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Ceramics International 39 (2013) 2107–2110

CERAMICS INTERNATIONAL

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Short communication

Microstructure and mechanical properties of silicon carbide monofilament reinforced zirconium diboride matrix composites

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Received 15 June 2012; received in revised form 4 July 2012; accepted 4 July 2012
Available online 20 July 2012

Abstract

Unidirectional SiC monofilament-reinforced ZrB_2 matrix composites were fabricated by hot pressing at 2173 K for 1 h under a uniaxial load of 30 MPa. The fracture toughness of SiC monofilament-reinforced ZrB_2 matrix composites was 6.1 MPa $m^{1/2}$, which was much higher than that of monolithic ZrB_2 and SiC particles reinforced ZrB_2 matrix composites. The improvement of toughness for SiC monofilament-reinforced ZrB_2 matrix composites was mostly attributed to the addition of the SiC monofilament, which could activate fiber pull-out and crack deflection. In addition, the high strength of SiC monofilament itself was also a major reason for the improvement of toughness of SiC monofilament-reinforced ZrB_2 matrix composites.

Keywords: A. Hot pressing; C. Mechanical properties; C. Toughness and toughening

1. Introduction

Zirconium diboride (ZrB_2) is of particular interest among the ultrahigh-temperature ceramics (UHTCs) due to its combination of suitable properties, such as high melting point, relatively low theoretical density, high electrical conductivity, good chemical inertness and super wear resistance. These make it a potential candidate for ultrahigh-temperature structural applications in aerospace, such as re-entry vehicles and hypersonic cruise aircraft [1–3]. However, its reliability in high-performance applications is still in doubt. This concern can be attributed to its low fracture toughness and resulting susceptibility to brittle fracture. Moreover, brittle fracture can lead to unexpected catastrophic failure, thus its fracture toughness must be improved.

The incorporation of a variety of elastic reinforcing phases which can activate toughening mechanisms such as crack deflection or crack bridging into the base phase to form a ceramic matrix composite is an advisable strategy. Follow this guideline, recent contributed reports on ZrB_2 matrix composites were obtained by the addition of short carbon fiber [4,5], silicon carbide whiskers [6,7] and SiC chopped fibers [8,9]. Compared with the above-mentioned additives, SiC monofilament has higher strength, elastic modulus and excellent thermal stability, which should be an excellent reinforcement for ceramic materials. SiC monofilament reinforced Si_3N_4 matrix ceramics were reported [10–12]. These materials showed relatively high fracture toughness. However, the microstructure and mechanical properties of SiC monofilament reinforced ZrB_2 matrix ceramics have not been reported.

The purpose of this work is to report the processing method and an initial investigation of the microstructure and fracture toughness of SiC monofilament reinforced zirconium diboride matrix ceramics (abbreviated as SiC_{mf}/ZrB_2).

2. Experimental

Commercially available ZrB_2 powder was supplied by Dandong Chemical Engineering Institute Co., Ltd, China, and the mean particle size and purity is 2 μm and 99%. Si_3N_4 powder used in this work was provided by Hebei Shinuorui New Material Co., Ltd, China, and the mean

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0272-8842/$36.00
http://dx.doi.org/10.1016/j.ceramint.2012.07.018

particle size and purity is 1 μm and 99%. Non-commercially available SiC monofilament was supplied by our lab, which was fabricated by CVD of β-SiC from methyltrichlorosilane on a carbon core and uncoated with a $(SiC–C)_n$ multilayer on the exterior of the monofilament [13–15]. The mean diameter of monofilament is approximately 140 μm and the carbon core is about 33 μm. Average tensile strength of SiC monofilaments is 1.4 GPa. The density is 3.1 g/cm^3. Fig. 1 shows a scanning electron micrograph of the monofilament.

Fabrication process of the SiC_{mf}/ZrB_2 was shown in Fig. 2. To fabricate the SiC_{mf}/ZrB_2, silicon nitride (3 wt%) was used as a sintering aid. The matrix powder and additive powder were ball-mixed for 5 h in an agate bottle using agate balls and ethanol as the grinding media. The SiC continuous monofilaments were cut into short filaments (5 cm). Short SiC monofilaments were unidirectionally arranged on the same plane and then stacked alternately with the mixed slurry in a graphite die. Ultimately 10 SiC monofilament layers were made in the mixed slurry. The mixture of ZrB_2 plus 10 vol% SiC monofilament was cold-pressing under a uniaxial load of 20 MPa to form a compact. Next, the compact was hot-pressed at a maximum temperature of 2173 K for 1 h under a uniaxial load of 30 MPa.

