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Microstructure of C/C composites prepared by chemical vapor infiltration method with vaporized kerosene as a precursor

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Abstract

The microstructures of two types of C/C composites prepared from different carbon felts by a rapid densification method, thermal gradient chemical vapor infiltration with vaporized kerosene as a precursor, at 1080–1120 °C for 6 h were characterized by polarized light microscopy (PLM), scanning electron microscopy (SEM), X-ray diffraction (XRD) and Raman micro-spectrometry techniques. The experimental results show that the fibers in the two composites are both surrounded by ring-shaped pyrocarbons with rough laminar texture, but the thickness, the surface morphology of the pyrocarbons and the graphitizability of the composites depend much on the configurations of carbon felts. The C/C composite fabricated from a higher porosity carbon felt possesses larger thickness and rougher surface of pyrocarbon, and has a lower graphitizability after heat treatment at 2300 °C for 2 h.

Keywords: Carbon/carbon composite; Microstructure; Pyrolytic carbon; Raman micro-spectrometry

1. Introduction

The application of carbon/carbon (C/C) composites, mainly in aerospace industry as protection materials for atmosphere re-entry and disc brakes of aircraft, requires a basic knowledge of their structural characteristics. The characteristics, including macroporosity, pyrocarbon texture, fiber orientations, microcrack and interfaces at different levels, etc. control the mechanical, thermal and frictional properties of C/C composites [1]. Therefore, during the past decades most attentions were paid on the microstructural investigations to obtain C/C composites with desired properties for many applications [2–6].

Since chemical vapor infiltration (CVI) is commonly utilized for C/C composites preparation [7], a correlation between the microstructures and the fabrication parameters has been intensively studied [8–10] and comprehensively reviewed by Oberlin [12] and Delhaes [11]. In general, the microstructures depend on the reinforcement configuration, the fabrication routes and processing conditions, as well as the used precursor and the surface property of carbon fiber [1,11,13–14], but a clear explanation for these relationships is still unavailable. On the other hand, new CVI techniques and the using of complex precursor have been developed to speed up fabrication process. Naturally, microstructural information of the fabricated C/C composites is also continuously investigated to give instructive and significant feedback on the fabrication process.

Recently, we designed a new method, which combines the advantages of regular film boiling CVI and classical thermal gradient CVI, to realize a rapid deposition of C/C composites [15]. In this method, two (upper and lower) heat-sources are utilized to vaporize liquid precursor and to deposit pyrocarbons on preforms, respectively, in a cold wall reactor where the liquid precursor and the preform are separated. The reasons for rapid deposition have been attributed to the using of mixture of hydrocarbons as a precursor, the thermal gradient existing across the preform, and the short convection and diffusion paths of hydrocarbon from the liquid precursor to the depositing zone.

Although principle analyses of this rapid densification method has been preformed by our previous work, further investigation is necessary and significative to get a better knowledge about the influences of the fabrication route and felt configurations on the microstructures of the composites. For this purpose, two types of C/C composites were prepared from different carbon felts in the present paper. The microstructures of the composites were studied by polarized light microscopy (PLM),

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Table 1
The parameter of the carbon felt used as perform

Type of carbon felt	Single fiber diameter (μm)	Felt thickness (mm)	Bulk density (g/cm^3)	Porosity (%)
A	∼10	10	∼0.15	∼90
B	∼6	10	∼0.52	∼70

Table 2
The density, average density increase rate and porosity of the C/C composites

Type of C/C	Density (g/cm^3)	Density increase rate (g/(cm^3 h))	Porosity (%)
A	1.68	0.255	19
B	1.71	0.196	13

scanning electron microscope (SEM), X-ray diffraction (XRD) and Raman micro-spectrometry techniques.

2. Experimental procedure

2.1. Material preparation

Two types of carbon felts were used as preforms. Type A carbon felt obtained from Lanzhou carbon fiber plant, China, is made of short-cut carbon fibers in random directions. Type B carbon felt is a needle punched felt made of layered non-woven cloth in which carbon fiber is made in Jilin carbon plant, China. The parameters of the two felts are summarized in Table 1, where the porosities are calculated based on 1.76 g/cm^3 of fiber density. A liquid hydrocarbon mixture, kerosene (molecular formula: C_9H_n–$C_{16}H_m$) with a boiling temperature range of 180–260 °C, was chosen as a precursor.

