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ScienceDirect
Ceramics International 33 (2007) 1395–1398
CERAMICS INTERNATIONAL
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Short communication

Unidirectional carbon fiber and SiC particulate Co-reinforced fused silica composite

G.H. Zhou [a,b], S.W. Wang [a,*], X.X. Huang [a], J.K. Guo [a]

[a] Shanghai Institute of Ceramics, Chinese Academy of Sciences, 1295 Dingxi Road, Shanghai 200050, PR China
[b] Graduate School of Chinese Academy of Sciences, Beijing 100049, PR China

Received 8 September 2005; received in revised form 3 March 2006; accepted 14 April 2006
Available online 25 September 2006

Abstract

Unidirectional carbon fiber (uni-C_f) reinforced fused silica composite with the addition of 20 wt.% SiC particulate (SiC_p) (uni-C_f/SiO_2 + 20 wt.% SiC_p) was prepared by slurry infiltration and hot-pressing. The room temperature mechanical properties were investigated and the fracture features of composites were observed. While the flexural strength parallel to the fiber direction decreased from 667.3 to 431.8 MPa, that of perpendicular to the fiber direction increased from 18.0 to 54.3 MPa for uni-C_f/SiO_2 composite after the addition of SiC_p. Obviously, the anisotropy of mechanical properties was strongly modified. The increased flexural strength might be ascribed to the increase of the fiber/matrix interfacial bonding strength caused by the SiC_p addition.

Keywords: B. Composites; C. Mechanical properties; D. Glass ceramics; D. SiC; Fused silica

1. Introduction

In the area of space lightweight structural materials, carbon fiber reinforced glass matrix composites have demonstrated a wide range of attributes which include high strength, high stiffness, excellent toughness, low density, unique wear resistance, and environmental stability for structural applications [1–4]. Fused silica is one of the glass matrix material for heat shields and space lightweight structural applications because of its prominent properties such as low density (2.2 g/cm^3), low coefficient of thermal expansion (CTE), low thermal conductivity, high softening temperature and excellent chemical inertness [5,6]. However, monolithic fused silica is rarely used due to its intrinsic brittleness and very low mechanical properties [7]. In 1970s, Guo et al. studied the unidirectional carbon fiber reinforced fused silica glass composite. The results showed its flexural strength was 12-fold that of fused silica, its impact strength was upgraded by 40-fold, and its work of fracture was increased by three orders of magnitude [8]. However, this composite showed anisotropy in mechanical properties. That is, flexural strength parallel to the fiber direction was up to 600 MPa. However, its strength perpendicular to the fiber direction was lower than 20 MPa. In 1990s, Han et al. prepared a fused silica matrix composite reinforced by chopped carbon fiber and Si_3N_4, BN particle [9–11]. Though mechanical properties of this composite did not show anisotropy, a flexural strength of 73.2 MPa was obtained and the reinforced effort was far from being comparable with those of fiber yarn [8,12]. The present work was undertaken to modify the anisotropy of unidirectional carbon fiber reinforced SiO_2 composite (uni-C_f/SiO_2) with the addition of submicron SiC particulate (SiC_p). The mechanical properties and microstructure of the hot-pressed composites were investigated. The toughening mechanism of the composite was discussed.

2. Experimental procedures

Fused silica powder (SiO_2 >99 wt.% pure, and average particle size around 2.8 μm), PAN-based carbon fiber (2800 MPa average tensile strength, and 6–7 μm in diameter), and silicon carbide powder (α-SiC >98 wt.% pure, and average particle size around 0.5 μm) were used as starting materials. The powders were mixed in deionized water with

* Corresponding author. Tel.: +86 21 52414320; fax: +86 21 52413903.
E-mail address: swwang51@mail.sic.ac.cn (S.W. Wang).

0272-8842/$32.00
doi:10.1016/j.ceramint.2006.04.008

carboxymethyl cellulose (CMC) as a binder and isopropyl alcohol as a dispersant, and then ball-milled with agate balls. The prepreg was prepared by infiltrating the continuous carbon fiber into the slurry and then dried, stacked in a graphite die and hot-pressed at 1350 °C and 20 MPa in a N_2 atmosphere. For comparison, fused silica and unidirectional carbon fiber reinforced fused silica composites were also prepared. The content of carbon fiber was approximately 30 vol.% in the composites.

