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Fabrication and characterization of random chopped fiber reinforced reaction bonded silicon carbide composite

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Abstract

This paper deals with the microstructure and mechanical properties of reaction bonded silicon carbide reinforced with random chopped carbon fibers of 3 mm length. The composites were fabricated by dispersing chopped carbon fibers into bimodal SiC/C suspension, forming green body through slip casting, and then reaction sintering at 1700 °C. The effect of the chopped fiber fraction on microstructure and mechanical properties was evaluated. A significant increase of fracture toughness was obtained as the carbon fiber fraction approaches 30 vol.%. The chopped fibers had reacted with liquid silicon during reaction sintering, so little fiber pullout was observed. Crack deflection and bridging is the predominant mechanism for the composite toughening.

Keywords: A. Slip casting; B. Fibers; C. Toughness and toughening; E. Engine components

1. Introduction

Reaction bonded silicon carbide (RBSC) has attracted increasing research interest because it can provide mechanical properties superior to conventional sintered silicon carbide, with low sintering temperature, short sintering time, high relative density and near-net shaping. In several recent researches [1,2], RBSC has been selected as a main candidate for the preparation of large scale and ultra-lightweight optical mirrors because of its good shaping capability and high thermal stability. The major problem encountered, however, is the low damage tolerance for structure components [3]. Especially, for the development of large scale and light weight mirror, a higher mechanical strength for the RBSC blank is required so as to make it possible to machine a high precision reflection surface [4].

High performance RBSC can be obtained by improving its microstructure and mechanical properties, for example, by introducing fine SiC particle, alloyed melt, and BN [5–8]. The increment of the fracture toughness, achieved so far, still cannot meet the demands for the large scale components. Continuous fiber reinforcing is a common toughening technique for ceramic matrix composites, promoting the mechanical performance via fiber pulling-out, fiber debonding, crack deflection and so on [9–11]. Continuous carbon fiber is a prevailing selection and has been employed to reinforce silicon carbide in view of its high flexural strength and fracture toughness. However, the fabrication of continuous carbon fiber reinforced silicon carbide, generally through CVI (chemical vapor infiltration), PIP (polymer infiltration and pyrolysis), LSI (liquid silicon infiltration) and HP (hot pressing) [12,13], encounters the problem of complex processes, long produce period and high cost [14,15]. This limits its practical application for preparation of mirrors with large scale and complex shape.

Compared to continuous carbon fiber, chopped carbon fiber has the advantages of lower fabrication cost and high adaptability for conventional manufacturing techniques [16,17]. Since the physics behavior of carbon fibers is different along its longitudinal and radial directions, a distinct anisotropy occurs in mechanical as well as thermophysical properties for the fiber reinforced SiC composites. To the contrary, this anisotropy can be avoided by making a homogenous random distribution of the chopped fibers [18].

Slip casting is a simple and cheap consolidation process for producing RBSC ceramics with high density and homogeneous microstructure, and particularly is available to prepare the components with complex geometry. Since the green body

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obtained by slip casting may have the size as precise as designed, the post-processing on it, such as milling, turning, and drilling [19], may be greatly reduced. In the previous study [2], slip casting has been employed to fabricate near net shape, extremely complex and large scale mirrors. The purpose of this work is to investigate the microstructure and mechanical properties of the random chopped carbon fiber reinforced reaction bonded SiC fabricated by slip casting. The effect of carbon fiber fraction on mechanical properties of the ceramics was examined.

2. Experimental

2.1. Raw materials and fabrication

Continuous carbon fiber (IM400-6K, TOHO, Japan) with diameter of 6 μm was cut into short segments of 3 mm length. The elastic modulus and density of the continuous carbon fiber were 294 GPa and 1.74 g/cm^3, respectively. To remove the organic binder, the chopped carbon fibers were desized with 0.2 M dilute nitric acid for 24 h and then washed with deionized water. Then the chopped carbon fibers were pre-dispersed with ultrasound vibration. Here carboxymethyl cellulose (CMC, molecular weight 20,000, GONSO, Shanghai, China) was chosen as dispersant. Two commercial α-SiC (purity 98.5%, Huanyu, Zibo, China) powders of 10 μm and 60 μm, and amorphous carbon were chosen as the matrix. The raw powders together with dispersant and plasticizer were ball milled with silicon carbide balls in water for 10 h. Then the as-treated chopped carbon fibers were added into the SiC/C suspension and a further ball milling for another 2 h was made. Four specimens were prepared with different volume fraction of chopped carbon fibers, 10 vol.%, 20 vol.%, 30 vol.%, and 40 vol.%. They were denoted as SC10, SC20, SC30 and SC40, respectively. The slurry was casted in a plaster mould. After drying, the obtained green body was sintered by liquid silicon infiltration at 1700 °C for 90 min.

