In-situ study of the fracture behavior of SiCf/SiC composite material under three-point bending

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Abstract. The mechanical properties and fracture behavior of a SiCf/SiC composite material were studied by in-situ three-point bending testing. The deformation and cracking behavior of SiCf/SiC composite material was captured by the equipped scanning electron microscope. The experimental results indicate that the fracture property was highly related to the microstructure of the matrix and the SiC fibres. The in-situ observation showed that the composite material exhibited complex crack initiation and propagation process under three-point bending. Vertical microcracks initiated at the root of the preset notch and propagated vertically till blocked by the horizontal fibers. Driven by the large local stress, horizontal cracks were also found initiated adjacent to the notch root and propagated horizontally along the interface between the fibers and the matrix.

1. Introduction

Due to the superior high temperature mechanical properties, low density and low coefficient of thermal expansion, SiCf/SiC composites have been developed and applied in high temperature structure components such as gas turbines [1-3]. Also, the SiCf/SiC composites are regarded as candidate materials for fusion and fission energy applications thank to their excellent irradiation performance and pseudo-ductile fracture mode [4]. Despite of this, the mechanical properties and fracture mechanisms of SiCf/SiC composites need to be further studied to continually enhance their mechanical performances at both room and high temperatures.

Much work has been done on the mechanical properties of the SiCf/SiC composites [4-8]. Yu et al. [4] studied the mechanical behavior of three-dimensional SiCf/SiC composites with alternating PyC/SiC interphases fabricate by a precursor infiltration and pyrolysis (PIP) process. Based on a single-edge notched beam (SENB) test and three-point bending test, they found that PyC/SiC interphases resulted in optimum interfacial adhesion between the fibers and the matrix and enhanced the fracture toughness of the composites. Furthermore, the fracture toughness of the composites with PyC coating in thickness of 0.53 μm rose to 23.1 MPa·m^{1/2}. Li et al. [5] investigated the tensile
behavior of the SiCf/SiC composites containing chemically vapor deposited (CVD) zirconia interphase. Based on the microstructure examination and fracture analysis, the strength of fibers and fracture mode of the composites were highly related to the composition of the zirconia interphase. Mao et al. [6] investigated the cracking resistance of Cf/SiC composites according to the SENB and digital image correlation (DIC) techniques, and the fracture strength and fracture toughness were estimated as 223.23 MPa and 7.81 MPa·m$^{1/2}$, respectively. Apparently the fracture toughness of the fiber enhanced composites was in a large range, which may be closely related to their microstructures, thickness of the interface, interfacial adhesion, etc. For the newly developed Hi-Nicalon type SiCf/SiC composites, knowledge of their microstructures, mechanical performance and fracture modes are limited. In-situ mechanical tests and analyses need to be carried out to further study the strength, fracture toughness, and the cracking behaviors.

The present study focused on the fracture behavior of the SiCf/SiC composite prepared by the PIP method. In-situ three-point bending test was performed to evaluate the fracture toughness and fracture mode. And the deformation and cracking behaviors were analysed by the scanning electron microscope.

2. Materials and testing method

2.1 Materials
Two-dimensional preforms were woven using domestic Hi–Nicalon type SiC fibers. The SiC fibers were provided by Xiamen University (Xiamen, China). The fiber diameter is 14 μm, density is 2.79 g/cm, tensile strength is 2.7 GPa, and the modulus is 270 GPa. The oxygen content is 0.5 wt% and the C/Si mole ratio is 1.41. The properties of the domestic Hi–Nicalon type SiC fiber are close to those of Hi–Nicalon fiber [9-10]. Pyrolytic carbon (PyC) layers with a thickness of about 400 nm were coated on the surface of the preforms by CVD method. SiCf/SiC composites were prepared by PIP process. The preforms were impregnated with liquid state Polycarbosilane (PCS) by a vacuum infiltration method and pyrolyzed at 800 °C in an inert Argon atmosphere. The impregnation and pyrolysis process were repeated 10 times until weight increase was less than 1%. The final porosity of the composites is 6 %–9 %.

2.2 In-situ three-point bending test
To prepare the three-point bending test samples, the SiCf/SiC composites were machined into cuboids following the geometry of 30mm·3 mm·4.6 mm. A notch with a depth of 1.2 mm was prepared in the middle of the sample. All tests were performed at room temperature in the vacuum chamber of an SEM using a specially designed servo-hydraulic testing system, as seen in Figure 1. The bending test was under displacement-control mode with a rate of 0.001 mm/s. The tests were paused at different required strains and held to capture the micro-cracking and microstructure evolution of the samples under the secondary electron mode of the SEM. After testing, each sample was subjected to fractographic analyses in the SEM (TESCAN MIRA 3, Czech).
Figure 1. In-situ three-point bending testing system with an SEM: (a) SEM and control system, and (b) testing sample loading unit

3. Results and discussion

3.1 Microstructure observation

The microstructure of SiC$_f$/SiC composites is shown in Figure 2. Two-dimensional orthogonal fibers were in the matrix. Note that several micro-voids existed in the matrix, which were induced during fabrication process. Figure 3 displays the XRD results of SiC$_f$/SiC composites. The SiC$_f$/SiC mainly consist of SiC face-centered cubic (FCC) crystal structures. And the average grain size can be estimated by the Scherrer law expressed as

$$D = \frac{0.89 \lambda}{B \cos(\theta)}$$

(1)

where $D$ is the average grain size, $\lambda$ is the incident X-ray wavelength ($\lambda = 0.154$ nm, Kα(Cu)), $B$ is the full width at half maximum, and $\theta$ is the diffraction angle. Calculated by Eq. (1), the change in the FWHM value and the average grain size of the (111), (220), (311) plane are 23.2 nm, 44.3 nm and 121 nm (beyond 100 nm), respectively.

