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# High toughness and electrical discharge machinable B<sub>4</sub>C-TiB<sub>2</sub>-SiC composites fabricated at low sintering temperature



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#### ABSTRACT

Dense and electrical discharge machinable  $B_4C$ -TiB<sub>2</sub>-SiC triple-phase ceramic composites with high fracture toughness and high hardness were successfully fabricated via reactive hot-pressing sintering using  $B_4C$  and nano-layered Ti<sub>3</sub>SiC<sub>2</sub> with different ratios as raw materials at a relatively low temperature of 1850 °C. The results showed that the mechanical properties of  $B_4C$ -TiB<sub>2</sub>-SiC composite were remarkably enhanced than that of monolithic  $B_4C$  ceramic. The micro-hardness, flexural strength, fracture toughness and compressive strength of  $B_4C$ -TiB<sub>2</sub>-SiC composite prepared with  $B_4C$ -30 vol% Ti<sub>3</sub>SiC<sub>2</sub> starting powder were 31.6 GPa, 492.3 MPa, 8.0 MPa·m<sup>1/2</sup> and 2727 MPa, respectively. High hardness was attributed to the low open-porosity and grain refinement, while the high toughness was mainly ascribed to the crack deflection due to the dispersed TiB<sub>2</sub> and SiC particles in the  $B_4C$  matrix. Moreover, the composites exhibited significantly improved machinability than monolithic  $B_4C$ , as evidenced by electrical discharge machinag.

#### 1. Introduction

Boron carbide (B<sub>4</sub>C) is an extremely attractive engineering material with extremely high hardness, excellent chemical stability, good abrasion resistance and satisfactory radiation resistance [1-3]. Therefore, B<sub>4</sub>C ceramics have great potential applications in military, aerospace and industrial as light-weight ballistic armors, body armors, blasting nozzles, cutting tools as well as wear resistant components. Additionally, owing to the neutron absorption capacity of isotope  ${}^{10}B$ ,  $B_4C$ ceramics are widely applied in the nuclear industry as controlling rods, neutron detectors and shielding materials [4-6]. Nevertheless, B<sub>4</sub>C exhibits several shortcomings such as poor sinterability and relatively low fracture toughness [7]. In addition, considering the high hardness and low fracture toughness of B<sub>4</sub>C, the machining of B<sub>4</sub>C ceramics is costly and time consuming. These disadvantages seriously restrict the wide applications of B<sub>4</sub>C-based ceramics. Therefore, it is of great importance to lower the sintering temperature of B<sub>4</sub>C ceramics and to improve their mechanical properties and machinability.

Many efforts have been made to improve strength and toughness of monolithic  $B_4C$  ceramics, and addition of silicon carbide (SiC) and titanium diboride (TiB<sub>2</sub>) as sintering additive were found to be effectively for it [8–12]. Both SiC and TiB<sub>2</sub> materials possess many excellent chemical and physical properties, such as high hardness, good chemical stability and good oxidation resistance [13–15]. Therefore, the addition

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of SiC or TiB<sub>2</sub> could maximize the retention of unique properties of B<sub>4</sub>C. The B<sub>4</sub>C-TiB<sub>2</sub> and B<sub>4</sub>C-SiC composites usually fabricated by reactive hot pressing with different strating materials [16,17]. For example, Skorokhod et al. [18] prepared a B<sub>4</sub>C-15 vol% TiB<sub>2</sub> composite with a fracture toughness of 6.1 MPa·m<sup>1/2</sup> by in situ reactions of B<sub>4</sub>C, TiO<sub>2</sub> and C using hot pressing at 2000 °C. Zhang et al. [19] fabricated B<sub>4</sub>C-SiC composites from B<sub>4</sub>C, Si, and C powders (sintering temperature: 1700-1900 °C) and the fracture toughness of 6.0 MPa·m<sup>1/2</sup> was obtained with 1900 °C. In order to further improve the fracture toughness of B<sub>4</sub>C ceramics, Zhang et al. [20] prepared B<sub>4</sub>C-20 wt% (TiB<sub>2</sub>-SiC) triple-phase composites by in-situ reactions of B<sub>4</sub>C, TiC and Si using hot pressing at 1950 °C and the fracture toughness of which reached 6.38 MPa m<sup>1/2</sup>. However, TiB<sub>2</sub>-SiC agglomerates are randomly dispersed in the B<sub>4</sub>C matrix, and hence the controlling of second phase distribution in matrix is important for improving the overall mechanical performance of composites. Up to date, most of B<sub>4</sub>C composites were sintered at high temperatures and have a microstructure of inhomogeneous and coarse grain size. In order to obtain high-performance B<sub>4</sub>C-base composites at relatively lower sintering temperature, nano-layered Ti<sub>3</sub>AlC<sub>2</sub> was chosen as a novel sintering aid in our previous study and composites with significantly improved mechanical properties were obtained at a relatively low sintering temperature [5,8]. However, the existence of Al-rich phase had a detrimental influence on the hardness and strength of the composites.