2.2. Characterization

Bulk density of the specimens was measured by the Archimedes method. Flexural strength was tested with crosshead speed of 0.5 mm/min and span of 30 mm. The tested bars were in dimension of 3 mm × 4 mm × 36 mm and were polished with diamond slurry of 3.5 μm. Fracture toughness was evaluated using a single edge notched beam (SENB) with crosshead speed of 0.05 mm/min and span of 20 mm. The tested bars were in dimension of 2 mm × 4 mm × 22 mm and

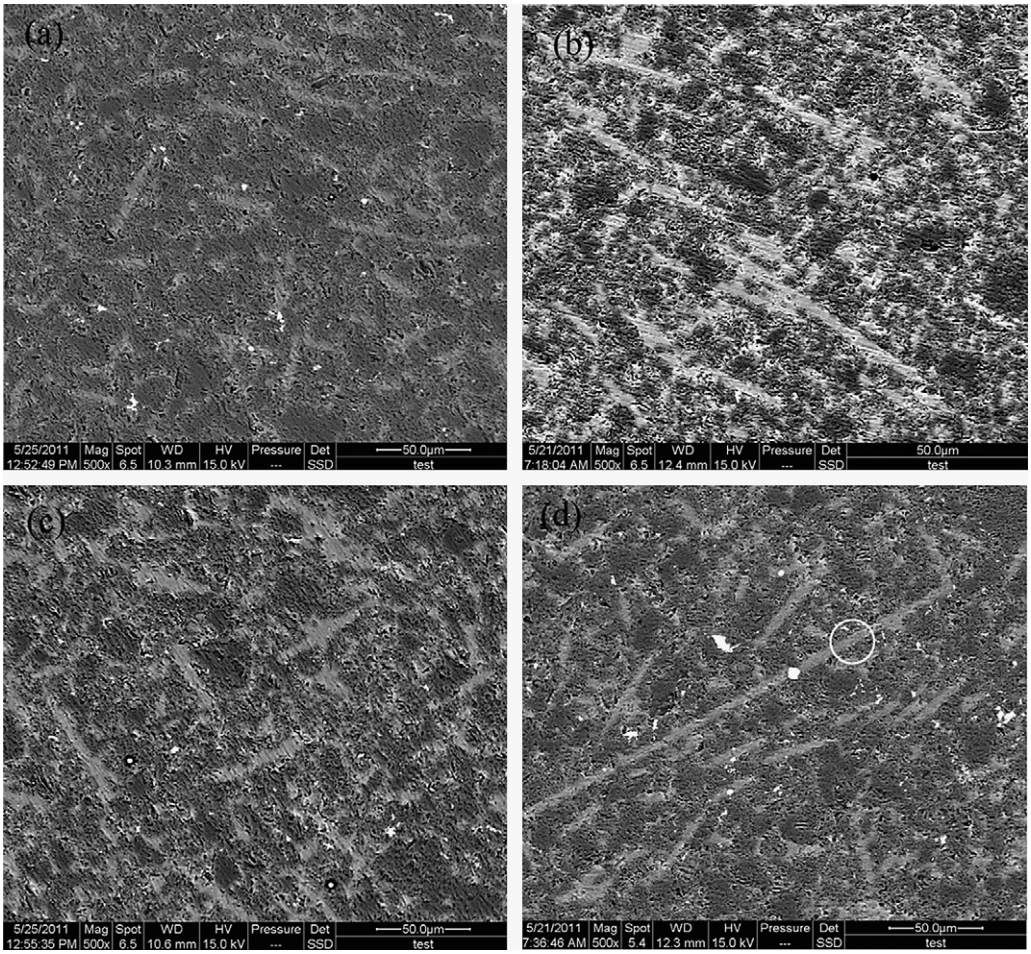


Fig. 1. Morphology (SEM, BSE) of the polished surfaces with fiber fraction range from 10 to 40 vol.%; (a), (b), (c) and (d) correspond to the specimens SC10, SC20, SC30 and SC40, respectively.

