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Low temperature mechanical behavior of 1D-C_f/SiO_2 composite

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Abstract

Unidirectional carbon fiber reinforced fused silica (1D-C_f/SiO_2) composite was prepared by slurry infiltration and hot-pressing. The flexural strength and the coefficient of thermal expansion (CTE) at room and liquid nitrogen temperature (77 K) were investigated. The flexural strength of the composite tested at 77 K was 878 MPa, higher than that 667 MPa at room temperature. Moreover, the CTE of the composite at 77 K was higher than that at room temperature. Due to the difference of CTE between the matrix and fiber, gaps appeared at the fiber/matrix interface of as-prepared specimens. However, they may be healed up because of the thermal expansion of carbon fiber at 77 K. It led to a higher interfacial sliding resistance and changed the weak fiber/matrix interfacial bonding. Thus, it was helpful for the load transfer from matrix to fiber.

Keywords: B. Composites; B. Interfaces; C. Mechanical properties; C. Thermal expansion

1. Introduction

In the area of aerospace 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]. Carbon fiber reinforced fused silica (C_f/SiO_2) composite is one of the glass matrix composites for heat shields because of its prominent properties such as low density (~1.95 g/cm^3), low coefficient of thermal expansion (CTE, 0.69×10^{-6} K^{-1}), low thermal conductivity, high softening temperature, and excellent chemical inertness [5,6]. The C_f/SiO_2 composite is expected to be used as lightweight structural materials in the aerospace environment with temperature change. The application of C_f/SiO_2 at high temperature has been demonstrated [7]. However, at low temperature, no data has been obtained to support this material to expand its application. Low temperature may result in the changes of some properties such as mechanical and thermal physical properties. Especially, the mechanical properties at low temperatures are critical to the structure design and safety of the spacecraft. Thus, it is necessary to evaluate thermal and mechanical properties of the composites at low temperatures. At present, the low temperature mechanical properties are focused on alloys [8], metal matrix composites (MMCs) [9,10], and polymer matrix composites (PMCs) [11–13]. However, little has been devoted to ceramics matrix composites (CMCs).

In this paper, the flexural strength and CTEs of the unidirectional C_f/SiO_2 (1D-C_f/SiO_2) composite at liquid nitrogen temperature (77 K) are investigated, providing some necessary information for their applications in aerospace environment. For comparison, the corresponding data at room temperature are also reported.

2. Experimental procedures

Home-made fused silica powder (99 wt.% purity, average particle size 2.8 μm) and PAN-based carbon fiber (average tensile strength 2800 MPa, 6–7 μm in diameter) were used as starting materials. The slurry was prepared by general ball-milling fused silica powders with carboxymethyl cellulose as a binder and isopropyl alcohol as a dispersant, using agate balls and deionized water as media. The prepreg was prepared by infiltrating the continuous carbon fiber into the as-prepared slurry and then dried, stacked in a graphite die

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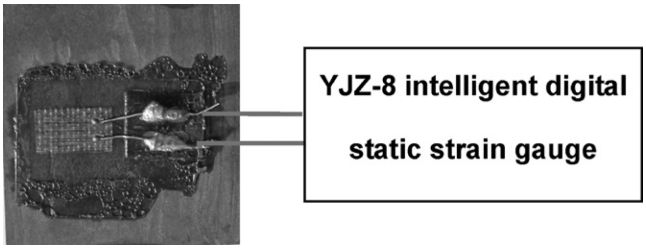

Fig. 1. The schematic of CTE test at 77 K.

and hot-pressed with 20 MPa at 1350 °C under N_2 atmosphere. The content of carbon fiber was approximately 30 vol.% in the composite. The fractured surface of the prepared composite was observed by electron probe microanalyser (EPMA, JXA-8100, Japan).

The specimens for flexural strength were machined into bars with dimension of 36 mm × 4 mm × 3 mm before a three-point bending test was conducted at a cross-head speed of 0.5 mm/min and a span of 30 mm on a universal testing machine (Instron-5566). Five bars were tested for each measurement.

