Micromechanisms associated with the dynamic compressive failure of hot-pressed boron carbide

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Abstract

Brittle failure in boron carbide has been studied in dynamic uniaxial compression using a Kolsky bar technique. A detailed study of fragments was performed using SEM-EDS, to identify the mechanisms responsible for failure. Microstructural characterization and fracture surface observations revealed that carbon inclusions oriented at certain angles with respect to the direction of loading might act as possible crack initiation sites. Cracks developed from these inhomogeneities had a tensile character, and were linked to the wing crack mechanism.

Keywords: Boron Carbide, Brittle Failure, Wing Cracks, Kolsky Bar Technique

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Boron carbide is an attractive advanced ceramic because of its low density, excellent hardness and wear resistance [1, 2]. As with many other ceramics, the processing of pure boron carbide to high densities requires different additives for better densification [1, 3, 4, 5]. The consolidated material can also contain non-oxide impurities like free carbon, which acts also as a sintering aid [5, 6, 7]. All of these can form secondary phases or precipitates at the grain boundaries or within the grains. Cracks can initiate from these inhomogeneities, and their subsequent propagation is strongly coupled to the highly inhomogeneous and evolving fields ahead of the crack tips, and perhaps by other cracks in the

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vicinity. These failure mechanisms have been extensively studied for many years in failure of brittle solids [8, 9, 10, 11, 12]. However, fundamental issues related to the dynamic nucleation and propagation of cracks are poorly understood for advanced ceramics.

In previous work [13], we investigated the rate-dependent compressive failure and fragmentation of a hot-pressed boron carbide using quantitative fragment analysis. In the current study, we investigate the impact of the pre-existing microstructural inhomogeneities on microcracking and failure mechanisms in hot-pressed boron carbide through a detailed microstructural characterization. The experiments were conducted using commercial hot-pressed boron carbide (CoorsTek, Inc.) with a density of 2.51 g/cm^3, and equiaxed grains with an average size of $\sim$15 µm, as provided by the manufacturer. Prior to testing, the microstructure was characterized to determine the mesoscale inhomogeneities of interest by using a TESCAN MIRA3 field emission Scanning Electron Microscope (SEM) coupled with Energy Dispersive Spectrometry (EDS). A chemical composition analysis was carried out with the resolution of 0.5 µm. Identified microstructural inhomogeneities such as carbon, aluminum nitride and boron nitride inclusions were afterwards quantified and measured by taking a series of optical microscopy images treated with image processing tools developed in Matlab software (MathWorks, Inc.) [13].

The Kolsky bar experimental setup used in this study, and the testing procedure for ceramic materials is presented in [14]. The compression specimens (with dimensions of 3.5 mm $\times$4 mm $\times$5.3 mm) were cut from a plate 8 mm thick with the loading axis parallel to the hot-pressing direction. For the purposes of subsequent discussion, the coordinate system (X_1, X_2 and X_3) is associated with the specimen in the following manner: X_3 is the hot-pressing (and compressive loading) direction; X_1 and X_2 are principal directions lying in the hot-pressed surface. The dynamic compression tests were carried out at a strain-rate of $\sim 10^3$ s^{-1}. To understand the failure mechanisms, the collected fragments of the specimens were investigated by SEM-EDS and optical microscopy with image processing.

Figures 1(a—b) show optical micrographs of the microstructure in two representative planes, where (1a) shows a plane normal to the hot-pressing direction and (1b) shows a plane through the thickness of the material and containing the hot-pressing direction (vertical in this case). Three different characteristic inhomogeneities can be distinguished from these images: free carbon (labeled 'A'), other non-metallic inclusions, identified later as aluminum nitride and boron nitride (labeled 'B'), and pores (labeled 'C'). Their three-dimensional shapes can be deduced from these figures. The carbon inclusions of larger size can be described as having a flake-like geometry, whereas the smaller size inclusions have rather irregular shapes and are located at triple junctions and grain boundaries. Similar irregularity was observed in the case of other non-metallic inclusions. It should be noticed that for most of the large carbon inclusions the major axis is oriented almost perpendicular to the hot-pressing direction (Fig. 1b). This will have important implications for the process of failure, as discussed later. The pores, typically smaller than 1 µm and angular in shape, were observed either individually or arranged as clusters. These observations are in line with other boron carbide materials processed by hot-pressing and investigated elsewhere [7, 15].

Inclusion morphology and statistics were quantified as follows. A total of 350 images covering an area of $\sim$20 mm^2 was examined. The measurement of the minimum inclusion size in the size distribution was limited by the resolution of the optical microscope, and is $\sim$0.5 µm. The shape factor of the inclusion was determined based on its aspect ratio, $R=a/b$, where a is the longest chord and b is the longest transverse chord (orthogonal to the major chord). Inclusions were also characterized by their surface area, motivated by the irregular shapes of the inclusions. The area of the inclusion was determined by a digital image processing system counting all pixels contained in the inclusion. The inclusion contribution (defined by the number and area fraction) to the inclusion size distribution (described by the major chord) was determined by the number/area of this inclusion divided by the total number/area of all inclusions.

