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# Structure and mechanical properties of boron-rich boron carbides

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ABSTRACT

A series of boron-rich boron carbides are investigated to explore the effect of boron/carbon ratios on the microstructure and mechanical properties of the complex material. It has been found that excess boron substitution gives rise to expanded lattice constants, orientational asymmetry of the chain structure and distortion of the icosahedra of rhombohedra $B_4C$ units in comparison with conventional carbon-rich boron carbides. Microstructure characterization reveals a high density of stacking faults and growth twins in boron-rich boron carbides. Nanoindentation measurements demonstrate that the hardness and modulus of boron-rich boron carbides decrease with the increase of boron content while the $B_{10.2}C$ sample with the highest boron substitution shows relatively high hardness and modulus. This study suggests that the structure of single-phase covalent materials could be tailored by self-alloying for improved mechanical properties.

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## 1. Introduction

Boron carbide is a covalent ceramic material with nominal stoichiometry $B_4C$. The crystal structure of boron carbide is very complex, consisting of 12-atom icosahedra with a 3-atom chain in a rhombohedral unit. It has been widely used as high performance ceramic materials where hardness and weight are critical because boron carbide has a low density of about 2.5 g/cm$^3$, ultra-high hardness, good thermal stability and low material costs. [1–5] Similar to other ceramic and metallic materials, the mechanical properties of $B_4C$ strongly depend on chemical compositions, microstructure and fabrication processes. [2,3,6–12] Particularly, boron carbide has a wide composition range from 8.8 to 20 at.% C to form thermodynamically stable solid solutions as shown in the equilibrium phase diagram (Fig. 1). [2,3,8,13] Beyond ~20 at.% C, a mixture of boron carbide and graphite is often observed. Below ~8 at.% C, pure boron co-exists with boron carbide. [9,14] Several theoretical and experimental studies have suggested that the atomic bonding, electron density, mechanical properties and lattice constants of boron carbide change with boron/carbon ratios. [15–20] However, a systematic experimental investigation of the influence of boron/carbon ratios, particularly in the boron-rich side, on microstructure and mechanical properties of boron carbide solid solutions has not been reported. In this study, a series of boron-rich boron carbides, prepared by hot-pressing, are investigated to reveal the chemical effects on structure and properties of the complex covalent material, which will provide an experimental benchmark for designing boron carbide materials with optimal properties.

## 2. Experimental procedure

The boron carbide samples used in this study were fabricated and provided by 3 M$^{TM}$ Advanced Materials Division. Fine and pure grade of regular $B_4C$ (ESK Tetrabor; average particle size: 10 μm, purity >99%) and amorphous boron (H.C. Starck; 95% purity; average particle size: 0.8 μm) powders were mixed by ball milling to prepare boron-rich $B_4C$ with different compositions. A series of boron-rich $B_4C$ containing excess contents of boron (10, 33, 43 and 55 wt.% boron) were prepared by hot-pressing at 1900–2200 °C under a pressure of 13.8 MPa with a BN coated graphite die. $B_4C$ samples with 10, 33, 43 and 55 wt.% excess boron correspond to the carbon concentrations of 17.8, 13.2, 11.3 and 8.9 at.% C and the B/C ratios of 4.6 ($B_{4.6}C$), 6.5 ($B_{6.5}C$), 7.8 ($B_{7.8}C$), 10.2 ($B_{10.2}C$), respectively. The dimensions of the received samples are around 3 × 3 × 3 mm. For comparison, a commercial hot-pressed $B_4C$ sample is used as the reference [10]. As shown in the boron-carbon phase diagram (Fig. 1) [3,8,13], these samples all fall into the composition range of the single-phase boron carbide solid solutions and, thus, a pure boron phase is not expected. Since the regular $B_4C$ precursor powders contain a small amount of free carbon, the actual composition of boron-rich $B_4C$ after hot-pressing is expected to have a slightly lower B/C ratio. The degree of shift is hard to be

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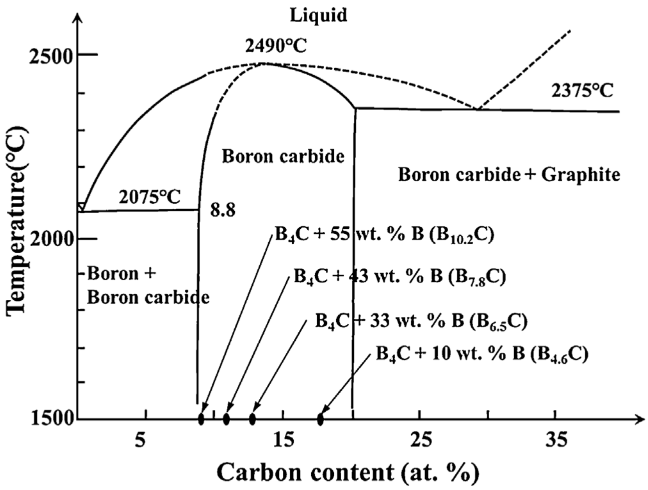


**Fig. 1.** The phase diagram of boron-carbon system. The samples with different B/C ratios used in this study are marked in X-axis.

estimated owing to the difficulty in measuring the content of free carbon in raw green powders.