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#### Table 1

Density of B<sub>4</sub>C-base composites sintered with different amount of Ti<sub>3</sub>SiC<sub>2</sub>.



Fig. 1. XRD profiles of B4C-base composites sintered with different amount of Ti3SiC2.

In order to obtain uniformly dispersed  $TiB_2$  and SiC phases in  $B_4C$  matrix, in this study, we explored the feasibility of preparation of  $B_4C$ - $TiB_2$ -SiC composites via reactive hot pressing using  $B_4C$  and nano-layered  $Ti_3SiC_2$  powders as raw materials. The microstructure, mechanical properties and machinability of sintered  $B_4C$ - $TiB_2$ -SiC composites were analyzed.

#### 2. Experimental

B<sub>4</sub>C-TiB<sub>2</sub>-SiC composites were prepared from B<sub>4</sub>C powders (Grade HS, H.C. Starck GmbH, Germany, purity ≥ 96.7%, ~0.5 µm) and Ti<sub>3</sub>SiC<sub>2</sub> powders (Forsman, China, purity ≥ 98.0%, 0.5–10 µm), which were mixed according to the formula of B<sub>4</sub>C-x vol% Ti<sub>3</sub>SiC<sub>2</sub> with x = 0, 20, 25 and 30. The powders were ball-milled for 8 h using ethanol as a ball-milling media. After ball-milling, the slurries were dried and sieved. Then, the mixed powders were put into a graphite die with an inner diameter of 50 mm and sintered in vacuum atmosphere using a hot-pressing furnace at 1850 °C with a heating rate of 10 °C/min and a dwell time of 30 min. A unidirectional pressure of 30 MPa was applied during sintering.

The bulk density and open-porosity of the sintered samples was measured according to the Archimedes's method. Microstructural investigations of the specimens were studied by scanning electron microscopy (SEM) with an energy dispersive spectrometer (EDS) system. The phases of specimens were characterized by X-ray diffraction (XRD). Vickers hardness (H<sub>V</sub>) was measured by a Vickers indenter with a load was 9.8 N for 15 s dwell. Specimens with a dimension of 2 mm imes 2 mm  $\times$  4 mm were prepared for compression test at room temperature. The strain rate was 0.5 mm/min. The rectangular bars for the flexural strength measurement with a dimension of 3 mm imes 4 mm imes 35 mm was prepared by electrical discharge machining (EDM). The fracture toughness was determined using the single-edge notched samples with the rectangular bars of 2 mm  $\times$  4 mm  $\times$  35 mm with a 2 mm deep and 0.2 mm wide notch. The span of 30 mm was applied to the three-point bending test. The crosshead speed was 0.05 mm/min. The machinability of specimens was evaluated by EDM machine (NOVICK, AR1300, China).



Fig. 2. Polished surfaces of B<sub>4</sub>C-base composites sintered with different amount of Ti<sub>3</sub>SiC<sub>2</sub>. (a) 0 vol%, (b) 20 vol%, (c) 25 vol% and (d) 30 vol%.

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