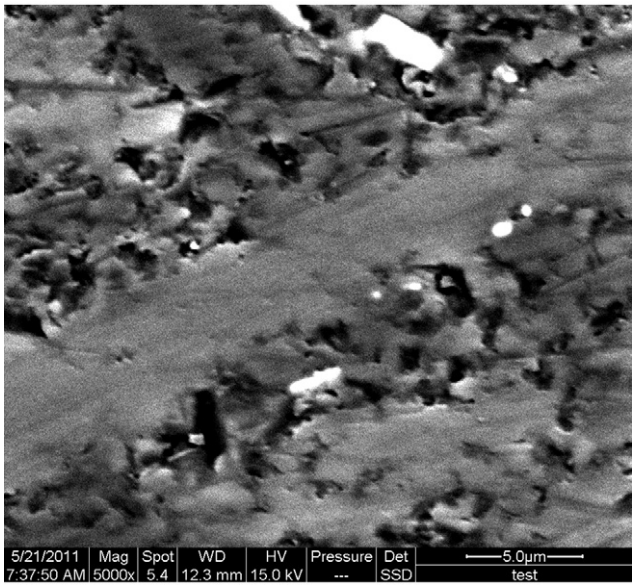


Fig. 2. Magnification of the random chopped fiber.

tested with a notch of 2 mm in depth and 0.02 mm in width. The data for each specimen was averaged over five tests. Microstructure of the polished and fracture surfaces was observed with laser scanning confocal microscope (OLS3100, OLYMPUS, Japan) and scanning electron microscopy (QUANTA 200, FEI, USA). Finally, a simultaneous chemical analysis was made with energy dispersive spectroscopy (EDS).

3. Results and discussion

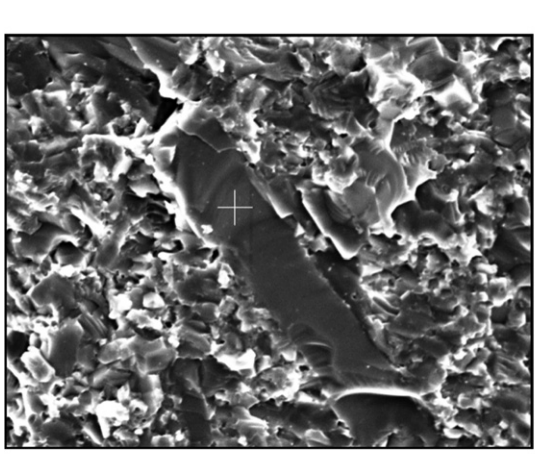
3.1. Microstructure

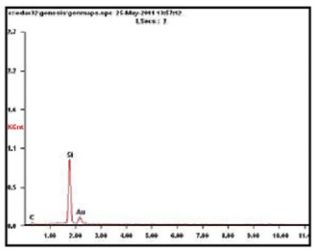
Due to the shorter length and higher dispersion compared with the continuous fiber reinforcements, the chopped fibers actually acted as fines when uniformly dispersed into the bimodal SiC/C suspension by ball milling. The uniformly dispersion results in a microstructural uniformity of the final products. Fig. 1 shows the back scatter micrographs of the polished surfaces of the finally obtained composites. The white phase is residual silicon and the gray phase is silicon carbide. The dark gray and light gray phases signify, respectively, the starting α-SiC and the β-SiC formed during sintering. Since the reaction temperature is high and dwell time is long, the carbon has been converted completely into β-SiC; therefore, no black phase corresponding to carbon can be seen in the matrix. From this figure, it is also seen that the distribution of the chopped carbon fibers is uniform. This uniform distribution will lead to an isotropy of the properties of the final composite, and improve the reliability of the load-bearing parts made of this composite. In addition, the uniform distribution of the chopped fibers indicates that the ball milling combined with CMC dispersant can prevent the fibers from aggregation.

The carbon fiber length in the final composite is 60 μm on average, far shorter than the starting one. This reduction is caused by high velocity impact during ball milling. To achieve the high fracture toughness with chopped fiber reinforcing, the homogenization of chopped fiber at the premise of avoiding damage is important. However, for high volume fraction dispersion, it has not been realized yet. Therefore, ball milling is an effective approach for chopped fibers dispersion and the reduction of the fibers length is inevitable.

