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Electrophoretic deposition of carbon nanotubes onto carbon fiber felt for production of carbon/carbon composites with improved mechanical and thermal properties

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ABSTRACT

Carbon nanotubes (CNTs) were introduced into carbon fiber felts by electrophoretic deposition (EPD) and then pyrocarbon (PyC) was infiltrated in the felts by thermal gradient chemical vapor infiltration using methane as precursor to prepare carbon nanotube (CNT) reinforced carbon/carbon composites (CNT-C/C composites). The effects of CNTs by EPD on microstructure, mechanical and thermal properties of carbon/carbon (C/C) composites were investigated. Results showed that CNTs had complicated effects on the microstructure of PyC in CNT-C/C composites: PyC next to carbon fiber (CF) was isotropic texture and PyC far from CF showed medium texture, while PyC in C/C composites without CNTs (REF-C/C composites) presented low texture. Compared with REF-C/C composites, compressive strength and modulus of CNT-C/C composites were increased by 37% and 19%, respectively, and fracture mode of the composites changed from pseudo-plastic fracture to brittle fracture; coefficient of thermal expansion of CNT-C/C composites in the direction perpendicular to carbon fiber plies was decreased by 30%.

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1. Introduction

Carbon/carbon (C/C) composites exhibit many excellent properties such as low density, high specific strength and modulus, high thermal conductivity, low thermal expansion and good shock resistance, which make them important candidates in aerospace applications [1–3]. However, further applications are limited because of the poor fiber/matrix (F/M) interfacial bonding and the low cohesion of pyrocarbon (PyC) matrix.

Carbon nanotubes (CNTs), with unique rolled graphitic layers and fascinating mechanical properties [4,5], can be used as secondary reinforcement of fiber/matrix composites to obtain better properties. Many works about CNT reinforced C/C composites (CNT-C/C composites) have been done [6–10] in the past few years. CNT-C/C composites were generally obtained by using in-situ growth method via catalytical chemical vapor deposition (CCVD). Although this method can obtain C/C composites with some improved performances such as excellent out-of-plane compressive property and interlaminar shearing strength [8], the preparation of CNT-doped preform by CCVD shows disadvantages of high cost and process complexity. Furthermore, due to suffering from tough chemical treatments and the metal catalyst particle etching during CCVD, carbon fibers (CFs) always have a seriously degraded mechanical property [11,12]. Electrophoretic deposition (EPD), as a low-cost and simple method with mild operating conditions, can avoid the above drawback of in-situ grown CNTs method according to the previous reports [13,14]. In addition, EPD also offers advantages of uniformity of deposits, control of deposit thickness, microstructural homogeneity, and the potential to infiltrate porous substrates [15–17]. An [13] et al. prepared the hierarchical-composite reinforced with CNTs introduced into carbon fiber fabric by EPD, indicating significant increases in shear strength and fracture energy. Song [14] et al. also reported that the tensile performance of carbon nanotube-carbon fiber hybrid reinforcements prepared by EPD showed great improvement in the tensile tests of CF-based reinforcements, and there is a higher efficiency of EPD in preparing CNT-doped preform compared with in-situ growth method.

In this work, CNT-C/C composites were prepared by the densification of two dimensional (2D) needle-punched CF felts doped with CNTs by EPD via thermal gradient chemical vapor infiltration (TCVI). Influences of EPD CNTs on microstructure and mechanical properties of C/C composites were studied. Furthermore, effect of EPD CNTs on coefficient of thermal expansion (CTE) in C/C composites, which is of great importance in the practical application, was also studied.

