

Preparation and oxidation behavior of three-dimensional braided carbon fiber coated by SiC

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Abstract

In this paper, SiC coating on three-dimensional (3-D) braided carbon fiber was obtained, which was infiltrated with SiO₂ sol into 3-D braided carbon fiber and then carbon–thermal reduced. The effect of carbon–thermal reduction process was examined. Thermal–oxidative stability of the 3-D braided carbon fiber coated with SiC was compared with original 3-D braided carbon fiber. The oxidation stability of the SiC coating/3-D braided carbon fiber was extended up to 900–1000 °C. After exposure to air at 900–1000 °C, the residual weight of the composite was 26–30%.

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1. Introduction

Commercially available carbon fibers are widely applied as reinforcement in the manufacture of advanced composites because of their excellent properties, such as high specific strength and modulus, low expansion coefficient, and relative flexibility. Carbon fiber-reinforced composites (ceramic, glasses, and polymers) have received much attention in recent years. Their good mechanical, thermal, and chemical properties, especially from the point of view of property/weight and property/cost ratios, make these materials widely applicable in many fields [1].

But carbon fiber exhibits a very poor oxidation resistance even at temperatures as low as 700 K, so the application of the carbon fiber-reinforced composites as high-temperature material has been mostly limited to inert atmospheres in order to prevent the oxidation of the carbon fiber [2]. Once the interfacial bond between fiber and matrix has been debonded by oxidation, a significant reduction in mechan-

ical properties of the composite is unavoidable [3]. For example, flexural strength of phenylethynyl-terminated poly(etherimide) composite decreased by 50% when the oxygen partial pressure was 40.4 kPa and exposure time was 180 h [4]. Therefore, the oxidation behavior of the fiber will affect the durability of CFRCS.

Two approaches have been presented to protect carbon fiber from oxidizing: (1) the use of inhibitors to slow down the oxidation rate of carbon, and (2) the use of barriers to decrease the diffusing rate of oxygen [5–9]. These coatings are made by sol–gel method, chemical vapour deposition, and impregnation of silicone resin. Three-dimensional (3-D) braided carbon fiber is a newly developed textile material, which lays a solid foundation for its application in composites for outstanding mechanical behaviors resulting from its three-directional and four-directional integrated structure. But little has been done on the oxidation protection of 3-D braided carbon fiber because it is not easy to prepare uniform coating on the braided carbon fiber.

Dipping method (solution, sol–gel, and precursor) is simple and inexpensive [10–13]. It will not lead to the degradation of fibers because of the low coating temperature.

In this work, SiC coating on 3-D braided carbon fiber was obtained, through the infiltration of a SiO₂ sol into 3-D

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braided carbon fiber. The oxidation behavior of the composites was examined.

2. Experimental

2.1. Materials

A commercial 3-D braided carbon fiber provided by Lanzhou Carbon Plant (PR China) was used in this work, which was basically a PAN-based carbon fiber. The diameter of the fiber is about 10 μm , and the specific gravity of 3-D braided carbon fiber is 0.2 g cm^{-3} (Fig. 1a).

The SiO_2 coating/3-D braided carbon fiber was fabricated by dip coating [14]. The thickness of the coating is about 0.5–0.6 μm (Fig. 1b).

2.2. Preparation of the SiC coating/3-D braided carbon fiber

The SiC coating was prepared by carbothermic reduction of SiO_2 coating/3-D braided carbon fiber. All synthesis reactions were carried out in a vacuum furnace under an argon flow at 1200–1500 $^{\circ}\text{C}$, held at high temperatures for 1 h, and then allowed to cool naturally.

The crystallinity of the coating was examined by X-ray diffraction (XRD; Damax-2000). Morphology and chemical

composition (through line scan program) were examined by scanning electron microscopy (SEM; JSM-56102Y).