Fig. 2 shows the morphology of a chopped fiber by locally magnifying the region circled in Fig. 1(d). No obvious fiber edge is observed, which implies a reaction of carbon fibers with the matrix during liquid silicon infiltration. The EDS diagram shown in Fig. 3 reveals that the composition of the fiber is Si and C. The atom ratio shown in the right of this figure represents a near stoichiometric SiC phase. It confirms that the carbon fiber has been completely converted into SiC for the Si–C reaction. As mechanical performances of the latter are lower than the former, the toughening of the chopped fibers is degraded by the interfacial reaction. Form the fracture surface, little pullout was observed. To optimizing the toughening of chopped fibers, a protective coating is necessary to prevent the Si–C reaction. This coating would enable the chopped fibers to pull out from the matrix during crack propagation.

The optical micrograph in Fig. 4 shows the morphology of the fracture surface of our random chopped fiber reinforced





Element	Wt%	At%
C K	28.10	47.75
Si K	71.90	52.25

Fig. 3. Morphology and surface composition of carbon fiber (fracture surface).

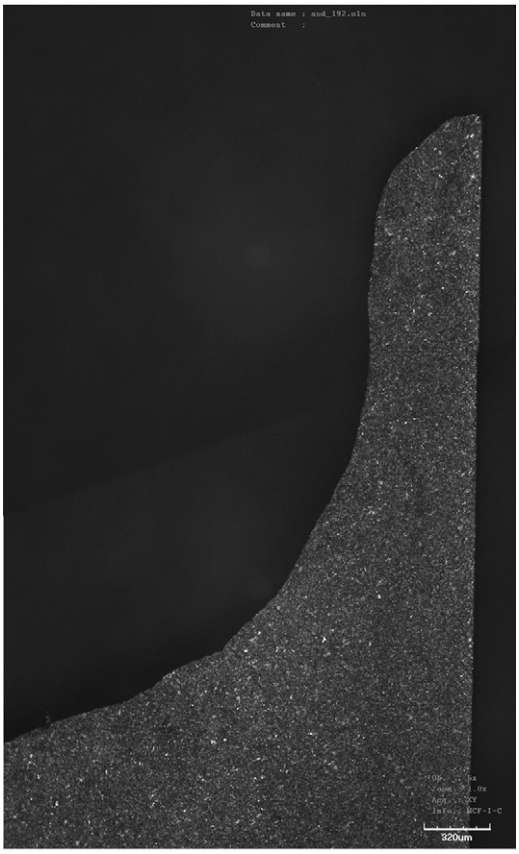


Fig. 4. Fracture surface of the random chopped fiber reinforced RBSC composite (optical micrograph).

RBSC composite. The fracture surface is smooth, indicating that the fracture behavior of the composite is a catastrophic one. We notice that, however, the crack path is more deflective than that of the monolithic RBSC ceramic, leading to an increased fracture area. This enlarged area consumes more fracture energy during crack propagating; hence, the fracture toughness of the random chopped fiber reinforced RBSC is improved. It is indicated that the random chopped fiber impels the crack path more deflective and makes the fracture surface larger. The

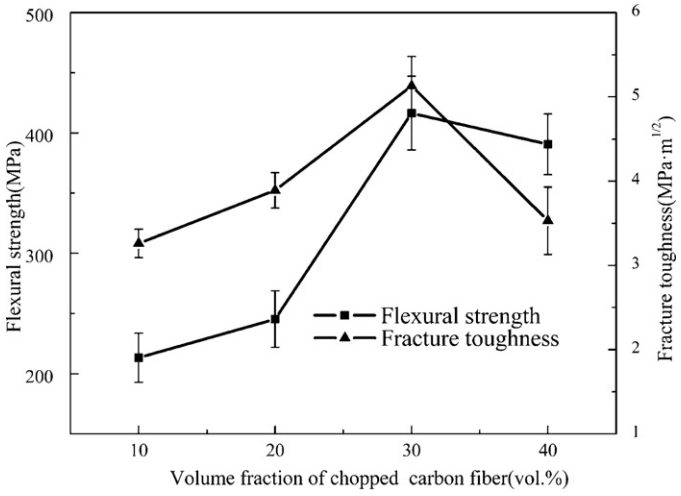


Fig. 5. Flexural strength and fracture toughness of the chopped random fiber reinforced RBSC composites as a function of fiber fraction.

reinforcing mechanism of random chopped fiber on mechanical properties would be discussed below.