The sintered boron carbide samples were carefully polished by diamond papers to obtain mirror-like surfaces for SEM and Raman characterizations and for nanoindentation measurements. The microstructures of boron-rich boron carbides were investigated by SEM (JEOL, JIB-4600F) and electron backscattered diffraction (EBSD) analyses. XRD patterns were collected with 2θ angles ranging from 15 to 85° by a Rigaku X-ray diffractometer (RINT 2200) with an X-ray Cu tube ($CuK_{\alpha}$, λ = 1.5418A°). Phase constitutions were identified using the JCPDS database. Raman spectra were acquired using a Renishaw micro-Raman spectrometer equipped with a charge coupled device (CCD) detector and an optical microscope. A 514.5 nm Ar ion laser was used as the excitation source. Raman spectra have been collected at a lower laser power in order to avoid sample damage and local heating effects. TEM specimens were first sliced from bulk boron-rich $B_4C$ samples, ground and polished to a thickness of 80–100 μm, and then dimpled to a thickness of approximately 15–25 μm for ion-beam milling to obtain

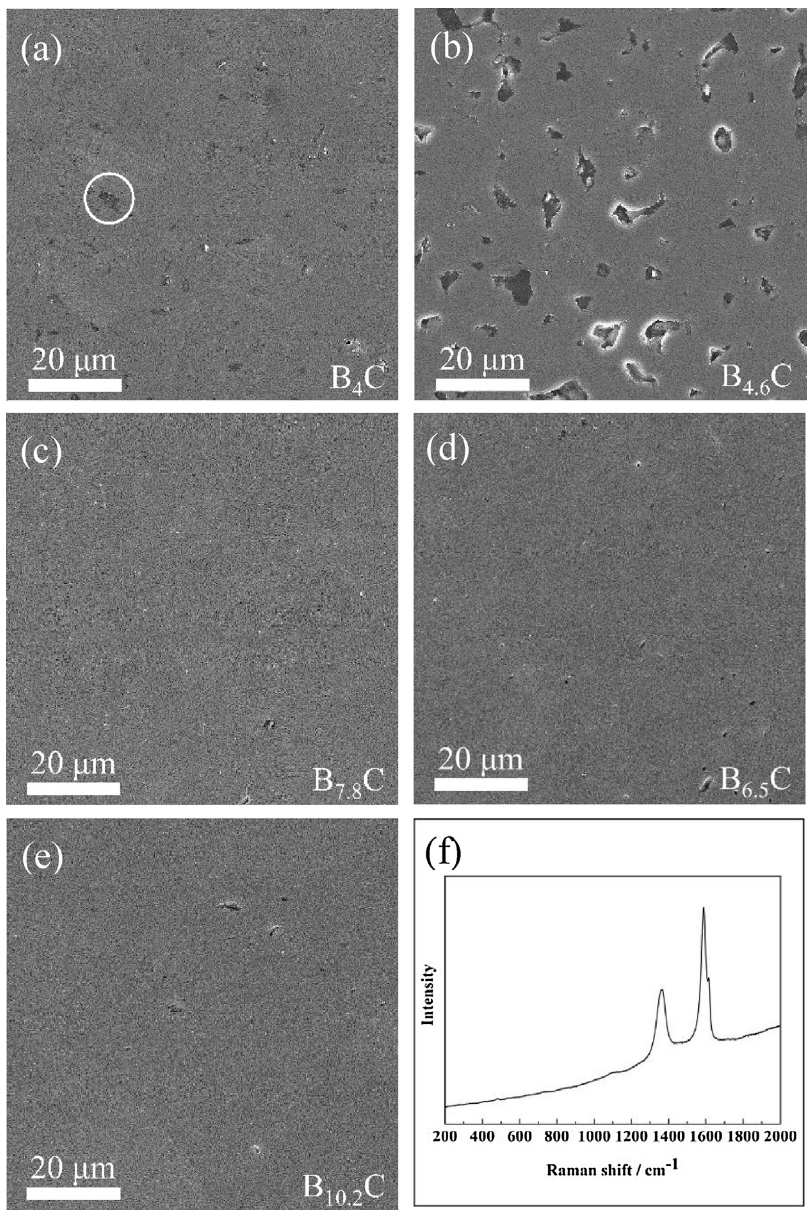


**Fig. 2.** SEM images of regular $B_4C$ and boron-rich boron carbides with different boron contents. (a) regular $B_4C$, (b) $B_{4.6}C$, (c) $B_{6.5}C$, (d) $B_{7.8}C$, (e) $B_{10.2}C$, (f) Raman spectra of black particles marked with white circle in (a), revealing the existence of free carbon in regular $B_4C$.

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