Flexural strength σ_f can be determined by the maximum load value as shown in the following equation [14]:

$$\sigma_f = \frac{3PL}{2bd^2} \tag{1}$$

where σ_f is the flexural strength (maximum stress at mid-span), P the applied load that leads the specimen to fail, L the support span, b the specimen width, and d the specimen thickness.

Effective engineering modulus E can be obtained from the slope of the initial straight line of the load–displacement curve by the following equation [12]:

$$E = \frac{L^3}{48I}\frac{F}{\delta} \tag{2}$$

where F/δ is the slope of the load–displacement curve and I the cross-sectional inertia of the specimen.

The specimens for CTE test were machined into pieces with dimension of 25 mm × 25 mm × 2 mm and then the wire strain gauge was attached to each side of the specimen. The subtle strain ($\mu\varepsilon$) is obtained from the subtle deformation from room temperature to liquid nitrogen temperature (77 K) by YJZ-8 intelligent digital static strain gauge (Tairui Jinxing Instrument Ltd., Beijing). Fig. 1 shows the schematic of CTE test. Finally, the CTE (α) is calculated by the following equation:

$$\alpha(T) = \frac{\mu\varepsilon(T)}{T - T_0}, \qquad \mu\varepsilon(T) = \frac{l(T) - l_0(T_0)}{l_0(T_0)} \tag{3}$$

where T_0 is the room temperature, T the tested temperature (liquid nitrogen temperature, 77 K in the present work), l_0 the specimen length at room temperature, and l the specimen length at the tested temperature.

Table 1
The flexural strength and CTEs at room temperature and 77 K

	Room temperature	77 K
Flexural strength, C_f (MPa)	667 ± 34	878 ± 47
CTE ($\times 10^{-6}$ K^{-1})		
C_f	0.69	2.51
$\perp C_f$	–	2.32

3. Results and discussion

Table 1 lists the flexural strength and CTEs of uni-C_f/SiO_2 composites measured at room temperature and 77 K, respectively. It can be seen that the flexural strength increases by 31.6% (from 667 MPa to 878 MPa) when the tested temperature decreases from room temperature to 77 K. Similarly, the CTEs of the composites at 77 K increase about 2.5 times (2.51×10^{-6} K^{-1} in direction parallel to the carbon fiber and 2.32×10^{-6} K^{-1} in direction perpendicular to the carbon fiber), comparing with that at room temperature (0.69×10^{-6} K^{-1} in direction parallel to the carbon fiber).

Fig. 2a and b shows the typical load–displacement curves of 1D-C_f/SiO_2 composite at room temperature and 77 K, respectively. With the increase of the load, either of the two curves exhibited elastic response in the initial stage, and then appeared a deviation at higher load, indicating the occurrence of microcrackings in the matrix. After that, the second elastic response appeared and continued up to the maximum load where a significant drop in load occurred, which was attributed to the failure of the fiber bundle. The final stage is a non-linear region, i.e. the tail of curve, revealing fiber pull-out, bridging, and sliding [6,15]. However, there are two distinct differences in the two load–displacement curves. Firstly, the load indicating the occurrence of microcrackings in the matrix is 60.5 MPa at 77 K, which is much lower than that 166.4 MPa at room temperature, as shown by the insert in Fig. 2. According to Eq. (2), the calculated moduli E are 340 GPa and 85 GPa at 77 K and room temperature, respectively. It indicates that microcracking initiation in the matrix occurs at a lower load