Specimens were polished and ultrasonic cleaned. The bulk density (ρ) of sintered specimens was measured using the Archimedes method. The microstructural features of the hot pressed composite were observed by scanning electron microscopy (SEM, Hitachi S-4800, Japan) with simultaneous chemical analysis by energy dispersive spectroscopy (EDS, EDAX Inc.). Phase composition was determined via X-ray diffractometry (Rigaku, Japan) using Cu Kα radiation. Flexural strength (σ) was tested in three point bending on $3 \times 4 \times 36$ mm^3 bars, using a 30 mm span and a crosshead speed of 0.5 mm/min. Fracture toughness (K_{IC}) was evaluated by a single-edge notched beam test with a 16 mm span and a crosshead speed of 0.05 mm/min using $2 \times 4 \times 22$ mm^3 test bars.

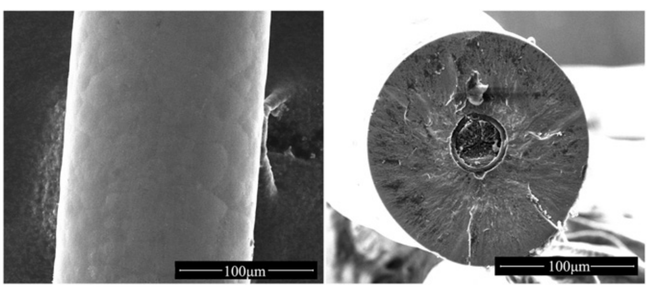

Fig. 1. SEM images of surface and cross-section of silicon carbide monofilament.

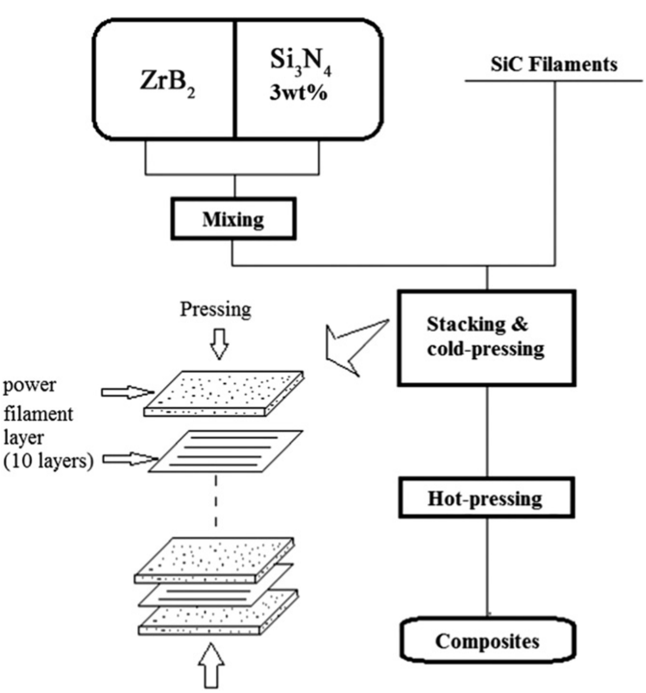

Fig. 2. Fabrication process of unidirectional SiC monofilament to reinforced ZrB_2 matrix composites.

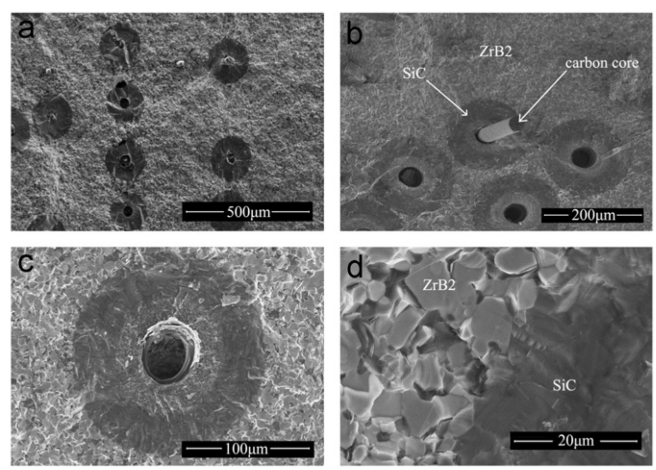

Fig. 3. (a) Fracture surfaces of SiC_{mf}/ZrB_2; (b) Core pullout on the fracture surface of SiC_{mf}/ZrB_2; (c) and (d) interface between SiC monofilament and ZrB_2 based.

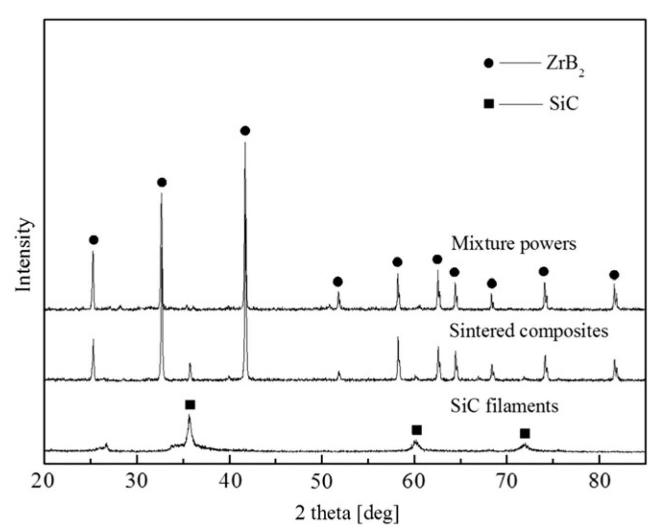

Fig. 4. XRD patterns of SiC_{mf}/ZrB_2 raw materials and sintered composites.

