hot-pressing direction. Each data point corresponds to the last accurate load cycle of a single test, as discussed earlier and indicated in Fig. 3. Both E and H values are significantly lower than the values reported for the BC material. The scatter of the indentation modulus might result from surface roughness, severe cracking of graphite-like layer structure, variations in the chemical composition of the inclusions or temperature fluctuations. The measured properties of graphite-like layer structure might also be different in the in-plane directions of the plate due to the structural anisotropy (not explored in this study). The structure (amorphous-like or crystalline forms), porosity, and apparent (bulk) density of each individual inclusion will also affect the overall measured properties. Note that the measurements performed on the inclusions in this study cannot be compared with those of commercially made and pure graphite materials [29]. This is because of small material volumes being probed, lower level of purity, and the dynamic *confinement* from penetration-dependent interactions with the surrounding BC matrix. It is also generally accepted that different hardness techniques are difficult to compare due to different scales and being influenced by different properties. In spite of that, however, this elastic properties mismatch is a significant factor in developing stress concentration in the BC structure under static and dynamic loading conditions. The stress concentrations can initiate cracks, and consequently contribute to the failure of the structure.

3.3. Strength and failure mechanisms

Figure 6 presents uniaxial compression strength measurements in dynamic and quasi-static regimes for loading along two representative axes of the plate. The most prominent finding is a lower strength value measured in the hot-pressing (X_3) direction of the plate at all rates examined. The second general observation is that slightly higher strength values, although not statistically significant, are observed under dynamic loading conditions. The first observation is believed to be related to the preferential orientation of flake-like carbon inclusions. In our previous study [15] it was found that large carbon inclusions are the most favorable nucleation sites for cracks because of their relative size and aspect ratio. In that case, the material was dynamically loaded in the hot-pressing (X_3) direction, and it was shown that the orientation of these inclusions favors the nucleation of cracks through the so-called wing-crack mechanism [30, 31, 32]. This can be explained by the 'sliding' of the carbonaceous graphite-like structure related to its low coefficient of friction. Such wing cracks were also observed on the fracture surfaces of the samples compressed in the hot-pressing (X_3) direction in this study, and are discussed later. In the case of X_1/X_2 loading directions, Hogan and co-workers [33] have suggested that the failure process is still controlled by flake-like carbon inclusions, but it is more difficult to activate cracks from carbon inclusions oriented parallel to the loading direction.

The flake-like inclusions also have an indirect effect on the observed strain-rate sensitivity of the material. Several theoretical studies have shown that the strain rate dependence of the peak (failure) stress of brittle materials is strongly dependent on the characteristics of such defects [33, 34, 35, 36, 37]. The rate dependence was also observed experimentally in many advanced ceramics, including BC [34, 38, 39, 40].

In high-purity ceramic materials, the observation of transgranular or intergranular fracture surfaces is dependent in most cases on the grain boundary strength. In the case of intergranular fracture, the crack propagates along the grain boundaries. Consequently, strong grain boundaries tend to lead to trans-

granular fracture. The transgranular mode of fracture is sometimes characterized by cleavage and cleavage steps, as observed in this study on all fracture surfaces after the tests. Such an example of the fracture surface at high magnification is presented in Fig. 7. This SEM micrograph also shows a large flake-like carbon inclusion (labeled 'C_I' in Fig. 7), which played a major role in the failure process. Note the cracks growing from both sides of the large carbon inclusion, that interact with the smaller inclusion (labeled 'C_{II}') having the same flake-like layer structure. Here, the normal to the overall fragment surface is perpendicular to the hot-pressing (and loading) direction since the carbon inclusions are almost perpendicular to the surface. This canonical example of the wing crack formation was observed for all tests performed in the hot-pressing (X_3) direction. As discussed above, the number of inclusions that are likely to have been the crack initiators is significantly smaller in the case of in-plane loading directions. Thus, the orientation, size and distribution of flake-like carbon inclusions affects the local stress field within the material, and thereby the whole failure process. Note that McCauley reported similar anisotropy in resistance to both initiation and propagation of microcracks in Ba-Mica/Al_2O_3 with a microstructure similar to that of the BC investigated in this study [41, 42]. It is therefore important to develop strategies for processing BCs that overcome the tendency of additives to form large inclusions in order to improve the dynamic behavior of this class of materials.

4. Summary

Microstructural characterizations and mechanical properties of hot-pressed BC have been studied experimentally by SEM/EBSD/EDS analysis, nanoindentation and compression tests performed at different strain rates. The results have shown a noticeable difference in the developed strength in two principal orientations of the BC plate (parallel and perpendicular to the hot-pressing direction). This was related to the elastic anisotropy of the constituent BC crystals and to the mismatch of the in situ properties between BC crystals and

process-induced inclusions. A detailed SEM study of the fracture surfaces have revealed that the transgranular fracture is a dominant failure mode in this BC material. It has been shown that large carbon inclusions are involved in the fracture process, and their sizes and orientations in relation to the applied load play important roles during failure. The influence of microstructural anisotropy on the mechanical response of the material has been discussed. These studies have demonstrated that the anisotropic compressive strength is related to the anisotropy of elastic properties and to orientation of large carbon inclusions, from which cracks of tensile character can develop. The results provide new insight into the relationship between the microstructure, process-induced inclusions and mechanical response of hot-pressed BC. These results can also be used to support materials processing and manufacture of improved BCs.

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List of Figures