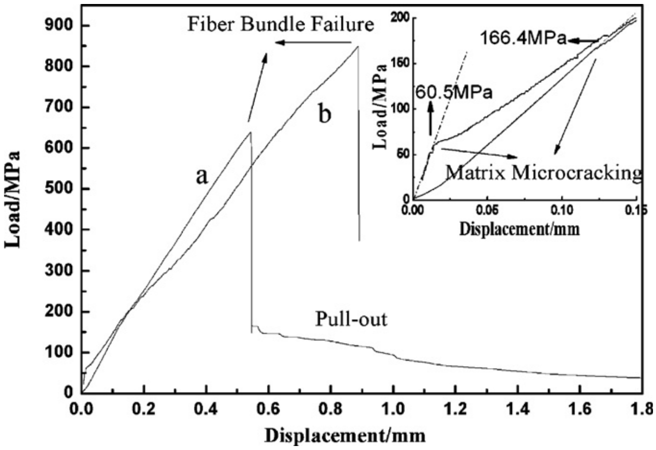

Fig. 2. Load–displacement curves of 1D-C_f/SiO_2: (a) tested at room temperature and (b) tested at 77 K. Insert: the magnification of the initial slope curves.

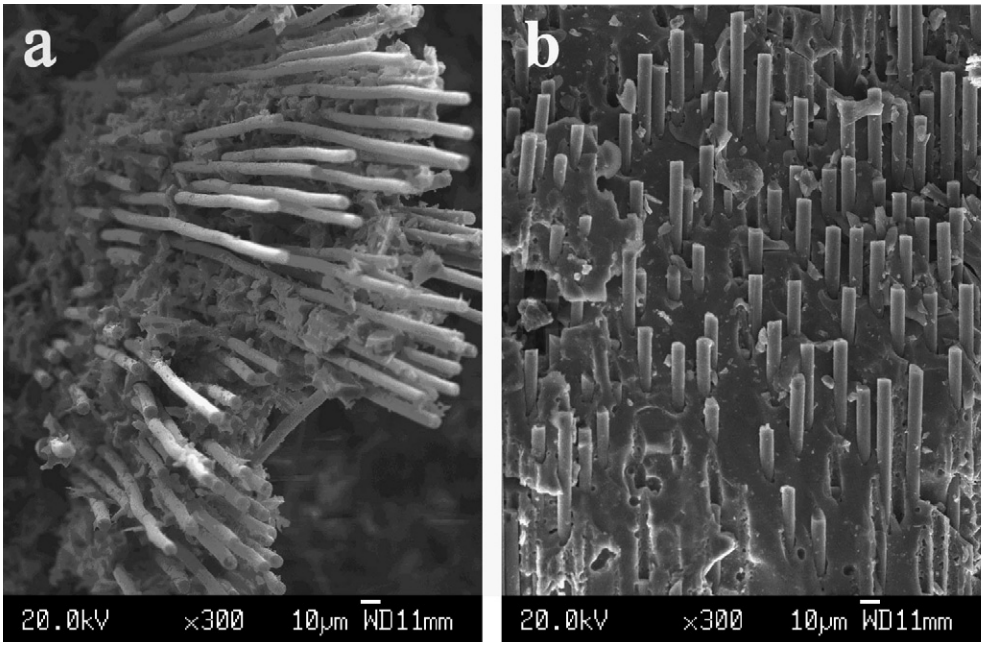

Fig. 3. The fracture morphologies of 1D-C_f/SiO_2: (a) at room temperature and (b) at 77 K.

level at 77 K. According to Ref. [16], the axial CTE of carbon fiber at room temperature is approximately $1 \times 10^{-6}\,K^{-1}$ that well matches that of fused silica matrix ($0.54 \times 10^{-6}\,K^{-1}$), reasonably induced that the matrix does not contain any transverse pre-existing cracks incurred by the residual thermal stresses in the 1D-C_f/SiO_2 composite. However, on the basis of the experimental results listed in Table 1, the specimen expands in the direction parallel to the carbon fiber when it is soaked in liquid nitrogen. Scott [17] and Scheel and Heuse [18] reported that the CTE of fused silica was approximately $0.5 \times 10^{-6}\,K^{-1}$ at 77 K, almost the same as that at room temperature. So the expansion of 1D-C_f/SiO_2 composite at low temperature should be attributed to the expansion of carbon fiber. In the direction parallel to the carbon fiber, the residual stresses of carbon fiber should be compressive while that of fused silica matrix is tensile. This is the reason for the microcrackings initiates at lower load at 77 K.

