Fabrication and dynamic compressive response of reaction sintering H-BN ceramic

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Abstract. Hexagonal boron nitride ceramic (h-BN) based on the nitridation of B powders was obtained by reaction sintering method. The dynamic compressive response was investigated using a Split Hopkinson pressure bar tester at high strain rates in the range of $8.8 \times 10^1$–$1.6 \times 10^3$ s⁻¹. By increasing the strain rate, the dynamic strength and the failure strain increased continuously. The compressive stress–strain curves exhibited universal three deformation regions, including inelastic deformation region, rapid loading region and failure region. The failure mode of the h-BN ceramic depended upon the strain rate.

1. Introduction

The hexagonal boron nitride (h-BN) presents unique chemical and physical properties, including low relative permittivity, low dielectric loss, low density, high melting point, high thermal conductivity, excellent manufacturability, and excellent corrosion and oxidation resistance [1-5]. Because of these advantages, h-BN has been widely applied in these fields such as aerospace, metallurgy, refractory, lubricants, and chemical engineering [6-9]. Among them, one of the most important applications is as a wave-transparent material in the aerospace field, in which it would work in various serious conditions, such as vibration, cyclical load, thermal shock, oxidation, corrosion, wear, and impact [10]. Therefore, it is of particular importance to evaluate the mechanical properties of h-BN ceramic, especially under dynamic loadings.

In recent years, enormous investigations were conducted on h-BN ceramic. However, most of the investigations were regarding to the preparation process and static mechanical properties [11-14]. The effects of dynamic loading have not received significant attention yet, even though it is the common condition in many structural applications [7]. To evaluate the dynamic mechanical properties and give a more comprehensive understanding of h-BN ceramic, this study will focus on the dynamic behavior and failure mechanism under uniaxial compression. This work will provide a reference for the dynamic mechanical testing and future research on h-BN ceramic material.
2. Experimental procedures

2.1. Fabrication
The starting BN (Shanghai Yuyi, China) and B (mean particle size < 3 μm, purity >95%, Dandong Chemical Industry Institute, China) powders, together with a certain amount of sintering additions, were mixed with ethanol using agate balls for 24 h in a plastic bottle. The mixture was then dried, crushed, and screened through a 200-mesh sieve. The powder was compacted by die-pressing (SL-45, Shanghai Mechanics, China) under three different pressures. Sintering was conducted in a graphite-resistant furnace at various temperatures for 2 h under 0.2 MPa N2 pressure.

2.2. Characterization
The crystal structures were determined by X-ray diffraction (XRD, CuKα). The flexural strength of the BN ceramic was evaluated by three-point bending test at a constant crosshead speed of 0.5 mm/min. The dimension of the specimens was 3 mm × 4 mm × 40 mm (height × width × length, respectively) with the span of 30 mm. The surfaces of specimens were polished to 0.5 mm before conducting the three-point bending test.

The dynamic mechanical responses of the BN ceramic were measured in compression using a Split Hopkinson pressure bar (SHPB) at room temperature. The wave speed and Young's modulus of the incident and transmitted bars are 4840m/s and 210 GPa, respectively. The dimensions of the specimen were φ8×8 mm. The dynamic tests were carried out at various strain rates ranging from 0.8×10³ s⁻¹ to 1.6×10³ s⁻¹, and the loading direction is parallel to the thickness direction. In the case of compression experiments, the engineering strain (ε) and stress (σ) in the specimen are calculated according to the one-dimensional elastic stress wave theory [15].

The microstructures of cross surfaces of the as-sintered ceramics were observed using a Scanning Electron Microscopy (SEM, S-4800, Japan).

3. Result and Dissecussion

3.1. Quasi-static properties
Figure 1 shows the X-ray diffraction of the h-BN ceramic. As shown in figure 1, only the peaks marked with h-BN are observed, indicating full chemical reaction between B powder and N2 gas. The reaction formulas are given as follows:

\[ 2B + N_2 \rightarrow 2 \text{BN} \]  \hspace{1cm} (1)

\[ 2B + N_2(g) + x \text{h-BN} \rightarrow (2 + x) \text{BN} \]  \hspace{1cm} (2)

![Figure 1. XRD patterns of h-BN ceramic.](image1)

![Figure 2. SEM of h-BN ceramic.](image2)

SEM images of the as-prepared samples after reactive sintering are given in figure 2. As shown in figure 2, card-house-shaped particles with diameter of about 1 μm were obtained. The fine particles obtained may cause fine-grain strengthening to improve nitride strength. The increase of fine particles may enhance product compactness, which can also lead to increases in strength.
Figure 3 shows the stress-displacement curves under a quasi-static load of the as-prepared samples after reactive sintering. The stress-displacement curve is close to the trend line, with failure strength of 180.86MPa and displacement of 0.047mm. The as-prepared samples present mainly brittle deformation behavior under quasi-static compression, without obvious yield phenomenon.

Under a quasi-static load, the ceramic will occur longitudinal splitting or shear fracture and crush into two or three pieces. Fracture cones formed by shear fracture and sheets formed by longitudinal splitting are observed in h-BN ceramics, as presented in figure 4(a). Figure 4(b) further gives the microstructure of fracture section, in which ceramic mostly emerged lamellar or step cleavage fracture, resulting from the growth of cracks inside the material. The passivation of crack tip in ceramics, interface crack or crack deflection occurs in the expansion process, leading to enhance of energy consumption and increasing the toughness.