The experimental device for preparing C/C composite is schematically shown in Fig. 1. During the processing, two susceptors are inductively heated by the inductor around the reactor. The lower susceptor is used to boil and vaporize the liquid kerosene which provides gaseous hydrocarbon for deposition. The upper susceptor is used to heat the surface of the preform. A thermocouple is located at the upper surface of the preform to measure the densification temperature. The densification of the preform was performed at 1080–1120 °C for 6 h. During the densification, the upper surface of the preform is at a higher temperature (>1000 °C) than that of the lower surface (∼170 °C as measured in our experimental conditions). Therefore, an average temperature gradient (>80 °C/mm) is formed in the thickness direction of the preform.

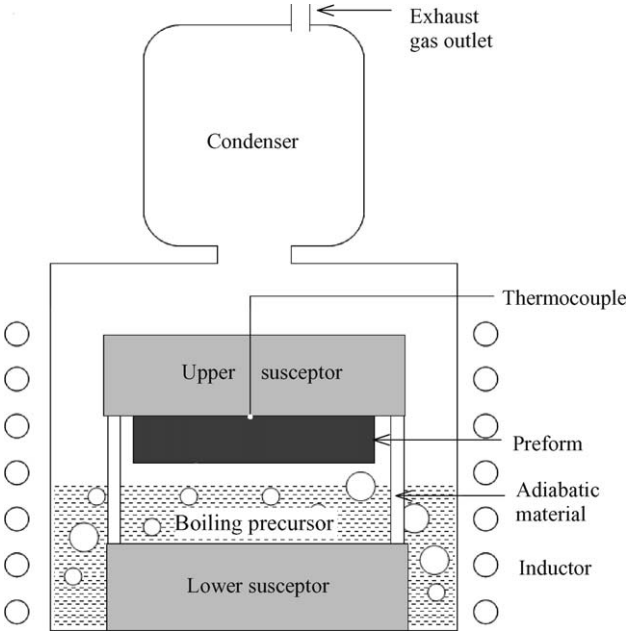

Fig. 1. Sketch of experimental device for preparing C/C composites.

2.2. Characterization

The densities and open porosities of the prepared C/C composites were measured by Archimedes principle for which distilled water was used; the average density increase rate was calculated. These results are given in Table 2.

Microscopy observations on the polished surfaces and fracture surfaces of the C/C composites were carried out by polarized light microscope (PLM, Reichert, MeF3) and scanning electron microscope (SEM, Hitachi, S-2700) operated at 25 kV and 20 mA, respectively.

Some composite materials were heat treated at 2300 °C for 2 h under argon atmosphere on the purpose of investigating their graphitization degree. The crystalline structures of these composites were examined via X-ray diffraction (XRD, D/MAX-RA X-ray diffractometer) between 10 and 80° (2θ) to determine d-spacing and crystallite size L_c. According to Bragg's law, the d-spacing of the carbon (0 0 2) plane was determined using the following equation:

$$d = \frac{\lambda}{2\sin\theta} \quad (1)$$

where λ is the wavelength of Cu Kα radiation (0.1541 nm) and θ the diffraction angle in radians. Crystallite size L_c was obtained from the Scherrer equation:

$$L_c = \frac{0.9\lambda}{B\cos\theta} \quad (2)$$

where B is the half maximum intensity in radians of the (0 0 2) peak.

The polished surfaces of the heat-treated C/C composites were analyzed using Raman micro-spectroscopy (Jasco NRS-3100). A He–Ne laser was used with a fixed wavelength of 632.8 nm and the delivering power is around 1 mW on ∼2 μm^2.

3. Results and discussion

3.1. Morphology and texture of the C/C composites

The polarized light micrographs of the C/C composites are shown in Fig. 2. At a low magnification observation of type A composite (Fig. 2a), it is clearly observed that the cross