3.2. Mechanical properties

Fig. 5 illustrates how the flexural strength and fracture toughness of the composite change with the volume fraction of chopped fibers. The flexural strength increasing from 213 MPa to 416 MPa is observed as the fiber fraction increases from 10 to 30 vol.%. But a decrease occurs when the fiber fraction is above 30 vol.%. The peak value of the flexural strength is about 50% larger than that of the monolithic ceramics from slip casting [19]. The variation of fracture toughness is similar to that of the flexural strength. The fracture toughness approaches its peak value of 5.1 MPa $m^{1/2}$ at 30 vol.% of carbon fibers.

The primary toughening mechanism of the fiber reinforced ceramic matrix composite is the fiber pulling-out, debonding, bridging and crack deflection. From the micrograph shown in Fig. 6(a), it is seen that the random chopped fiber exhibited a typical brittle fracture caused by the reaction with liquid

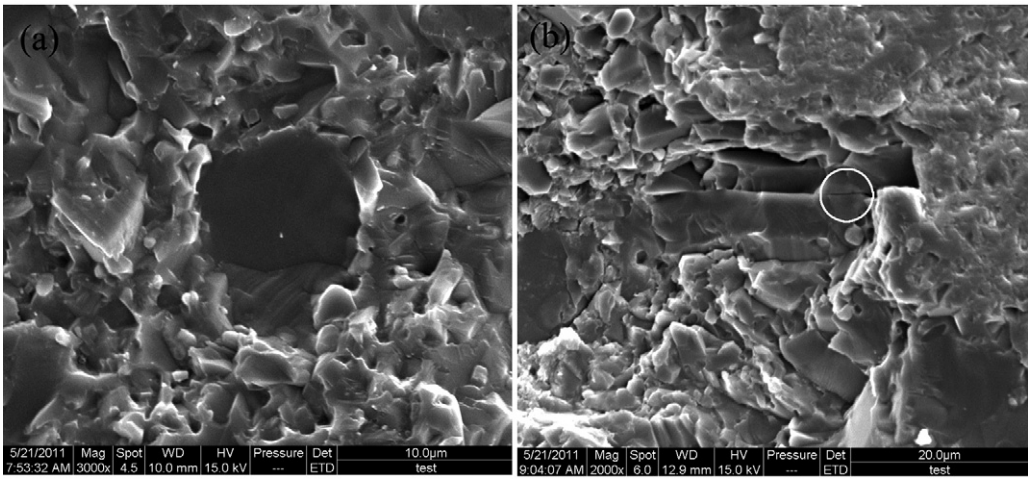


Fig. 6. Microstructure of the random chopped fiber in the composite; (a) fiber fracture because of Si–C reaction and (b) fiber debonding and surface damage.

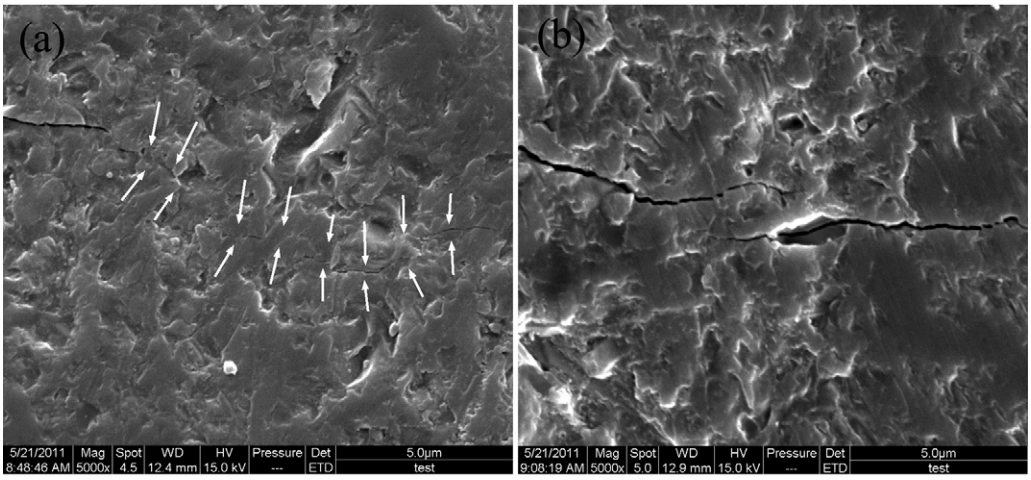


Fig. 7. Crack deflection (a) and bridging (b) in the composite.