Density measurements were performed based on Archimedes principle. The specimens were machined into bars of 36 mm × 4 mm × 3 mm to measure the flexural strength by the three-point bending method with a span of 30 mm and a cross-head speed of 0.5 mm/min at room temperature (RT) in air. Single-edge notched-beam (SENB) samples were fabricated by notching the segments of tested flexure specimens with a 0.20 mm thick diamond wafering saw. The 30 mm × 6 mm × 3 mm SENB samples were tested in three-point loading with a span of 24 mm and a cross-head speed of 0.05 mm/min. Fracture toughness K_{IC} was calculated by the ASTME 399-74 formula [13]. The flexural strength and the fracture toughness measurements were conducted by Instron-1195 testing machine. Five specimens were tested for each sample. The fracture surface of composite was observed by electron probe X-ray microanalyser (EPMA, JXA-8100). The microstructural features of interface were examined using transmission electronic microscope (TEM, Model 200CX, JEOL, Japan).

3. Results and discussion

Table 1 is the mechanical properties of several SiO_2-based composites. Enhanced by unidirectional carbon fiber, the flexural strength and the fracture toughness of the hot-pressed fused silica increased from 54.8 to 667.3 MPa and 1.0 to 20.1 MPa m$^{1/2}$ parallel to the fiber direction ($||C_f$), respectively. Nevertheless, the flexural strength of uni-C_f/SiO_2 composite was only 18.0 MPa perpendicular to the fiber direction ($\perp C_f$). Parallel to the fiber direction, the reinforcing effect of C_f was significant and the continuous fiber played an important role in carrying the load. On the contrary, perpendicular to the fiber direction, there was no C_f to carry the load and the interstices between carbon fibers resulted in the decrease of the mechanical properties, to values lower than the matrix itself. After SiC_p addition, it was seen that though the flexural strength parallel to the fiber direction decreased from 667.3 to 431.8 MPa, that perpendicular to the fiber direction increased from 18.0 to 54.3 MPa, and the fracture toughness also had a slight increase from 20.1 to 21.9 MPa m$^{1/2}$. This is because the viscosity of fused silica increased after SiC_p addition, which would retard the process of densification at the same sintering temperature. Density measurements showed the relative density (R.D.) of uni-C_f/SiO_2 composite was 97.6%. However, R.D. of uni-C_f/SiO_2 + 20 wt.% SiC_p composite was 95.0%. The lower R.D. was responsible for the decrease of flexural strength parallel to the fiber direction for uni-C_f/SiO_2 + 20 wt.% SiC_p composite. On the other hand, the increase of flexural strength perpendicular to the fiber direction may be attributed to the enhancement of the fiber/matrix interfacial bonding strength by the addition of SiC_p, which was similar to the case of 10 vol.% chopped C_f/SiO_2 + 5 vol.% Si_3N_4 composite [14].

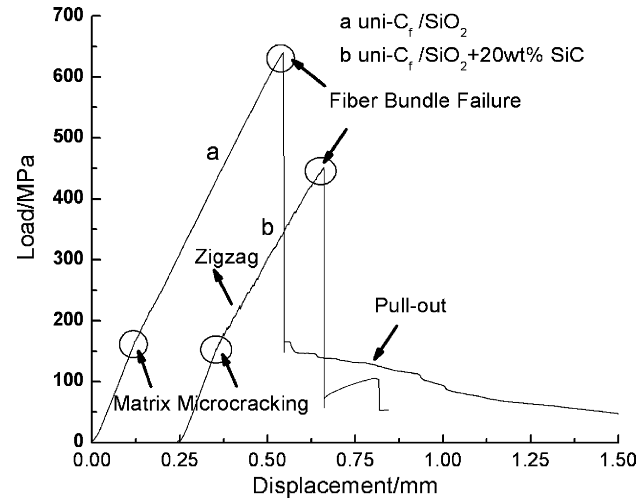

Fig. 1. Load–displacement curves: (a) uni-C_f/SiO_2 and (b) uni-C_f/SiO_2 + 20 wt.% SiC_p.