2.3. Oxidation tests

A muffle furnace was used for the isothermal weight loss experiments; static air was employed and the temperature accuracy was estimated as $\pm 10^{\circ}\text{C}$. The tests were carried out at temperatures ranging from 300 to 1200 $^{\circ}\text{C}$, at intervals of 50 $^{\circ}\text{C}$, for 30 min. The weight change was noted with an electronic balance (sensitivity: $\pm 0.1 \text{ mg}$).

The USA-TA5000 TG thermal analyzer (TA Instruments, USA) was employed for the study of oxidation kinetics and mechanism of fiber (thermobalance with a sensitivity of $\pm 0.1 \text{ mg}$, at temperatures ranging from room temperature to 1000 $^{\circ}\text{C}$ under static air, at a rate of $10^{\circ}\text{C min}^{-1}$).

3. Results and discussion

3.1. Formation of SiC coating

Fig. 2 shows the XRD spectrum of materials prepared by carbothermal reduction. Three different phases, namely, SiC, SiO_2 , and C, are identified. From samples sintered at 1300 $^{\circ}\text{C}$, the main peak intensity of SiC is weaker compared with that sintered at 1550 $^{\circ}\text{C}$. Fig. 3 shows the SEM photograph of the single fiber coated with SiC and the line scan spectrum of the fiber, which is sintered at 1550 $^{\circ}\text{C}$. From these figures, we can see that there is pure SiC phase at the edge of fiber. From outside to inside, the content of SiC decreased, the coat served as gradient, and, contrarily, the content of C increased from the edge to the center. SiC coating was coated symmetrically and combined well with fiber; there is no crack between the coating and the fiber.

3.2. Thermal analysis

3.2.1. The isothermal oxidation properties

The isothermal oxidation–weight loss and Arrhenius curves of fiber are shown in Fig. 4. From the oxidation weight–loss curves (Fig. 4A), it is obvious that the oxidation process of fiber began from 600 $^{\circ}\text{C}$; the weight loss reached a maximum at about 1000 $^{\circ}\text{C}$; at that temperature, the weight loss was about 67–72%. After that, with the temperature increasing, the weight increased, which maybe caused by the oxidation of the coating.

For the carbon materials, the oxidation weight loss was proportional to the time when weight loss was below 70% [15] (Eq. (1)):

$$\frac{m_0 - m}{m_0} = kt \quad (1)$$

where m_0 is the initial mass of the sample, m is that of at time t , and k is a reaction rate constant at a fixed temperature.

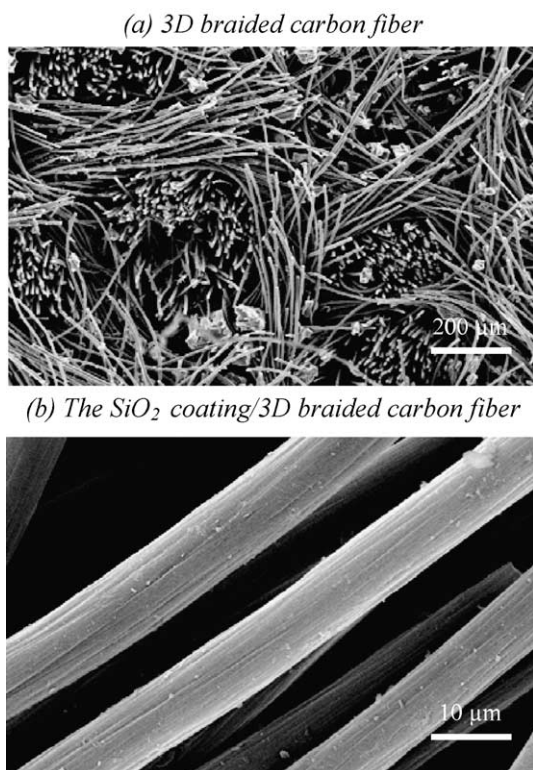


Fig. 1. SEM photographs of experimental materials: (a) 1300 $^{\circ}\text{C}$, (b) 1550 $^{\circ}\text{C}$.

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