3.2. Dynamic behavior
Figure 5 gives the time dependence of original signals of the incident, reflected and transmitted waves for the as-prepared h-BN ceramics, measured under a strain rate of 1000/s. When the impact bar impact one end of the incident bar, a compressive stress pulse will be produced and propagate along the incident bar to the other end contacting with the specimen. Part of the incident wave will be reflected on the interface between impact bar and as-prepared h-BN specimen, while the other part will transmit to the transmitted bar at the same time. Several reflections and transmissions will occur in a very short time until the stress wave is attenuated to a very low level even zero, resulting in the time delay ($\Delta t$) of the reflected and transmitted strain pulses.
Figure 5. The original voltage signals.

From the recorded signals shown in figure 5, the stress-strain relationship was converted. Figure 6(a) presents the stress-strain curves at strain rates ranging from 1000 to 1600/s. An inverted “V” type true stress-true strain curve is observed at various strain rates, and the largest stress increases with increasing strain rate. Under dynamic compression, elastic deformation without obvious yield phenomenon will occur first at low stress; when the stress reached its limitation, rapid decay will be observed. h-BN ceramics present the inelastic deformation before the damage due to the micro-cracks caused by impact load. The reactive sintering h-BN ceramics exhibits similar dynamic compressive behavior to those observed in other brittle materials [16,17], indicating the h-BN ceramics is a brittle material.

Figure 6. Dynamic compressive characteristics of the h-BN ceramics with various strain rates (a) Stress–strain curves. (b) Effect of strain rate on strength and failure strain.

The relationships between strain rate and strength/failure strain are shown in figure 6(b). As can be seen, both dynamic strength and strain of h-BN ceramics increase with increasing strain rate. The fracture strength and strain increase from 163.8 MPa to 231.8 MPa and 0.0047% to 0.0084%, respectively, when the strain rate increases from 800 m/s to 1600 m/s, indicating both of them is very sensitive to strain rate. This phenomenon may attribute to their failure modes during the compressive process. The different compressive strengths may result from the different fracture modes at various strain rates.

3.3. Fracture mechasim
To obtain a comprehensive idea of the failure mode of the h-BN ceramic, the impact fracture mode at various strain rates were investigate. Figure 7 gives the macroscopic images of impact fracture fragments at various strain rates. The ceramic exhibits different failure modes at various strain rates.
Damage of the ceramic specimens is deepened as strain rate increasing, the dimension of the crushed blocks decreases significantly. At low strain rate (800 s\(^{-1}\)), specimens are crushed into several large blocks with fracture surface along the longitudinal cleavage planes and shear fracture surface inclining to the loading axis. At higher strain rate (1200 s\(^{-1}\)), more flakes is obtained, the specimens are crushed in to pieces. When the strain rate increases to 1600s\(^{-1}\), finest pieces can is obtained. Generally, the damage of the ceramic is due to the generation and development of cracks. At low strain rate, sufficient time is provided for the cracks to complete the nucleating and expanding process, resulting in lower stress and large blocks. Once a high strain rate is applied, the cracks didn’t have sufficient time to absorb more energy, resulting higher stress and more cracks and deepen damage.

Figure 7. Macroscopic images of impact fracture fragments at various strain rates
(a) \(\dot{\varepsilon}=800/s\); (b) \(\dot{\varepsilon}=1200/s\); (c) \(\dot{\varepsilon}=1600/s\).

To further investigate the fracture mechanism at various loading speed, SEM photograph of the h-BN sample chips tested by the split Hopkinson pressure bar were shown in figure 8. At a low loading speed, the specimen exhibits mainly intergranular fracture with small amount of transgranular fracture. While at high loading speed, obvious transgranular fracture can be observed with small part of intergranular fracture. The initiation and growth of cracks is easier along the defect in materials. When the loading rate is relative low, there is enough time for the cracks spreading along the region with lower strength. Therefore, this phenomenon contributes to mainly intergranular fracture. At high loading rate, because of the shorter time available for relaxing the stress concentration, the cracks hardly propagates and deflects, which leads to a transgranular fracture. Besides, the higher energy at high loading rate was satisfied with the dynamic condition of transgranular fracture, resulting in transgranular fracture.

Figure 8. SEM micrographs of impact fracture fragments at various strain rates
(a) \(\dot{\varepsilon}=1000/s\); (b) \(\dot{\varepsilon}=1400/s\).

4. Conclusions
The h-BN ceramics materials have been fabricated by reaction sintering, and its dynamic compressive
behaviors at various strain rates were investigated systematically. The conclusions are as follows:

(1) Stress-displacement curve of h-BN ceramic exhibits an approximate linear relationship under a quasi-static loading.

(2) The static compression stress-strain curves of h-BN ceramic materials show an inverted “V” type true stress-true strain curve at various strain rates, and the largest stress increases with increasing strain rate.

(3) The macroscopic impact of fracture fragments show different sizes closely related to the loading speed. SEM micrographs of impact fracture fragments also indicate that the h-BN exhibits mainly intergranular fracture with small amount of transgranular fracture at low loading speed, obvious transgranular fracture can be observed with small part of intergranular fracture when the loading speed is very high.

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