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The preparation and properties of SiC_w/B_4C composites infiltrated with molten silicon

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Abstract

Silicon carbide whisker (SiC_w) toughened B_4C composites have been prepared by pressureless infiltration of B_4C–SiC_w–C preforms with molten silicon under vacuum at 1500 °C. The effect of SiC_w addition on bulk density, hardness, bending strength, fracture toughness and microstructure of SiC_w/B_4C composites is discussed. It is revealed that the addition of SiC_w improves the fracture toughness of B_4C ceramic, but reduces its bending strength at the same time. The maximum fracture toughness for SiC_w/B_4C composite with 24 wt% SiC_w addition is 4.88 MPa $m^{1/2}$, which is about 9% higher than that of the one without SiC_w, but at the same time, the bending strength reduces to the minimum value 243 MPa, reduced by 25%. XRD analysis shows that the phase composition of reaction bonded SiC_w/B_4C composites is B_4C, SiC, Si, and B_{12} (C, Si, B)$_3$, with no residual C. And the main toughening mechanism of SiC_w is whisker pulling up.

Keywords: B. Whiskers; C. Toughness and toughening; B_4C; Reaction bonding

1. Introduction

The outstanding properties of B_4C, such as low specific weight, high hardness, good wear resistance, good mechanical properties, high melting point, adequate resistance to chemical agents and high neutron absorption cross-section, make it a valuable potential material for a variety of applications [1–5]. However, the realization of this potential is hindered by two prime issues: one is the very high temperature required for its sintering and the other is the low fracture toughness [4,6–9]. The high sintering temperature not only leads to rapid grain coarsening, but also generates equipment-related problems [10,11]. Its low fracture toughness has seriously hampered its practical application. Thus, the work of reducing the high sintering temperature and improving its fracture toughness is of great significance.

The "reaction bonding" process has been proved to be a useful approach to fabricate fully dense, boron carbide-based composites at a low temperature (1450–1600 °C) [12–14]. Using this method, reaction-bonded boron carbide (RBBC) can be prepared by pressureless infiltration of preforms, made of boron carbide with or without the addition of free carbon, with molten silicon in a graphite furnace under vacuum [15–18].

The methods of toughening boron carbide ceramic include particle toughening, whisker toughening, fiber toughening and so on [19–21]. Among all the methods mentioned above, many works focus on the whisker toughening, resulting from its good toughening effect and the simple preparation process. In the present paper, SiC whisker is considered to be an ideal toughening phase due to its high strength, high elastic modulus, good chemical stability, good resistance to high temperature and other characteristics [22–24]. In this work, SiC_w was introduced into B_4C ceramic to improve its fracture toughness, and dense SiC_w/B_4C composites with good fracture toughness were prepared by reaction bonding at 1500 °C.

2. Experimental

Silicon carbide whiskers (Guangzhou Jiechuang Trade Co., Ltd., China), boron carbide powders (two kinds: F100 and F500, Mudanjiang Jingangzuan Boron Carbide Co., Ltd., China) and carbon black powders were used as major raw

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Table 1
The properties of B_4C powders.

Type	Purity (%)	B_{total} (%)	C_{total} (%)	$d50$ (μm)
F100	96–98	77–80	17–21	125
F500	94–95	74–78	17–21	12.8 ± 1.0

materials. The properties of B_4C powders are shown in Table 1, and the morphologies of raw materials are illustrated in Fig. 1.

Firstly, the original boron carbide powders were soaked with HCl to remove B_2O_3 and the silicon carbide whiskers were soaked with HF to remove SiO_2 in the raw material. Then the boron carbide powders and the silicon carbide whiskers were washed with distilled water to neutral condition. After that, they were dried in a vacuum oven. Secondly, the boron carbide powders and carbon black powders combined with paraffin wax solution (20 wt%) and dispersant (tetramethyl ammonium hydroxide) were ball-milled in distilled water for 24 h in an ND8-4L planetary mill. Then, the SiC whiskers, which were pre-dispersed in distilled water for 45 min by ultrasonication with tetramethyl ammonium hydroxide (TMAH, 25 wt%, Jiangsu, China) as dispersant, were mixed with the milled powders for 6 h. After milling, the slurries were dried at 60 °C in a vacuum oven to obtain the mixed powders. Then, the mixed powders were sieved through 60 meshes and were pressed into green compacts with dimensions 50 mm × 25 mm × 6 mm at 100 MPa for 30 s. After heat treatment at 900 °C for 5 h, the green compacts were transformed into the B_4C–SiC_w–C preforms. Finally, the preforms were reaction sintered by liquid Si infiltration (LSI) in a resistance heated graphite furnace at 1500 °C for 30 min in vacuum. The detailed processing procedure is shown in Fig. 2.

