



Short communication

Simultaneously enhanced toughness and strain tolerance of SiC-based ceramic composite by in-situ formation of VB₂ particles

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ABSTRACT

SiC-30vol%VB₂ ceramic composite was pressureless densified at 2150 °C with excess B₄C and C as sintering aids after in-situ formation of VB₂ in SiC matrix. The sintered bulk gained a considerably high fracture toughness of $7.0 \pm 0.4 \text{ MPa m}^{1/2}$, which was ~ 2.4 times as high as that of the monolithic SiC ceramic, owing to the existences of weak heterophase boundaries, thermal residual stresses and microcracks. Meanwhile, since the VB₂ particle has a lower elastic modulus than SiC and significantly suppressed the grain growth of SiC, the composite exhibited a high flexural strength of $458 \pm 36 \text{ MPa}$ and a relatively low Young's modulus of $356 \pm 6 \text{ GPa}$, resulting in an increase of $\sim 59.3\%$ in mechanical strain tolerance (1.29×10^{-3}) compared with that of single-phase SiC ceramic. Besides, the residual stresses and microcracks also induced a lower-than-expected Vickers hardness of $20.8 \pm 0.5 \text{ GPa}$ in the composite.

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1. Introduction

Silicon carbide ceramics are widely used structural materials due to their good high-temperature strength, and superior wear and oxidation resistances [1–4]. However, the insufficient toughness still remains to limit their broader applications under stress. Particulate toughening is an available method to overcome the brittleness. Specifically, previous studies have shown that the introduction of TiB₂ or TiC particles in SiC matrix is effective for the improvement of fracture toughness [5–11]. Because these toughening inclusions possess higher thermal expansion coefficient (CTE) and elastic modulus than SiC, residual stresses are created by thermal expansion mismatch and contribute to the enhanced fracture toughness of ceramic composites [7–9]. However, these highly toughened composites also gained elasticity [11,12], which would deteriorate the mechanical strain tolerance (flexural strength/elastic modulus). In many applications, ceramic

materials are assembled with other metallic components to form a hybrid structural module. The failure of these ceramics usually occurred at the junction regions due to the mismatch of CTE and elastic strains between different materials. If the elastic modulus of SiC ceramic can be lowered to be compatible with metallic materials while retaining high strength, the fracture can be effectively avoided [13,14]. Thus high strain-to-failure is crucial to improve the reliability of SiC ceramic in hybrid systems. In order to broaden the range of applications, it is necessary to develop new SiC-based materials with simultaneously enhanced fracture toughness and mechanical strain tolerance.

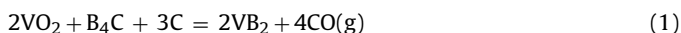
VB₂ belongs to the transition-metal diborides and possesses many attractive properties, such as high melting point, extraordinary high thermal expansion coefficient (even higher than TiB₂) and moderate elastic modulus (lower than SiC) [15–17]. With the addition of VB₂ particles in SiC matrix, the generation of thermal residual stresses and weakened Young's modulus, together with refined microstructure induced by Zener pinning of grain boundaries, can be expected in SiC-VB₂ composite, which thus has a great potential to obtain superior fracture toughness and strain-to-failure simultaneously. However, until now, the reports on SiC-VB₂ composite are scarce and the correlations between microstructure and mechanical properties have not been well studied [18]. Besides, pure and fine VB₂ powder is not commercially available. Many researches demonstrate that the internally synthesized inclusions have fine particle sizes and homogeneous distributions in SiC matrix, which enable the composites to gain more excellent

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properties than the conventional counterparts [14,19]. Therefore, the following reaction is currently proposed for the in-situ formation of VB₂ in SiC matrix:



The volume content of VB₂ in SiC-VB₂ composite can be adjusted by changing the quantity of SiC powder, and excess B₄C and C are used as sintering additives.

In the present work, 30 vol% VB₂ was synthesized to prepare highly toughened and strain tolerant SiC-VB₂ composite by pressureless densification, and the microstructures and mechanical properties of the composite were systematically investigated. Moreover, a comparison with the monolithic SiC ceramic was also included, in order to study the corresponding mechanisms for the enhanced toughness and strain tolerance of the composite.

2. Experimental procedure

The starting powders included α -SiC (UFP PA, purity 99%, mean particle size 0.36 μm ; SYCERA New Materials Co., Ltd., Shanghai, China), VO₂ (particle size 200–300 nm, provided by Shanghai Institute of Ceramics and the details were described elsewhere [20]) and B₄C (purity 97%, mean particle size 1.5 μm ; Mudanjiang Jingtangzuan Boron Carbide Co., Ltd, Mudanjiang, China). Phenolic resin (Shanghai QiNan Adhesive Material Factory, Shanghai, China) with a carbon yield of ~ 60 wt% after pyrolysis was used as a carbon source.

SiC-30vol%VB₂ ceramic composite (marked as sample SCV) was obtained over a two-step sintering process, including a first step for the in-situ formation of VB₂ and a second step for the pressureless densification. In the first step, α -SiC powder was added into the reactants in reaction (1), and additional 0.6 wt% B₄C and 1.5 wt% C were also introduced as sintering additives. Then the starting powders were mixed in ethanol and ball-milled for 12 h using SiC milling media. The dried mixture was heated at 1400 °C for 1 h in vacuum to complete the reaction. In the second step, the as-synthesized powder was ball-milled again in ethanol for 24 h. Then the dried slurry was crushed, sieved and uniaxially dry pressed in steel dies at ~ 60 MPa, followed by cold isostatic pressing at ~ 300 MPa for 180 s. Finally, the green compacts were pressureless densified at 2150 °C for 1 h in argon. All the heat-treatment processes were carried out in a high-temperature graphite resistance furnace (Zhuzhou Norbert High Temperature Instrument Ltd. Co., China). Monolithic SiC ceramic with the same additive compositions was also prepared for comparison and labeled as Sample SC.

