Investigation on Impact Induced Damage in Boron Carbide with Simultaneous X-ray Imaging and Diffraction

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Abstract. The macro-scale mechanical behaviors of materials under impact can be dominated by the meso and micro-scale deformation mechanisms and cracking patterns. In combination with the X-ray imaging and diffraction techniques, impact tests are performed on boron carbide (B₄C) ceramic bulks with two different porosities by a miniature split Hopkinson pressure bar (SHPB). After a transient quasi-elastic deformation process, catastrophic failure occurs in the high-porosity bulk, with a weak failure strength. Abundant tensile and shear cracks nucleate in the low porosity bulk, but cannot connect into a cracks network due to the high adhesive force, and finally the dissociative failure occurs with several main cracks penetrating. By analyzing the diffraction peak splitting phenomenon, the extent of shear deformation is quantitatively characterized to be 60-70% of the compression deformation. The damage extent is expressed by the voids volume induced by cracks. The macroscopic stress increment of B₄C bulk under impact can be expressed by a function of the damage extent evolution.

1. Introduction
As a brittle ceramic material, boron carbide becomes an attractive material in aerospace, vessel and defense industries, with its low density and high strength [1]. The failure patterns of brittle materials under quasi-static loading have been studied widely. Under uniaxial quasi-static compression, brittle materials are generally damaged by tensile cracks [2]; but when confining pressure exists, shear cracks can dominate the failure [3]. With the increasing of loading rate, more cracks can be activated in the brittle materials [4], and the networks of cracks is easier to form, leading to catastrophic fragmentation. However, it is difficult to analyze the meso and micro-scale mechanisms of impact induced damage, due to the short time duration and the catastrophic damage evolution [5].

The variation of the structure inside material under impact can be recorded by the simultaneous X-ray imaging [6]. As a similar experimental technique, the simultaneous X-ray diffraction owns additional advantages [7, 8], such as, the diffraction profiles can rarely be affected by the overlap of inner structure, and the resolution ratio achieves the size of crystal cells. Based on the combination of simultaneous X-ray imaging and diffraction methods, Huang et al. have studied the different mesoscopic and microscopic failure mechanisms of single silicon under uniaxial impact.

In this paper, B₄C ceramic bulks with different porosities are dynamically compressed by a miniature SHPB device. By employing the simultaneous X-ray imaging and diffraction techniques, the meso and micro-scale inner structure variations of the bulks under impact are recorded. According to the X-ray information, the failure patterns, as well as the compression and shear deformation
mechanisms of B4C ceramics are investigated. An incremental model of macroscopic mechanical response for B4C ceramics under impact is developed based on the quantitatively measured damage evolution.

2. Samples and Experiments

2.1. Boron Carbide Bulks
The ceramic bulk samples used in this paper were sintered by B₄C particles with the size of ~ 2 μm. Length of the sample along the impact direction is 3 mm, area of the cross section vertical to the impact direction is 3 mm × 2 mm. Samples with two different porosities were employed in the experiments, i.e. sample A and sample B. Density and porosity of sample A are 2.35 g/cm³ and 6.7 % respectively, and those of sample B are 2.49 g/cm³ and 1.2 % respectively. The typical SEM graphics on the overall and local surfaces of sample A and B are exhibited in figure 1. As shown in figure 1(a), owing to the high porosity, voids can be seen on the surface of sample A. Dissociative particles can be observed in the enlarged image of the voids, as shown in figure 1(b), which implies that the adhesive forces between particles in sample A is relatively small. As presented in figure 1(c), due to the low porosity, particles are condensed in sample B, and no apparent void can be observed on the surface, which implies that the adhesive forces between particles in sample B is relatively strong.

![Figure 1](image_url)

**Figure 1.** Micrographs of the B₄C bulk samples. (a) Overall surface topography of sample with high porosity. (b) Local surface topography of sample with high porosity. (c) Overall surface topography of sample with low porosity. (d) Local surface topography of sample with low porosity.

2.2. Impact Experiments with Simultaneous X-ray Imaging And Diffraction
As presented in figure 2, the experimental device consists of a mini SHPB and a simultaneous X-ray imaging/diffraction system [5-7]. The resistance strain gauges are attached on the incident and transmission bars to measure the impact pulse signals. During the impact, an X-ray beamline with the area of 2 mm × 1.7 mm illuminated the middle of sample. Part of the X-ray beamline penetrated the sample and formed images on a scintillator, which captured by the imaging camera as image sequences; the other part of the X-ray beamline was diffracted by the sample, and formed diffraction patterns on a scintillator, which were enlarged and captured as diffraction sequences. Both the image and diffraction sequences are recorded every 10 μs.
3. **High-Porosity Bulk: Catastrophic Failure**

Due to the extremely high modulus of B₄C bulk, the elastic strain of sample during the impact is negligible. Therefore, the stress-time curves are adopt to represent the impact behaviors of samples in the present study. The stress-time curve for sample A with high porosity is shown in figure 3, on which the red markers denote the X-ray imaging instants 1 to 6. At the instant 1 of 4.6 μs, the stress of sample A is almost zero. But the stress sharply reaches the peak around the instant 2 of 14.6 μs. From instant 2-3, the stress rapidly decreases to zero. And after the instant 3 of 24.6 μs, the stress vibrates around zero, which means that the sample is entirely crushed and blasted into fragments (as exhibited in the inset of figure 3), totally losing the bearing capacity.