Figure 2a shows inclusion number/area fraction versus inclusion size distribution as determined by the image analysis. There is a noticeable difference between these two distributions (number fraction and area fraction), as is to be expected. A large number of small-size inclusions present in the microstructure significantly contribute to the number-based distribution, whereas their measured total surface area is small in the area fraction distribution. The average number-weighted size of inclusion was estimated to be approximately 2 µm, while area-weighted size was about 7 µm. Regardless of the distribution of the inclusions size, the vast majority of identified inclusions were smaller than the average boron carbide grain size (which is ∼15 µm). The fraction of large inclusions is small, but important.

Figure 2b shows a scatter plot of all identified inclusions, showing a correlation between their aspect ratios and sizes. The plot shows a trend, where the average aspect ratio increases as the inclusion size increases. This is largely because the population of larger size inclusions is dominated by flake-like carbon inclusions. Indeed, light microscopy observations revealed that only these inclusions were larger than boron carbide grains. The average aspect ratio for inclusions with the size smaller than the area-weighted mean size was approximately 2.5 (aspect ratios smaller than 2.5 are observed for 80% of the entire inclusion population).

Figure 2c shows the orientation of identified inclusions in relation to the hot-pressed surface. In the case of inclusions with aspect ratio smaller than 2.5, the distribution of orientations is close to random, with a slight orientation preference in relation to X_3 axis. The rest of the population shows much stronger preference in the orientation of the major dimension. Roughly, more than 90% of all inclusions with aspect ratio 2.5 or higher are oriented within 45° in relation to X_3 axis. Such a morphology can cause marked anisotropy in resistance to both initiation and propagation of microcracks [16, 17].

All specimens under uniaxial dynamic compression failed by axial splitting, where cracks developed parallel to the compression axis first, and followed by transverse cracks contributing to the further fragmentation of the sample. More detailed discussion of the failure mode of boron carbide under uniaxial compression, and visualized using a high-speed camera is provided in previous work by Ramesh and co-workers [13, 18]. The compressive failure strength measured during these tests was 3.73±0.32 GPa. These strength results are of the same order as those obtained by Sano [15] for similar hot-pressed boron carbide at loading rates of the same order of magnitude.

A typical fragment collected from a dynamic test is shown in the SEM micrograph in Fig. 3a, with one boxed area of interest shown at higher magnification in Fig. 3b. Examinations of the fracture surface revealed cleavage planes. As a general observation, transgranular cleavage appears to be the primary crack propagation mode, whereas intergranular cracking was rarely observed. This indicates that (at least at these strain rates) the material has strong grain boundaries.

Figs. 3(c–d) present EDS results for the specific area shown in Fig. 3b, where the boron distribution is shown in Fig. 3c, and an overlay of Carbon, Aluminum and Nitrogen is shown in Fig. 3d. There are many small B-type inclusions present in the flat regions of the fragment (B-type inclusions correspond primarily to aluminum nitride and boron nitride). It appears that these B-type inhomogeneities do not affect the failure path, in that the crack propagates through the inclusion along the same plane as the neighboring boron carbide grains. The bonding between the boron carbide grains and these inclusions thus appears to be strong. In the case of free carbon, the interaction with the propagating crack appears to depend on the size of the inclusion. For example, most smaller carbon inclusions present at grain boundaries behave similarly to the aluminum nitride and boron nitride inclusions, and do not significantly affect the crack path as observed on the flat regions. When the carbon inclusion is relatively large compared to the surrounding boron carbide structure, the crack seems to be deflected, the fracture surface is rougher, and characteristic stair-step-like failure can be distinguished at the edges of the fragment. The size of carbon inclusions associated with this characteristic failure surface is larger than 10 µm.

This characteristic failure is presented in Fig. 3e, which is a magnified view of the rectangular boxed region in Fig. 3b. The figure presents two cracks, which initiated at the carbon inclusion (labeled ‘A’) and propagated through the boron carbide grains. In both cases, cracks extension occurs along a plane close to parallel to the compression axis, and the tip of the crack (labeled ‘CT’) can be distinguished in the vicinity of the inclusion. It appears that the larger inclusions (and regions populated by inclusions of significant sizes) act as stress concentrators and contribute to micro-crack nucleation and macro-crack development in the boron carbide structure. Another interesting observation is that the macro-crack propagation direction is typically deflected towards a large carbon particle located in the vicinity. Indeed, in many instances, when the crack propagated through the carbon inclusions in the boron carbide matrix, such a deflection or shear through the inclusion was observed (labeled ‘CD’). This suggests that the interface strength between the matrix and large carbon inclusions is weaker than the grain boundaries or other non-metallic inclusions (labeled ‘B’). The last characteristic microstructural inhomogeneities, the pore clusters (labeled ‘C’), do not seem to contribute significantly to failure at this strain-rate and for this macroscopically uniaxial compression stress-state. This is based on our observations that when the crack propagates through regions that contain individual pores or clusters of pores, the orientation of the cleavage plane remains unchanged.