Table 1
Density and mechanical properties of the zirconium diboride matrix composites. (measured density ρ, fracture toughness K_{IC}, flexural strength σ).

Materials	σ (MPa)	K_{IC} (MPa m$^{1/2}$)	ρ (g/cm^3)	Relative density (%)
SiC_{mf}/ZrB_2	613 ± 59	6.1 ± 0.5	5.66	97.6
ZrB_2	565 ± 53	3.5 ± 0.3	6.26	99.8
SiC_p/ZrB_2	713 ± 48	4.1 ± 0.3	5.54	93.2

3. Results and discussion

The bulk density of SiC_{mf}/ZrB_2 billets was 5.66 g/cm^3. Using a rule of mixture calculation and assuming that the true densities are 6.1 g/cm^3 for ZrB_2, 3.1 g/cm^3 for SiC monofilament, the theoretical density of the SiC_{mf}/ZrB_2 was calculated to be 5.8 g/cm^3. Based on this true density, the relative density of the SiC_{mf}/ZrB_2 was calculated to be 97.6%.

SEM images of the fracture surface of the SiC_{mf}/ZrB_2 were shown in Fig. 3. Fig. 3(a) showed that the crack overcame not only propagating resistance in the matrix itself, but also the resistance to fracture of the SiC monofilament. Many undulations in the fracture surface of the composite showed that crack deflection should be a complex toughening mechanism. Fig. 3(b) showed that the SiC filaments were not pulled out, but the carbon cores which were the center core of the SiC monofilaments, were pulled out. As indicated in Fig. 3(b), pull-out of the carbon cores were visibly observed, therefore fiber pull-out must be one of the main fracture behaviors. As shown in Figs. 3(c) and (d), another characteristic feature of the fracture surface, which was helpful in identifying the toughening mechanism, was that the fracture surface of the SiC monofilaments was nearly coplanar with the fracture surface of the matrix. It seemed that SiC monofilaments in the composites retained higher strength than the based and the interface cohesion between filament and matrix was strong. On one hand, the high strength of SiC monofilament should be another reason of the improvement of the fracture toughness. On the other hand, too strong cohesion between SiC monofilament and matrix was difficult to induce non-brittle fracture. So improvement of interfacial layer between SiC monofilament and matrix was necessary. Other investigators found that coating layers should be perfect the interface between fiber and matrix components, then further increase the fracture toughness [12].

The XRD profiles from the mixed powders, SiC filaments and hot pressed material were shown in Fig. 4. According to the XRD patterns, no new phase was observed in the obtained composites that compared with the mixture powers and SiC filaments, which indicated that there was no appreciable chemical reaction between filament and matrix in sintering process. Thus, the assumption of reaction between SiC whisker and ZrB_2 particle as primary cause for the strong cohesion between filament and matrix could be ruled out, since no new phase was detected. As shown in Fig. 1 and Fig. 3(d), the surface of the SiC monofilament and the interface between filament and matrix were scraggly. Thus, the strong cohesion between filament and matrix might be a mechanical cohesion.

The mechanical properties of the SiC_{mf}/ZrB_2 were listed in Table 1. The measured fracture toughness as high as 6.1 MPa m$^{1/2}$ was much higher than that of monolithic ZrB_2 (3.5 MPa m$^{1/2}$) and ZrB_2 plus 10 vol% SiC particles ceramics (abbreviated as SiC_p/ZrB_2) (4.1 MPa m$^{1/2}$) [16]. The improvement in toughness was mainly attributed to carbon core pull-out and high filament strength, because these interaction effects absorbed fracture energy.

4. Conclusions

In conclusion, the ZrB_2 plus 10 vol% SiC monofilaments ceramic was fabricated by hot pressing. The relative density of the ZrB_2 plus 10 vol% SiC monofilaments ceramic was calculated to be 97.6%. The measured fracture toughness of the ZrB_2 plus 10 vol% SiC monofilaments ceramic was 6.1 MPa m$^{1/2}$, which increased by approximately 74% compared to the reported results of monolithic ZrB_2 (3.5 MPa m$^{1/2}$) and 49% for ZrB_2 plus 10 vol% SiC particles composites (4.1 MPa m$^{1/2}$). The observed toughening mechanisms were attributed to fiber pull-out and high filament strength. This study clearly showed that the introduction of SiC monofilaments to ZrB_2 ceramic is a promising way to improve the fracture toughness of this material.

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