silicon. But fiber debonding still can be observed, referring to Fig. 6(b). This implies that the chopped fibers consume a part of the fracture energy. A crack appearing between the fiber and the matrix, marked by the white circle in Fig. 6(b), indicates that there exists the possibility of the chopped fiber pulling-out through processing optimization.

Cracks on the polished surface were introduced by Vickers' indentation. The crack deflection and bridging are shown in Fig. 7. Fig. 7(a) illustrates one of the introduced crack paths, indicated by white arrows. It is seen that the crack deflection angle is large. This denotes that fracture energy is mainly consumed in a very limited area, and the predominant toughening mechanism is crack deflection and bridging.

3.3. Bulk density

The variation of bulk density of random chopped fiber reinforced RBSC composites with fiber fraction is shown in Fig. 8. There is a gradual increase for the bulk density in the range of 10–30 vol.% of the carbon fiber fraction. It is resulted from the reaction that the low density carbon was converted to high density β-SiC during the liquid silicon infiltration process. When the carbon fiber fraction exceeds 30 vol.%, there is a small decrease of the bulk density. This may be attributed to the increase of pores in the green body, which were filled by the residual silicon of lower density during liquid silicon infiltration.

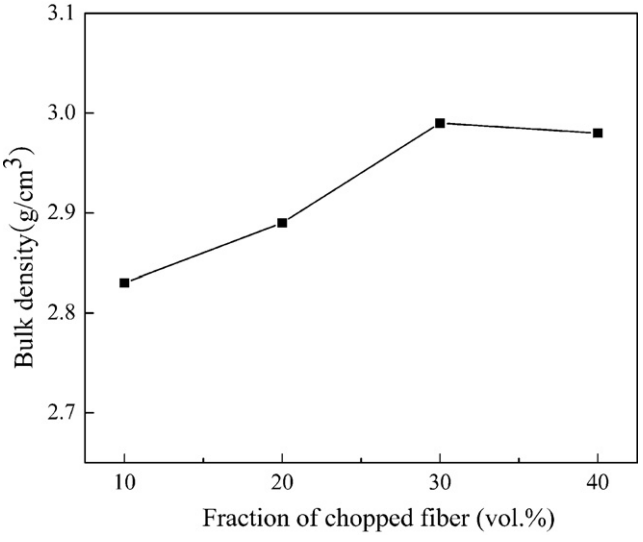


Fig. 8. Variation of bulk density of random chopped fiber reinforced RBSC composites with increase of fiber fraction from 10 to 40 vol.%.

As mentioned in [20], the residual silicon is a negative phase for RBSC because of its strong brittleness ($K_{IC} \leq 1.0$ MPa $m^{1/2}$). The increasing of residual silicon will damage the mechanical properties of the final composite. The drop of the bulk density at 40 vol.% of the carbon fiber fraction is in accordance with that of the flexural strength and fracture toughness shown in Fig. 5.

4. Conclusions

RBSCs were fabricated by uniformly dispersing chopped carbon fibers into bimodal SiC/C suspension. The main advantage of this method is the low cost and the mass production capabilities for fabricating large scale and complex shape components (especially, RBSC mirrors) with improved mechanical properties. The maximum values of flexural strength and fracture toughness are 416 MPa and 5.1 MPa $m^{1/2}$, respectively, superior to those of the monolithic RBSC formed via slip casting. The optimum fraction ratio of C_f/SiC is 30% when coarse and fine SiC particles are 60 and 10 μm in size, respectively. The interaction of fiber debonding, crack deflection and crack bridging consumes crack energy during propagation and leads to the improved toughness. During liquid silicon infiltration, the carbon fiber reacted with liquid silicon and was converted into SiC fiber. The pullout of the chopped fiber is seldom observed due to the strong bonding strength. This indicates that a protective layer on the carbon fiber is necessary to avoid the siliconization reaction of the carbon fiber during liquid silicon infiltration, and accordingly leads to a further improvement of the fracture toughness.

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