



Preparation and oxidation behavior of a double-layer coating for three-dimensional braided carbon fiber



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ARTICLE INFO

Article history:

Received 16 November 2015

Revised 19 January 2016

Accepted in revised form 30 January 2016

Available online 22 April 2016

Keywords:

Carbon fiber

Coatings

Microstructure

Oxidation

ABSTRACT

Double-layer coatings, with Pyrolytic carbon (PyC) as inner layer and SiC/SiO₂ as outer layer by a precursor infiltration and pyrolysis (PIP) process, were prepared on the surface of three-dimensional (3-D) braided carbon fiber. The double-layer coatings (PyC-SiC/SiO₂) were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS). The microstructure, tensile strength and the oxidation resistant properties of the carbon fiber with and without coating were studied. Result showed that the best coating quality obtained was use of 10 wt% polycarbosilane (PCS) with 5 wt% methyltriethoxysilane (MTES) precursor in divinylbenzene (DVB) pyrolyzed to 1400 °C for 2 immersion–pyrolysis cycles. The oxidation resistance of carbon fibers was improved obviously by the PyC-SiC/SiO₂ coatings. Tensile strength measurement revealed that the PyC-SiC/SiO₂ coated carbon fiber maintained 87.7% of its original strength.

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1. Introduction

Carbon fibers are the most significant reinforcement applied in advanced composites owing to their high specific strength, low expansion coefficient and relative flexibility [1–2]. However, carbon fiber displays a very poor oxidation resistance even at low temperatures. The application of coating is one of the most efficient surface modification approaches to enhancing the oxidation resistance of carbon fiber. PyC inner layer is commonly employed to protect carbon fibers in the deposition of SiC/SiO₂ coating [3–4]. SiC is widely applied as one of the most common coating ceramics because of its excellent mechanical properties at high temperature, high melting point and relatively good oxidation resistance up to 1500 °C in oxygen-rich atmosphere [5]. In addition, SiC is oxidized to SiO₂ in a dry oxygen atmosphere which may fill the cracks of the layer and increase the diffusion barrier efficiency [6–7].

There are several methods to prepare ceramic coating, such as chemical vapor deposition (CVD) [8–11], precursor infiltration and pyrolysis (PIP) [12–14] and sol-gel method [15–17]. Among these processes, the PIP route is being actively developed to prepare coating layers for its advantages of low processing temperature and controllable ceramic compositions [18]. Few papers were published about the PyC-SiC/SiO₂ coatings on the surface of 3-D braided carbon fiber and most of the papers concentrated on preparation of 1-D or 2-D carbon fiber. Three-dimensional (3-D) braided carbon

fiber is a newly developed textile material. It lays a foundation for its application in composites for prominent mechanical behaviors resulting from its three-dimensional structure. It has been seldom studied on the oxidation protection of 3-D braided carbon fiber because it is not easy to prepare the uniform coating on 3-D braided carbon fiber [19].

In this paper, a new double-layer, PyC as the lower layer and SiC/SiO₂ as the upper layer, on the surface of 3-D braided carbon fiber was developed. The microstructure and the oxidation resistant properties for the carbon fiber with and without coating in static air were investigated, and the tensile strength of the samples was also studied.

2. Experimental

2.1. Raw materials

The 3-D carbon fibers (Jiangsu Tianniao High Technology Co. Ltd., Jiangsu, China) used in this study were all polyacrylonitrile (PAN)-based fibers. The fiber volume density is about 0.31 g/cm³. Phenol-formaldehyde resin (Xinxiang Burmah Sail Industrial Co., Ltd., Xinxiang, China) and acetone (Sinopharm Chemical Reagent Co., Ltd., Shanghai, China) were used as precursor to form the PyC coating. PCS (Suzhou Sailifei Ceramic fiber Co. Ltd., Jiangsu, China), MTES (Qingdao Jacob Chemical Reagent Sales Co., Ltd., Qingdao, China) and DVB (Jintong Letai Chemical Product Co., Ltd., Beijing, China) were used as ceramic precursor to prepare the SiC/SiO₂ coating.