Curtin et al. pointed out that the deformation and failure of the composite are entirely controlled by the fiber properties in CMCs when the matrix cracking reaches the fully saturated state prior to composite failure [19]. Similarly, in the present work, the ultimate flexural strength and the displacement are mainly governed by the carbon fiber after the matrix microcrackings initiates, as shown in Fig. 2. Wang et al. [20] have investigated the single filament tensile strength of carbon fiber at 77 K and found that the average tensile strength increases by 11.6% with respect to that at room temperature and the strain extension is also augmented, which is also consistent to the above-mentioned results.

Fig. 3 shows typical fractured surface morphologies of the as-prepared composite. At room temperature (Fig. 3a), extensive fiber pull-out with very large pull-out lengths is observed. At 77 K (Fig. 3b), fiber pull-out is obviously shorter. It may result from the difference of the CTE and corresponding fiber/matrix interfacial bonding at different temperatures. As well known, the CTE is anisotropic due to the orientation of graphite grain. The CTE perpendicular to the carbon fiber varies from $9 \times 10^{-6}\,{}^\circ C^{-1}$ at 200 °C to $20 \times 10^{-6}\,{}^\circ C^{-1}$ at 1000 °C while that of the fused silica is just $0.54 \times 10^{-6}\,{}^\circ C^{-1}$ [21]. Gap would spontaneously appear at the fiber/matrix interface when the 1D-C_f/SiO_2 composite cooled-down from the fabricated temperature to room temperature, which has been reported in our previous work [22]. In addition, Stawovy et al. [23] also observed similar phenomena in the Nicalon® SiC-based fiber reinforced Blackglas® composite. On the other hand, in the direction perpendicular to the carbon fiber, the residual stress of carbon fiber is tensile while that of fused silica matrix is compressive. According to Curtin's theory, there is just a low value of interfacial sliding resistance due to residual roughness-induced clamping in the 1D-Cf/SiO_2 system [19,23]. Taking a CTE of $0.5 \times 10^{-6}\,K^{-1}$ for fused silica at 77 K into account, it is believed that the increase of CTE perpendicular to the carbon fiber from $0.69 \times 10^{-6}\,K^{-1}$ to $2.32 \times 10^{-6}\,K^{-1}$ may be attributed the higher thermal expansion of the carbon fiber than that of fused silica at 77 K. Thus, the gaps at the fiber/matrix interface are healed up, and then the radial residual stresses at the fiber/matrix change from tensile to compressive, leading to a higher sliding resistance. These would be helpful for the load transfer from matrix to fiber. Thus, the flexural strength at 77 K increases and the fiber pull-out is optimized and shorter than those at room temperature.

4. Conclusions

1. 1D-C_f/SiO_2 composite was prepared by slurry infiltration and hot-pressing. Flexural strength of the composite at 77 K

was 878 MPa, 210 MPa higher than that at room temperature. The possible reason is the augmented tensile strength of carbon fiber at 77 K.

2. The CTE of the composite at 77 K was higher than that at room temperature, which changed the fiber/matrix interface stress of the composite. So, in direction parallel to the fiber, the matrix stress was tensile and correspondingly the carbon fiber was compressive, which resulted in the microcracking initiation at a lower load at 77 K. In direction perpendicular to the fiber, the higher thermal expansion of carbon fiber made the gap at the fiber/matrix interface of as-prepared specimens healed up, which led to a higher interfacial sliding resistance and changed the weak fiber/matrix interfacial bonding. Thus, it is helpful for the load transfer from matrix to fiber and the fiber pull-out was optimized.

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