It is not surprising that inhomogeneities present in the microstructure lead to premature failure of the material. In hot-pressed boron carbide, like in many other ceramics [14, 19], carbon inclusions (typically generated as a results of processing aids) have a significant influence on the material performance. Consequently, one mechanism of failure that these ceramics have in common is the microcracking from carbon inclusions. The activation of this mechanism depends on the size and orientation of these inclusions with respect to the loading direction. Knowledge of the distributions of these defects is thus important for models that seek to understand and improve these ceramics, and so the quantitative characterization of these distributions (as in this paper) has general utility. Note that the orientation of carbon inclusions in relation to the compression axis and the crack path from these inclusions (Fig. 3e) is consistent with the so-called wing crack mechanism [20, 21, 22]. Moreover, our fracture surface observations revealed many examples of microcracks having similar geometric characteristics to the idealized wing crack. The illustrative example is presented in Fig. 4. In such a case, the crack propagates by the extension of the initial inhomogeneity along a curving path, which gradually becomes parallel with the direction of compression. This mechanism has been observed and discussed extensively for macroscopic brittle solids with pre-existing cracks under quasi-static compressive conditions [20, 21, 23, 24]. The wing crack mechanism under dynamic conditions has also been studied theoretically and numerically [25, 26, 27], but has not been directly observed before this work, largely because of experimental difficulties arising from the large crack densities associated with catastrophic dynamic failure. Also, very little experimental work has been done on wing crack formation from the microstructure standpoint. This study, however, provides some direct evidence of the formation of wing-crack-like features from pre-existing inhomogeneities in the microstructure under dynamic compression.

Finally, a link between defect distributions, mesoscale dynamic failure mechanisms, and the fragmentation distributions can be established. We have shown that the influence of processing-induced inhomogeneities on the failure processes is not equivalent. A quantitative defect analysis, such as presented here, helps to separate inclusions that contribute to dynamic failure, and consequently provide physical meaning of input parameters for the models. Note that several experimental and numerical studies have shown that the distribution of inclusions has important implications for failure processes [13, 14, 28, 29]. Consequently, the location of the fracture initiation and crack patterns that evolve during fracture play an important role in fragmentation. We observed a wide range of fragment sizes in response to dynamic compression. The large fragments, such as one shown in Fig. 3a, appear to be created by cracks propagated from preferentially oriented larger carbon inclusions. In contrast the smallest fragments appear to be the consequence of crack-crack interactions arising from closely spaced inhomogeneities such as those presented in Fig. 3b. Also, nanometer-size debris was commonly present on the fracture surfaces of the fragments (Fig. 3e). The most likely source of this debris is the abrasion of the free crack surfaces rubbing against each other during the fracture process. A more detailed discussion on the microstructure- and structure-dependent fragmentation in a hot-pressed boron carbide is provided in [13].

In conclusion, we investigated the failure mechanisms in a hot-pressed boron carbide under dynamic compression using the Kolsky bar technique. A detailed microstructural characterization prior to the tests revealed a large number fraction of inhomogeneities (free carbon, aluminum nitride, boron nitride) and clusters of pores. The larger carbon inclusions are found to be preferentially oriented with respect to the hot-pressing direction. Further examination of the fracture surfaces reveals that the population of large carbon inclusions contributes significantly to micro-crack nucleation and macro-crack development in the boron carbide structure. In contrast to large carbon inclusions, there appears to be little effect of small size inhomogeneities on dynamic failure processes. We observe that the wing-crack-like mechanism is active also in the micro-scale in this advanced structural ceramic under dynamic compression.

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Figure 1(a—b). Optical microscope image of boron carbide microstructure in two representative planes. Characteristic inhomogeneities, such as free carbon (A), other non-metallic inclusions such as aluminum nitride, boron nitride (B) and pores (C) are indicated.

Figure 2: (a) Inclusion number/area fraction versus inclusion size distribution; (b) Scatter plot of identified inclusions with a correlation between the aspect ratio and sizes; (c) Orientation of inclusions in relation to the major X_3 axis, where AR is the aspect ratio.

Figure 3: (a) Fragment collected after dynamic compression test and (b) zoomed-in investigated fracture surface; Corresponding EDS spectrum of the fracture surface for: (c) boron (*grey*), and (d) overlay of carbon (*purple*), aluminum (*light blue*) and nitrogen (*light pink*); (e) Zoomed-in area showing crack initiation and propagation in the vicinity of the carbon inclusion.

Figure 4. SEM micrograph of the wing-crack-like feature from carbon inclusion.