Fig. 1a and b show the typical load–displacement curves of uni-C_f/SiO_2 and uni-C_f/SiO_2 + 20 wt.% SiC_p composites, respectively. With the increase of the load, the two samples exhibit elastic response in the initial stage, and then a deviation appears at a load of about 170 MPa, indicating occurrence of microcracking in the matrix. After that, the second elastic response appears up to the maximum load where a significant drop in load occurs, which is attributed to fiber bundle failure. The final stage is the non-linear region—the tail of curve, revealing fiber pull-out, bridging, and sliding [6,15]. There are two differences in the load–displacement curves of the two samples. Firstly, the second elastic response of uni-C_f/SiO_2 composite is smooth (Fig. 1a), whereas, that of uni-C_f/SiO_2 + 20 wt.% SiC_p composite is zigzag, which is ascribed to

Table 1
Properties of SiO_2-based composites

Properties	Hot-pressed fused silica	Uni-C_f/SiO_2	Uni-C_f/SiO_2 + 20 wt.% SiC_p	10 vol.% chopped C_f/SiO_2 + 5 vol.% Si_3N_4 [14]
Density (g/cm^3)	2.17	2.02	2.10	2.15
Flexural strength (MPa)	54.8	667.3 ± 33.9 ($||C_f$) 18.0 ± 2.0 ($\perp C_f$)	431.8 ± 28.3 ($||C_f$) 54.3 ± 1.9 ($\perp C_f$)	73.2
Fracture toughness, K_{IC} (MPa m$^{1/2}$)	1.0	20.1 ± 2.0 ($||C_f$)	21.9 ± 1.4 ($||C_f$)	2.40

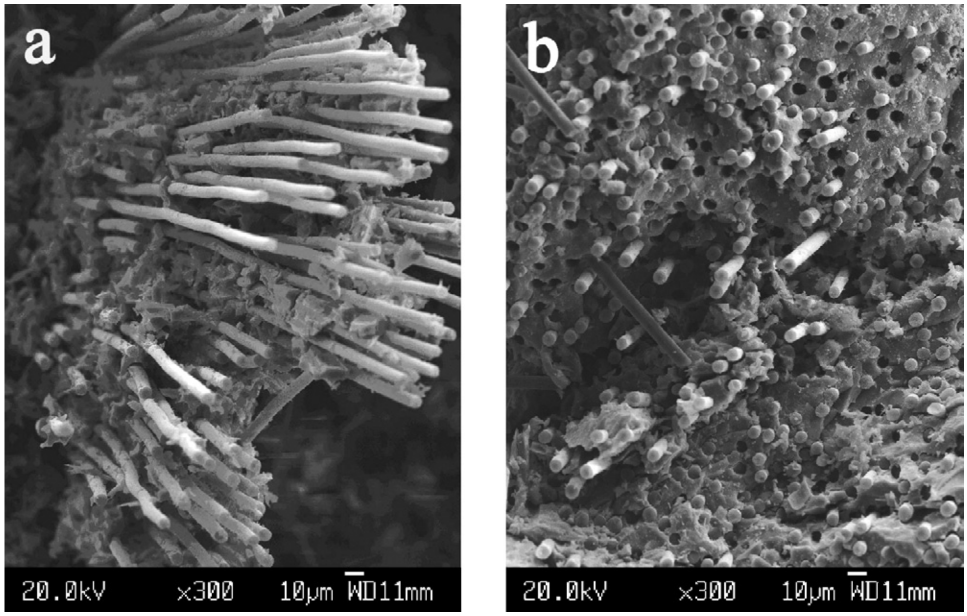

Fig. 2. The morphology of fracture surface: (a) uni-C_f/SiO_2 and (b) uni-C_f/SiO_2 + 20 wt.% SiC_p.

microcracking induced by the mismatch of the thermal expansion coefficients (CTE) between SiC_p and the SiO_2 matrix [6,15,16]. This microcracking also resulted in the decrease of flexural strength parallel to the fiber direction after SiC_p addition. The second difference is the non-linear region—the tail of load–displacement curves. The tail length of uni-C_f/SiO_2 composite is longer than that of uni-C_f/SiO_2 + 20 wt.% SiC_p composite. These two differences would be further discussed in the next part.