Bulk density of SiC_w/B_4C composites and porosity of B_4C–SiC_w–C preforms were measured by the Archimedes method. Crystal structure of the SiC_w/B_4C composites was identified by X-ray diffraction (X′Pert PRO, Holland PANalytical Company). Hardness of the SiC_w/B_4C composites was determined by a microhardness tester (HXD-1000TMSC/LCD, China). Microstructure of the SiC_w/B_4C composites was observed by scanning electron microscopy (SEM, S-3400N, Japan Hitachi Company). The tested bars for the flexural strength test had dimensions 3 mm × 4 mm × 36 mm and the flexural strength test was carried out with a crosshead speed of 0.5 mm/min and a span of 30 mm. The single edge notched beam (SENB) test was adopted to measure fracture toughness. The dimensions of the tested bars for the SENB test were 2 mm × 4 mm × 36 mm with a notch of 2 mm in depth and 0.2 mm in width. And the SENB tests were carried out with a crosshead speed of 0.05 mm/min and a span of 20 mm.

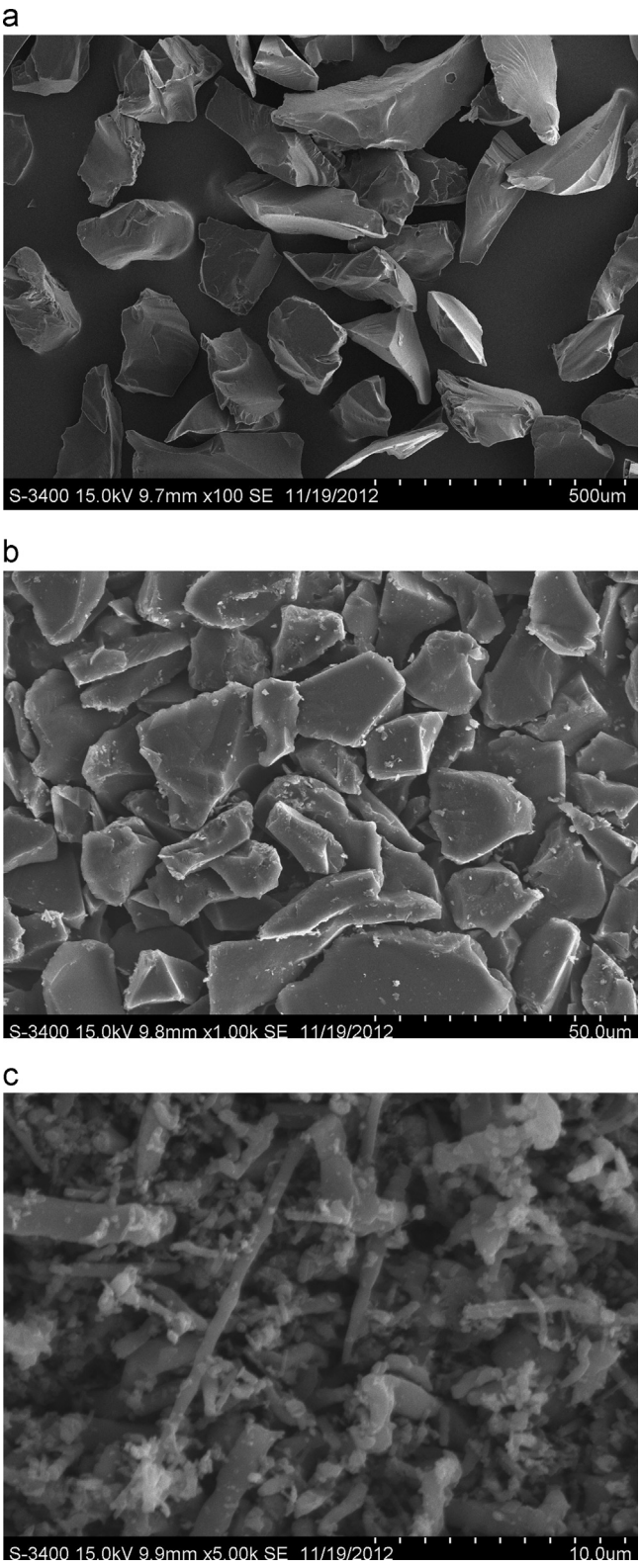

Fig. 1. SEM micrographs of raw materials. (a) F100; (b) F500 (c) SiC_w.

3. Results and discussion

3.1. Bulk density results

Variation in SiC_w addition with bulk density of SiC_w/B_4C composites is shown in Fig. 3. And variation in SiC_w addition with relative density and porosity of B_4C–SiC_w–C preforms is shown in Fig. 4. With the increase of SiC_w addition, the bulk density increases firstly and then decreases slowly (see Fig. 3). The increase of SiC_w addition reduces the stacking density of the mixed powders, and thus leads to high porosity and low