![Stress-time curve](image)

**Figure 3.** Stress-time curve of the high porosity sample under uniaxial impact loading, red markers on curve denote the X-ray imaging instants. Inset: SEM photograph of sample fragments.

This feature of catastrophic failure can also be verified from the X-ray image sequence of sample A, as shown in figure 4.
4. Low-Porosity Bulk: Dissociative Failure

4.1. Mesoscopic Analysis by X-ray Imaging
The stress-time curve of sample B with low porosity is presented in figure 5, on which the red markers denote the X-ray imaging instants, and the blue markers denote the X-ray diffraction instants. The loading process can be divided into two stages: the quasi-elastic stage before the loading instant of 10 μs, during which the stress increases almost linearly with loading time; the dynamic equilibrium stage after the loading instant of 10 μs, during which the stress keeps nearly a constant and exhibits a platform feature. At the stage of dynamic equilibrium, the increasing external load is offset with the cracks nucleation and extension inside the sample, leading to a steady-state of stress. Since that the sample with low porosity owns higher adhesion forces between particles, the stress platform for cracks propagation can reaches about 900 MPa, much higher than the peak stress of the sample with high porosity.

Figure 5. Stress-time curve of the high porosity sample under impact. On the curve, red markers denote the X-ray imaging instants, and blue markers denote the diffraction instants.
4.2. Microscopic Analysis by X-ray Diffraction

In order to investigate the deformation and failure mechanisms of sample with low porosity under impact, the simultaneous X-ray diffraction signals are analyzed. The scintillator sensor used in this paper can probe the diffraction angle \(2\theta\) from \(10^\circ\) to \(20^\circ\), and the diffraction patterns are shown in figure 6(a). It can be observed that the intensity of diffraction decreases with the impact process. For quantitative analysis, the diffraction patterns are integrated into diffraction profiles, which is concentrated at the \(2\theta\) extent from \(10^\circ\) to \(15^\circ\), as shown in figure 6(b). Peak of the initial diffraction profile \(d_1\) is located at the \(2\theta\) of about \(12^\circ\).

It can be read from figure 6(b) that the peak location and the width of the diffraction profile are almost invariant during the impact process. According to Bragg’s equation, the relation between the diffraction angle \(2\theta\) and the inter planar spacing \(d\) can be expressed as

\[
2d \sin \theta = n\lambda
\]  

(1)

Where \(\lambda\) is the wavelength of X-ray and \(n\) denotes the amount of wavelengths. Moreover, the half width \(\beta\) of diffraction profile and the crystal size \(L\) follow the Scherrer relation, which can be expressed as

\[
\beta = \frac{K\lambda}{LCos\theta}
\]  

(2)

Where \(K\) is a scale factor. It can be inferred from equations (1) and (2) that, the planar spacing and the crystal size keep nearly constant, which implies that the elastic deformation of sample B is negligible under impact.

![Figure 6](image)

Figure 6. (a) The diffraction patterns at different instants of low porosity sample under impact. (b) The diffraction profiles at different impact instants.

However, two other features can be observed according to figure 7(b): decreasing of the diffraction intensity and splitting of the diffraction peak.

When cracks activated, a large amount of interfaces appear inside the bulk, which scatters and dissipates the X-ray beamline. Since that the sample is uniform and isotropy, and the temperature rising can be ignored in the extremely short impact duration, the diffraction intensity can only be affected by the dissipation of X-ray intensity.

Besides the variation of planar spacing, the diffraction angle can also be affected by the rotation of crystal surface, as shown in figure 7. When the orientation of crystal surface is changed by \(\Delta\theta\), the diffraction angle will be changed by \(2\Delta\theta\).
Figure 7. Schematic diagram of the diffraction angle variation caused by the rotation of crystal surface.