2.2. Preparation of the PyC-SiC/SiO₂ coatings

In order to remove the epoxy resin glue on the surface of fibers, the fiber was treated at 400 °C for 1 h, in a tube furnace under flowing

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nitrogen. Then the PAN fiber was placed in a beaker containing activated by HNO_3 for 2 h. PyC precursor was prepared by using phenol-formaldehyde resin and acetone with a mass ratio of 1:19. The 3-D fabrics were impregnated to the precursor solution. Thereafter, the samples were cured at 120 °C for 1 h and then 180 °C for another 2 h in the oven. With a thermal treatment of 750 °C for 1 h, PyC layer were prepared. Subsequently, the coated preforms were dipped into a precursor solution containing PCS (10 wt%), MTES (5–15 wt%) and DVB (75–85 wt%) in vacuum. The samples were cured at 120 °C for 1.5 h and then at 150 °C for another 1.5 h, and were then pyrolyzed to 1200 °C and to 1400 °C under a nitrogen flow to form the SiC/SiO₂ coating. The heating rate was about 5 °C/min to the maximum temperature and hold time 1 h.

2.3. Characterization

The crystalline phase of the coated carbon fiber was evaluated by X-ray diffraction (XRD, X' Pert PRO, Holland PANalytical Company) with Cu K α radiation in a 2θ range of 5–90°. The surface morphologies of carbon fibers were investigated using a scanning electron microscopy (SEM, S-3400N, Japan Hitachi Company) equipped with an energy dispersive spectroscopy (EDS) detector. The oxidation resistant properties of carbon fibers with and without the coatings were also researched by isothermal oxidation. An electronic balance was used to note the weight change. The thickness of the coating was examined with SEM by measuring the diameter of the fiber before and after coating. Multiple measurements of the coating thickness have been conducted. Tensile strength of the carbon fibers with and without coating was measured by Instron 3345. The gauge length was 25 mm, and the loading speed was 0.3 mm/min. At least 20 fibers were mechanically tested for each treatment conditions for a single data point.

3. Results and discussion

Fig. 1 shows XRD patterns of the carbon fibers with the PyC-SiC/SiO₂ coatings prepared by ceramic precursor of 10 wt% PCS, 5 wt% MTES and 85 wt% DVB. Three different phases of SiO₂, SiC and C were identified. SiO₂ phase was mainly found in an amorphous structure at around $2\theta = 22.72^\circ$. Three peaks at $2\theta = 35.63^\circ$, 60.02° and 71.72° corresponded respectively to (111), (220) and (311) were observed for the SiC phase. The bands of diffraction at $2\theta = 26.38^\circ$ and 42.22° were generally found for the C. For samples heat treated at 1400 °C, the main peak intensity of SiC is stronger and the number of peaks is

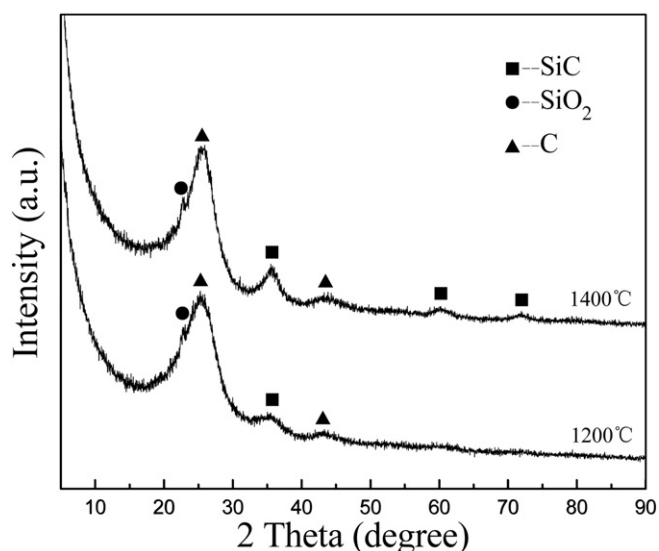


Fig. 1. XRD patterns of the PyC-SiC/SiO₂ coated carbon fibers.

more compared with that heat treated at 1200 °C. This shift was attributed to the carbothermal reduction reaction conveyed as follows [20]:

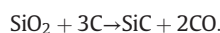


Fig. 2 shows the surface images of carbon fibers before and after being coated. The initial carbon fiber has a well-developed relief and the fibrils are oriented parallel to the fiber axis (Fig. 2(a)). PyC coating prepared by carbothermal reduction is shown in Fig. 2(b). It can be seen that the PyC coating prepared on carbon fibers was smooth and continuous. And the coating was combined well with fiber. Furthermore, no cracks were seen on the coating after preparation. The thickness of PyC coating was about 150–200 nm, which was uniformly coated on the carbon fiber. In Fig. 2(c), the coating appeared obviously cracking, spalling and caking. At 1200 °C, the main phase of the coating is amorphous SiCO glass, and the thermal expansion coefficient between carbon fiber and the coating dose not match, leading to unstable and cracking coating. At 1000–1200 °C, the phase of SiCO was separated into SiC, SiO₂ and C [21–22]. The reaction was expressed as follows:



When the samples sintered at 1400 °C, the coating was homogeneous and there was no crack (Fig. 2(d)). In this study, the good morphology of the coating obtained was use of heat treatment temperature at 1400 °C.

SEM images of the PyC-SiC/SiO₂ coated carbon fibers heat treated at 1400 °C are displayed in Fig. 3. Fig. 3(a) to (c), corresponding from one to three immersion-pyrolysis cycles, show the coatings prepared by using ceramic precursor consisting of 10 wt% PCS, 5 wt% MTES and 85 wt% DVB, while Fig. 3(d) for 10 wt% PCS, 10 wt% MTES and 80 wt% DVB by one immersion-pyrolysis cycle, and Fig. 3(e) for 10 wt% PCS, 15 wt% MTES and 75 wt% DVB by one immersion-pyrolysis cycle. In Fig. 3(a), the coating was relatively homogeneous and continuous on the fiber surface, but it was too thin and only very limited fiber surface was covered. Fig. 3(b) shows relatively dense, smooth, and uniform coatings. While the coating coated three times was so thick that prominent lumping, cracking and spalling occurred (Fig. 3(c)). Similar phenomena were observed to Fig. 3(d), but more serious in bridging and lumping. The carbon fiber periphery followed a rough terrain when MTES addition up to 15 wt% (Fig. 3(e)). In this study, the good morphology of the coating obtained was use of the 10 wt% PCS, 5 wt% MTES and 85 wt% DVB for 2 immersion-pyrolysis cycles. The C, O and Si elements peaks (Fig. 4), consistent with the result of XRD analysis, proved that the PyC-SiC/SiO₂ coatings exist on the surface of carbon fiber.

The magnified SEM images of the double-layer coating on the surface of carbon fibers were shown in Fig. 5. Obviously, a continuous coating was synthesized on the PyC coated carbon fibers (Fig. 5(a)). While the coating coated three times was so thick that prominent lumping, cracking and spalling occurred (Fig. 5(b)). Fig. 5(a) also shows that the coating on carbon fibers was uniform. Associated with the result of the XRD analysis, it can be deduced that, with rising immersion-pyrolysis cycles, the thickness of SiC/SiO₂ coating increased and that of the PyC coating decreased. This shift was mainly attributed to the carbothermal reduction reaction conveyed as follows: $\text{SiO}_2 + 3\text{C} \rightarrow \text{SiC} + 2\text{CO}$. The thickness of the SiC/SiO₂ coating had a close relationship with both the immersion-pyrolysis cycles and MTES concentration. The thickness of the outer coating was varied from 50 nm to 500 nm.

The influence of heat treatment temperature on the oxidation resistance of the PyC-SiC/SiO₂ coated carbon fibers (concentration of 5 wt% MTES) is given in Fig. 6. Compared to the uncoated carbon fibers or the coated carbon fibers with 1200 °C heat treatment temperature, the slope of the curve was quite more gently at an oxidation temperature of 700 °C for the coated carbon fibers heat treated at 1400 °C. From Fig. 6 we can also see that the coated carbon fibers heat treated at 1200 °C have lost 89.2% of its original weight after oxidized for 90 min in air while the uncoated carbon fibers were burnt out. The coated

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