Figure 2. The SEM images of SiC$_f$/SiC composites in different views: (a) normal section and (b) cross section
Figure 3. The XRD results of SiCf/SiC composites

3.2 In-situ observation on the cracking behavior

Figure 4 displays the load versus displacement curves of SiCf/SiC composites in three-point bending test. At the beginning, the loading increases linearly with the increase of displacement, and no cracks occur during this stage. When the load reaches the maximum value, the load decreases gradually with the increase of displacement. The load versus displacement curves shows nonlinear characteristics. In this stage, micro cracks in the matrix cracks are generated and grew continually. As the loading continues, the cracks will expand along the direction perpendicular to the fiber and connect with each other. At last, these cracks would merge into a long crack. The extension direction of matrix cracks is along the fiber, which is owing to weak interface binding between the fibers under the effect of stress concentration around the notch root, and the fibers split away off from the interface. Meanwhile, the maximum loading decreased owning to the degradation in the cracking resistance of the fiber and matrix. In the end, the ductile fracture in sample happened.
Figure 4. The load versus displacement curve of SiCf/SiC composites in three-point bending test

Figure 5. In-situ observation of the vertical crack initiation at the notch root under low bending strain

During three-point bending test, the fracture toughness can be calculated based on the following equations
\[ K_{IC} = \frac{P_l \times d_3}{t \times w^2} \times Y \left( \frac{l}{w} \right) \] (2)

\[ Y \left( \frac{l}{w} \right) = \frac{3}{2} \times \left( \frac{l}{w} \right)^{3/2} \times \frac{1.99 - \frac{l}{w} \left( 1 - \frac{l}{w} \right) \left[ 2.15 - 3.93 \left( \frac{l}{w} \right) + 2.7 \left( \frac{l}{w} \right)^2 \right]}{\left( 1 + 2 \frac{l}{w} \left( 1 - \frac{l}{w} \right)^{3/2} \right)} \] (3)

where \( K_{IC} \) is the fracture toughness, \( P_l \) is the bending load, \( t \) and \( w \) are the width and height of the sample, \( d_3 \) is the span between two lower supporting points, \( l \) is the crack length and \( Y \) is the shape function of \( \frac{l}{w} \). Based on the above equations, the fracture toughness was calculated to be about 15.25 MPa m^{1/2}. The magnitude of the fracture toughness is highly related to the microstructures and interfacial and vertical cracking behaviors.

Figure 5 displays the cracking initiation and propagation with low bending strain. As seen in Fig. 5(a), there is no defect crack before loading. As seen in Figure 5(c), when the displacement reached 0.056 mm, a vertical crack is found initiated at the notch root. And then, the crack continues to expand with increasing of the load. After that, instead of widening, the crack even closed. The crack appeared on the notch root expanded at a faster rate, which may be related to the arrangement of fibers. However, as seen in Figure 5(g), the crack did not pass through the fiber during the growth process but deflected along the fiber. It may be on account of moderate interface bond strength between the fiber and matrix. The crack deflection could reduce the stress concentration, making the SiC/SiC composites have stronger bearing capacity and better toughness.

Figure 6 shows the crack evolution with large bending strain. It can be seen that the initial small cracks were produced parallel to the direction of preset notch. However, horizontal cracks produced subsequently propagated perpendicular to the preset notch direction from the starting point of the initial crack, which is quite different from the crack growth mode of non-braided materials. With the increasing of the deflection, the horizontal crack is formed near the notch root and propagated along the direction of the fiber. Especially on the right side of the crack, the crack propagation rate is faster. The propagation speed and length of the horizontal crack are much larger than that of the vertical crack, which indicates that the bonding strength between each layer is much smaller than the strength of the fiber itself, and the crack is more likely to grow along the interface of the fiber matrix.
4. Conclusions
The fracture behavior of SiCf/SiC composite material was studied by in-situ test of three-point bending at room temperature. The fracture property is highly related to the microstructure of the matrix and the fibers. In the load versus displacement curves of SiCf/SiC composite material, the load increased linearly to the maximum and stepped down, which was related to the abrupt decrease of the bearing capacity of the sample due to the cracking between fiber and matrix. The small crack initiated at the notch root and continued to extend with the increasing of the load, and then rapidly closed. The crack was not able to penetrate the fiber due to the high strength of fiber, but deflected along the fiber. The crack deflection reduced the stress concentration at the notch root, making the material possess stronger bearing capacity and better toughness. Horizontal cracks also occurred at the fiber/matrix interface with a high propagation rate. The cracks grew laterally along the interface until the sample broke.

Acknowledgement
This project is supported by the Fund of Key Laboratory of Advanced Functional Composites Technology (Grant No. 6142906190108) and the National Natural Science Foundation of China (Grant No. U2032143, 11902370, 52005523, 51675110), Guangdong Major Project of Basic and Applied Basic Research (2019B030302011), International Sci & Tech Cooperation Program of GuangDong Province (2019A050510022), Key-Area Research and Development Program of GuangDong Province (2019B010943001, 2017B020235001), China Postdoctoral Science Foundation (2019M653173 and 2019TQ0374), Guangdong Education Department Fund (2016KQNCX005), and Fundamental Research Funds for the Central Universities (19lgpy304).

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