Typical fractographs of the composite are shown in Fig. 2. Without SiC_p addition (Fig. 2a), extensive fiber pull-out was observed in the fracture surface, and accompanying with very large pull-out lengths. While, fiber pull-out was short after SiC_p addition (Fig. 2b). Therefore, the different tail lengths in the load–displacement curves resulted for the two samples, respectively. Fiber pull-out lengths can also explain why flexural strength (parallel to the fiber direction) of uni-C_f/SiO_2 composite was higher than that of uni-C_f/SiO_2 + 20 wt.% SiC_p composite. In the light of interface theory of ceramic matrix composites (CMCs), strong fiber/matrix interfacial bonding results in the matrix microcracks propagating through the fiber with the increase of load. Thus, the fibers are easily broken. Whereas, in the case of weak fiber/matrix interfacial bonding, the matrix microcracks propagate along the weak interface, which results in the fiber/matrix interface debonding. Consequently, fibers are easily pulled out. According to the fiber pull-out lengths, it can be concluded that the fiber/matrix interfacial bonding of uni-C_f/SiO_2 + 20 wt.% SiC_p composite was stronger than that of uni-C_f/SiO_2 composite, which might be related to the fiber/matrix interfacial sliding stress.

Thouless et al. [17] reported that the distribution in fiber pull-out lengths in unidirectional CMCs should be related to the fiber/matrix interfacial sliding stress, fiber diameter, and Weibull modulus of the fiber. Presently, fiber/matrix interfacial

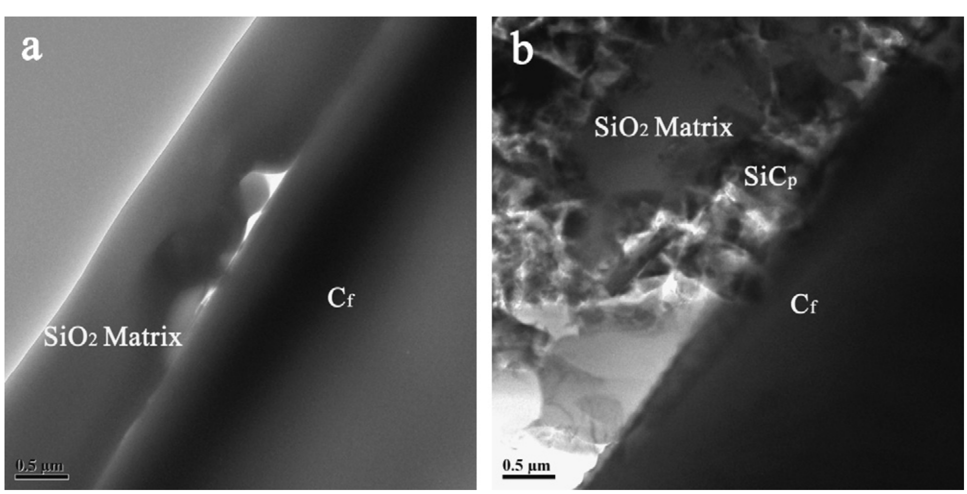

Fig. 3. The transmission electron micrographs of composites: (a) uni-C_f/SiO_2 and (b) uni-C_f/SiO_2 + 20 wt.% SiC_p.

sliding stress should play an important role in the fiber pull-out lengths. Fig. 3a and b show microstructures at the fiber/matrix interface for these two samples. Without SiC_p addition (Fig. 3a), the fiber/matrix interface is clear but a pore is entrapped. On the other hand, partial SiC_p were sandwiched into the fiber/matrix interface in the SiC_p added sample (Fig. 3b). The interfacial sliding stress caused by SiC_p at the fiber/matrix interface enhanced the interfacial bonding strength and retarded the slippage. As a result, during flexural test, the subtle effect of interface sliding stress among SiO_2 matrix, SiC_p and carbon fibers resulted in the zigzag curve of the second elastic response and an increase of flexural strength perpendicular to the fiber direction.

4. Conclusions

(1) Uni-C_f/SiO_2 and uni-C_f/SiO_2 + 20 wt.% SiC_p composites were prepared by slurry infiltration and hot-pressing. A flexural strength of 667.3 MPa and fracture toughness of 20.1 MPa $m^{1/2}$ parallel to the fiber direction for the uni-C_f/SiO_2 was attributed to the fiber pull-out.
(2) The flexural strength perpendicular to the fiber direction increased from 18.0 to 54.3 MPa after the addition of SiC_p. The anisotropy of mechanical properties was strongly modified for the uni-C_f/SiO_2 composite.
(3) The dispersion of SiC_p might enhance the interfacial sliding stress at the fiber/matrix interface, which contributed to the increase of the fiber/matrix interfacial bonding strength and the flexural strength perpendicular to the fiber direction.

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