The dilation volume caused by cracking in brittle material under impact is directly related to the damage [2, 5]. In this paper, the dilation volume $dV$ is employed as a representative of the cracking extent of sample, which is proportional to the dissipation intensity of the X-ray beamline. At the first instant of diffraction, which is also the instant for impact initiation, $dV$ is nearly zero since that the sample is intact, and the integral intensity of diffraction is called $I_0^*$; at the instant of that the diffraction integral intensity decreases to zero, $dV$ reaches the maximum, which is called $V_{dm}$. During the impact process, there exists a relation between $dV$ and the diffraction integral intensity $I^*$, which is

$$\frac{dV}{V_{dm}} = \frac{I_0^* - I^*}{I^*}$$

(3)

As shown in figure 8(a), since that $I^*$ decreases linearly during the impact, correspondingly the relative dilation volume increases linearly, which coincides with the dissociative failure pattern of brittle material [5].

Figure 8. (a) The evolution of relative dilation volume during the impact. (b) Comparison between the compression and shear strain of low porosity sample under impact.

Since that the rotation can either be clockwise or anticlockwise on the projection plane of X-ray, the diffraction angle is also varied in two contrary directions, which leads to the splitting and deviating of diffraction peak. Under the small deformation hypothesis, the shear strain is equal to the rotation angle ($\gamma = \Delta \theta$). Therefore, although $\gamma$ cannot be measured by the strain gauge directly, it can be
obtained by the deviation angle of the diffraction peak. It should be noted that the splitting and deviating of the diffraction peak is symmetrical about the original location, which is corresponding with the symmetry of shear strain tensor. Figure 8(b) presents the comparison between the compression and shear strain of low porosity sample under impact. It can be seen that the shear strain amplitudes are 60-70% of the compression strain amplitudes, during the whole impact process, which implies that there exists significant shear cracking.

5. Influences of Cracks Evolution on Macroscopic Mechanical Behaviors
The macroscopic mechanical behaviors of materials are depended on the mesoscopic and microscopic cracks to a great extent [4]. For brittle materials under impact, the propagation of cracks can continuously influence the bearing capacity. In this paper, the incremental stress of B_{4}C bulks is expressed as a function of the cracking extent $\eta$, which is

$$\Delta \sigma = E \dot{\varepsilon} (1 - \eta) \Delta t - \sigma_f \dot{\eta} \Delta t$$

Where $\dot{\varepsilon}$ is the strain rate of the non-damage part in sample, thus $E \dot{\varepsilon} (1 - \eta) \Delta t$ denotes the stress increment of non-damage part in sample at time interval $\Delta t$; $\sigma_f$ is the theoretical strength of B_{4}C, which is 4 GPa measured by quasi-static compression tests, therefore $-\sigma_f \dot{\eta} \Delta t$ denotes the stress drop caused by cracks propagation at time interval $\Delta t$. At the initial of impact, the damage extent $\eta$ is approximately zero; then $\eta$ increases linearly according to figure 8(a), with a constant rate $\dot{\eta}$; when $\eta$ equal to 1, the sample is entirely failure, and the stress becomes zero.

![Figure 9. Comparison of the compression stress-time curves by theoretical computation and experimental measurement, (a) high porosity sample, (b) low porosity sample.](image)

The compression stress-time curves of samples with high porosity and low porosity are presented in figure 9(a) and (b) respectively, where the black solid lines denote the experimental curves, and the red dotted lines denote the theoretical curves computed by equation (4). The curves of theoretical computation exhibit high consistency with those of experimental measurement.

6. Conclusions
In order to investigate the heterogeneity in deformation of B_{4}C ceramics under impact, samples with high and low porosities were compressed by a miniature SHPB. In combination with a simultaneous X-ray imaging and diffraction system, the failure patterns, as well as the mesoscopic and microscopic deformation mechanisms of B_{4}C ceramics are analyzed. The conclusions are listed as the following:
(1) Owing to the weak adhesion forces between particles, B$_4$C bulk with high porosity fails catastrophically at a low stress level, after short elastic deformation and cracks nucleation processes. The deformation heterogeneity exhibits a linear increasing tendency before failure, and an exponential growth tendency after failure.

(2) The load process of B$_4$C bulk with low porosity can be divided into two stages: the elastic deformation stage; the nucleation and propagation of various tensile and shear cracks. Since that the interaction of cracks is prevented by the strong adhesion forces between particles, there is no cracks network formed in the bulk. The final failure pattern exhibits a dissociative feature, with several main cracks breaking through the bulk. The deformation heterogeneity exhibits a linear increasing tendency during the whole impact process.

(3) The diffraction peak of the low-porosity sample splits into two separate peaks symmetrically. By measuring the deviated angle of the two peaks, the evolution of shear strain is quantitatively characterized, which is 60%-70% of the compression strain during the impact process. Network of shear cracks is one of the dominated failure patterns.

(4) The incremental relationship between the macro-scale stress and the meso & micro-scale damage has been built up. The theoretical computed stress-time curves of high & low porosity B$_4$C bulks is consistent with those of experimental acquired.

7. References
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