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Stressed oxidation behaviors of 2D C/SiC-BC_x composite under wet oxygen atmosphere

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ABSTRACT

To estimate the capability of SiC-BCx protecting carbon fibers under tensile stress, the creep behavior of 2D C/SiC-BCx Ceramic-matrix composites (CMCs) prepared by chemical vapor infiltration (CVI) has been tested and investigated in static wet oxygen atmosphere at 900 °C. Environmental degradation and Micro-mechanics of the composite was discussed according to the Microstructural analysis observed by scanning electron microscopy (SEM) and length change recorded by Instron material testing device. It is found the room temperature residual flexural strength increases with increasing creep stress. The strength improvement is attributed to the glass phase generation and local stress redistribution. The oxidative damage of 2D C/SiC-BC_x composite is more and more serious with the increase of the creep stress. The steady creep speed and accelerated creep speed lay on the oxidation speed of carbon fiber. The oxidation of BC_x layer and carbon fiber is controlled by gas diffusion.

1. Introduction

Carbon fiber-reinforced silicon carbide matrix (C/SiC) composite can be applied in aerospace, nuclear, aeronautic jet engine, industrial turbine and braking system [1,2]. However, the insufficient protection of SiC to load-bearing part (i.e., carbon fiber) is the main limitation for long-term applications in high-temperature oxidizing environments [3,4].

The multilayer self-sealing matrices were developed to improve the oxidation resistance of SiC matrix composites (C/SiC or SiC/SiC). B-C ceramic and Si-B-C ceramic were used to modify SiC matrix by CNRS-SEP and University of Bordeaux-1 in France [5–7]. The oxidation tests showed that the modified self-healing matrix substantially improved the oxidation resistance of SiC matrix composites. Based on multilayer self-healing matrix, the A400, A410, and A500 materials were developed [8], which were tested as seals of F100-PW-229 engine [9]. The application capabilities of A410 and A500 for long-term life have been demonstrated.

Previously, we have reported the fabrication and basic mechanical properties of 2D C/SiC-BC_x composite. The fracture behavior and mechanism, as well as the strength distribution and reliability of 2D C/SiC-BC_x composite, have been discussed [10–12]. The oxidation protection of SiC-BC_x multilayer to carbon fibers in static air atmosphere was much better than SiC [13]. It is known to us that the creep stress is another important factor for the stressed oxidation. The effect of temperature on the stressed oxidation was presented in previous paper [14]. However, the effect of creep stress on stressed oxidation of 2D C/SiC-BC_x composites has not been reported so far.

Therefore, in this work, the effect of creep stress on the residual flexural strength, microstructure evolution, creep behaviors and creep mechanism of 2D C/SiC-BC_x composite in static wet oxygen atmosphere was discussed. The protection capability of SiC-BC_x to carbon fibers under tensile stress in static wet oxygen atmosphere was estimated.

2. Material and methods

2.1. Preparation of specimens

Firstly, the 2D preforms were fabricated from laminated carbon cloth, which was set into fixed configuration by graphite mold

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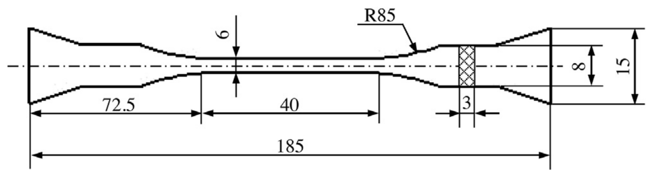

Fig. 1. Shape and sizes of the C/SiC-BC$_x$ composites specimen.

in our laboratory. Secondly, pyrolytic carbon (PyC) interphase was infiltrated into the fiber preforms using C_3H_6 precursor, yielding a thickness of 200 nm. The infiltration conditions for PyC interphase were as follows: temperature 870 °C, time 1 h, and pressure 500 Pa. Thirdly, four layers of SiC ceramic were infiltrated into fiber bundles by low pressure chemical vapor infiltration (LPCVI) method as part of matrix. Subsequently two layers of BC$_x$ ceramic and two layers of SiC ceramic were alternately infiltrated using LPCVI method as another part of matrix. Each layer of SiC ceramic was infiltrated at 1000 °C for 80 h at a reduced pressure of 2 kPa by using methyltrichlorosilane (MTS, CH_3SiCl_3) with a H_2: MTS molar ratio of 10. This process was achieved by bubbling hydrogen gas through the MTS. The argon was used as a dilution to slow down the chemical reaction rate during deposition. The deposition conditions for each BC$_x$ layer were as follows: temperature 950 °C, pressure 1 kPa, time 20 h, Boron trichloride ($BCl_3 \geq 99.99$ vol.% and iron ≤ 10 ppm) flow 50 ml min^{-1}, Methane ($CH_4 \geq 99.95$ vol.%) flow 100 ml min^{-1}, Hydrogen ($H_2 \geq 99.999$ vol.%) flow 500 ml min^{-1}, and Argon (Ar ≥ 99.99 vol.%) flow 500 ml min^{-1}. Then, the as-received composites were machined and polished according to the shape and sizes of sample as shown in Fig. 1 [14]. Finally, the machined specimens were coated with two layers of BC$_x$ ceramic and two layers of SiC ceramic alternately by chemical vapor deposition (CVD). The deposition conditions for the SiC and BC$_x$ coating were the same as the SiC and BC$_x$ matrix except for the deposition time of SiC coating, which was 40 h for each layer.