Phase analyses of the samples were determined by X-ray diffraction (XRD; D8 Advance, Bruker AXS, Karlsruhe, Germany) using Cu K α radiation. The microstructures were observed using transmission electron microscopy (TEM; Tecnai G2 F20, FEI Co., Hillsboro, USA) and scanning electron microscopy (SEM; S-4800, Hitachi, Tokyo, Japan). The bulk densities were measured by the Archimedes method, and the theoretical densities were calculated according to the rule of mixtures using density values of 3.21 g cm⁻³ for SiC, 5.07 g cm⁻³ for VB₂, 2.51 g cm⁻³ for B₄C and 2.27 g cm⁻³ for C, respectively. Test beams and cylinders were ground and polished to a 1 μm finish using diamond abrasives. The three-point bending strengths of the beams (3 mm \times 4 mm \times 36 mm) were measured by a universal tester (Instron-1195, Instron, Canton, MA, USA) using a 30 mm span and a cross-head speed of 0.5 mm min⁻¹. The Young's modulus was measured on the cylinders ($\emptyset 30$ mm \times 10 mm) by ultrasonic pulse-echo method using a high-frequency pulser/receiver (Model 5900 PR, Olympus, MA, USA) according to the American Standard ASTM E 494-95. Vickers hardness was measured by Vickers indentation (Model 300, Tukon,

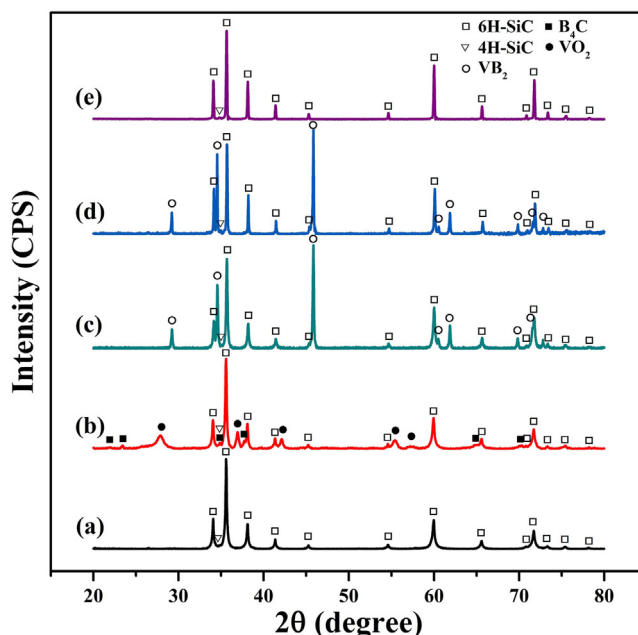


Fig. 1. XRD patterns of (a) the as-received SiC powder, (b) the starting powder of Sample SCV, (c) the synthesized SiC-VB₂ powder after vacuum soaking at 1400 °C, and the densified (d) SCV and (e) SC samples after sintering at 2150 °C in argon.

Canton, MA, USA) using a load of 5 kg and a dwell time of 10 s. Fracture toughness was evaluated by the indentation fracture (IF) method and calculated using the Antis equation [21]. Besides, in order to enhance the reliability of the fracture toughness values, the measurement was also conducted on bars (2 mm \times 4 mm \times 22 mm) using the single-edge notched beam (SENB) method with a span of 16 mm and a cross head speed of 0.05 mm min⁻¹. The depth and width of the notch were about 2 mm and 0.26 mm, respectively. All the mechanical properties were measured from at least five samples to calculate the average values.

3. Results and discussion

The XRD patterns for the as-received α -SiC powder, the dried starting powder of Sample SCV, the as-synthesized SiC-VB₂ powder, and the densified SCV and SC samples are shown in Fig. 1. The α -SiC powder was composed of 6H (ICDD-PDF: 74-1302) and a tiny amount of 4H (ICDD-PDF: 73-1664) polytypes, as shown in Fig. 1(a). The starting powder of Sample SCV exhibited the diffraction peaks of α -SiC (containing 6H and 4H polytypes), VO₂ and B₄C in Fig. 1(b). After vacuum heat-treatment at 1400 °C, the peaks of VO₂ and B₄C disappeared with the appearance of VB₂ peaks in Fig. 1(c), indicating the reaction was adequately completed. Although the excess B₄C and C were also present as sintering additives, they were possibly in amorphous states or too low concentrations to be detected in the XRD pattern. Fig. 1(d) demonstrated that both α -SiC and VB₂ peaks became sharper in sintered Sample SCV. Besides, the positions of α -SiC peaks remained basically unchanged owing to the very low solubility of V (3×10^{17} cm⁻³) and B (2.5×10^{20} cm⁻³) in SiC crystal and thus the quite limited dissolution of VB₂ in SiC [22,23]. Fig. 1(e) showed that only the diffraction peaks of 6H-SiC and 4H-SiC were detected in the monolithic SiC ceramic. By comparing all the above XRD patterns, it was found that 6H-SiC was always the main polytype of α -SiC while the peak intensities of 4H-SiC were consistently very weak throughout all the processes for both the SCV and SC samples. Thus, there was no apparent polytype transformation during the synthesis and sintering procedures of Sample SCV. Moreover, there was no distinct difference in the

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