2.2. *Creep test in static wet oxygen atmosphere*

The test equipment for complex coupling environments of load and static atmosphere is shown in Fig. 2 [14], which can simulate the environments of aero-engine without consideration of gas velocity. Fig. 2(a) is the information control GUI for the controlling of temperature, oxygen content and water vapor content. Fig. 2(b) is the overview of the equipment system, which includes Instron hydraulic universal servo tester (Model 8801, Instron Ltd., UK), an environment box with an electric resistant heater and a subsystem of information control hardware.

The creep tests were conducted using the above equipment at 900 C in wet oxygen atmosphere including H_2O (8 Vol.%), O_2 (12 Vol.%) and Ar (80 Vol.%). The details of test procedure were as

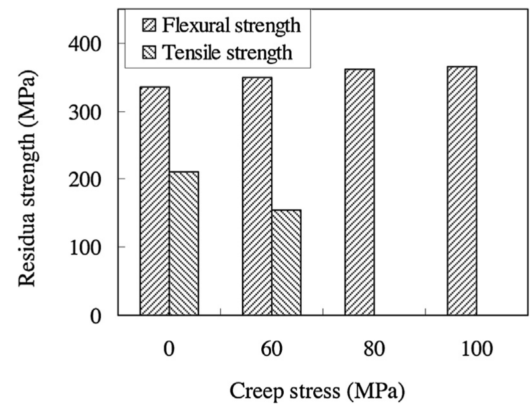

Fig. 3. The normalized residual strength of 2D C/SiC-BC$_x$ composites after creeping under different tensile stress at 900 °C.

follows. Firstly, the sample was fixed in the environment box by the Instron 8801 testing machine at room temperature. Secondly, the environment box was closed and heated up from room temperature to 900 °C with the rate of 10 °C/min under flowing argon gas. Then, the sample was loaded and the water-oxygen gas was introduced into the environment box. The creep stress was 60, 80 or 100 MPa, respectively. During the creep test, the total pressure of the environment box kept 100 kPa, and the water-oxygen gas flow was stable and stable and slow. After the sample was tested for 40 h, the residual three-point-bending strength was tested at room temperature with the loading ratio of 0.5 mm/min and the span width of 30 mm using a universal testing machine (Instron 1196, Instron Ltd., UK) to clarify the effect of matrix oxidation on strength. The residual tensile strength was tested at 900 °C with the loading ratio of 0.2 mm/min as soon as the sample finished the creep testing under 60 MPa for 40 h to estimate the oxidation of carbon fibers.

2.3. *Characterization and measurement of the composites*

The morphologies of 2D C/SiC-BC$_x$ composites after the creep test were observed in detailed with scanning electron microscope (SEM, JSM6700F).

3. Results and discussion

3.1. *Effect of stressed oxidation on residual flexural strength*

Fig. 3 shows the residual tensile strength at 900 °C and three-point flexural strength at room temperature after the 2D C/SiC-BC$_x$ composites crept under different tensile stresses at 900 °C. The residual tensile strength decreased 27.1% after crept under 60 MPa

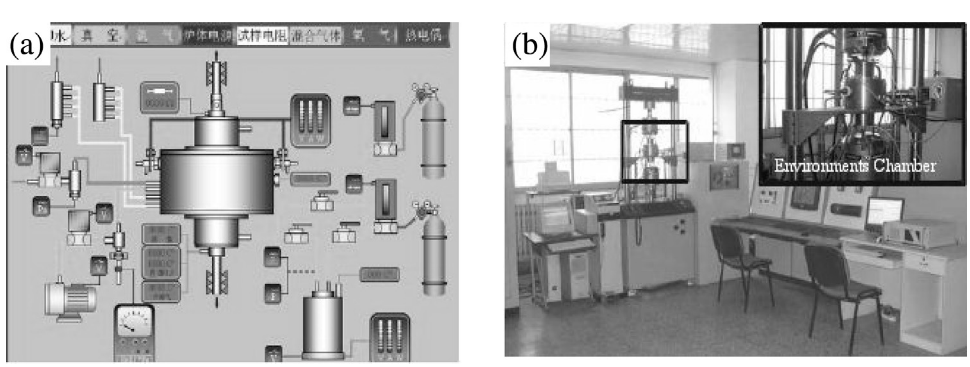

Fig. 2. Equipment for complex coupling environments of load and static atmosphere (a) Information control GUI (b) overview of equipment system.