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2. Experimental

2.1. Preparation of samples

2D needle-punched CF felts (T300 CFs, 6–7 μm, Jiangsu Tianniao High Technology Co., Ltd., China) with a density of 0.2 g/cm^3 and multi-walled CNTs without functional treatment (97% purity, Timesnano, China) were used as the starting materials. The outer diameter of the multi-walled CNTs ranges from 20 to 40 nm and the length of multi-walled CNTs are about 30 μm. For EPD, CNTs were dispersed in isopropyl alcohol by 3 h ultrasonic to obtain dispersions of 1.6 g/L. The felt connected with a copper wire as a cathode was fixed in a plastic frame, and then was immersed in an EPD cell containing stable CNTs suspension. A stainless steel plate was placed on the opposite side of the felt as an anode. EPD experiment with a PowerPac Basic (Bio-Rad Laboratories, Inc., USA) was performed at a constant voltage of 40 V and an electrode distance of 25 mm. Deposition time was 60 s, and the felt with 1.05 wt% CNTs would be obtained. Subsequently, it was densified for 60 h under ambient pressure at 1000 °C with methane as carbon source by TCVI to prepare CNT-C/C composites. And flow rate of natural gas was controlled at 0.8 m^3/h. For comparison, C/C composites without CNTs (REF-C/C composites) were also prepared under the same conditions. Finally, both composites were heat treated at 2500 °C for 2 h under Ar atmosphere with a ZGS-400 high-temperature graphitized furnace (Jinzhou Sante Vacuum Metallurgical Technology Industrial Co., Ltd., China).

2.2. Morphology and structure characterization

Textures of pyrocarbon (PyC) in the composites were studied with polarized light microscopy (PLM, Leica DMLP optical microscopy) at polished cross-sections. Raman spectroscopy (Renishaw in VIA, with 514.5 nm excitation) with an objectives of 50× magnification and an eyepiece of 10× magnification was used to measure microcrystal order of PyC. Diameter of the laser spot on the sample surface was 1 μm. Morphology of PyC and fracture surfaces of the composites were also observed using Quanta-600FEG field emission scanning electron microscopy (SEM). High-resolution transmission electron microscopy (HRTEM, Tecnai F30G^2) was used to observe microstructure and crystallinity of CNTs.

2.3. Mechanical properties tests

Compressive tests were performed on a SANS universal testing machine (CMT5304-30 kN). Size of samples was 5 mm × 5 mm × 5 mm and the loading direction was perpendicular to carbon fiber plies (*Z* direction). The number of effective samples for each case was no less than four. The tests were carried out with a constant speed of 0.5 mm/min at room temperature. Compressive strength was calculated according to the following equation:

$$\sigma_c = \frac{p}{bh} \tag{1}$$

where σ_c is the compressive strength (MPa); p is the maximum loading of fracture (N); b is the width of the sample (mm) and h is the thickness of the sample (mm).

2.4. Measurement of CTE

A Netzsch DIL 402C dilatometer was used for CTE measurement. The required samples were machined into rectangular bars of 15 mm × 4 mm × 4 mm. Measurements were conducted in a helium atmosphere from room temperature to 1500 °C with a heating rate of 5 °C per min. Tests were limited in *Z* direction and CTE was calculated by the following equation:

$$\alpha = \frac{\Delta L}{L(T_0)\Delta T} \tag{2}$$

where α is the CTE of the sample, $L(T_0)$ is the length of the sample at the room temperature (25 °C), $\Delta L = L(T) - L(T_0)$ and $\Delta T = T - T_0$ are changes of the length and the corresponding temperature, respectively.

3. Results and discussion

3.1. Microstructure and morphology of CNTs

Fig. 1(a) shows TEM image of CNTs used in this study. It can be confirmed that CNTs possess a multilayered structure and graphene sheets are almost parallel to the axial direction (shown by dotted line in Fig. 1(a) inset), revealing a high crystallinity intervening between ideally crystalline CNTs and relatively low crystalline carbon nanofibers [18]. Raman spectrum of CNTs (Fig. 1(b)) shows two first-order bands at around 1350 (D band) and 1580 cm^{-1} (G band). It has been demonstrated that the D band corresponds to the presence of disordered graphitic lattices and the G band corresponds to the ideal graphitic lattices [19]. Low ratio R of I_D to I_G (I_D and I_G mean the intensity of D band and G band, respectively) shows a high crystallinity of CNTs.

Fig. 2 shows SEM images of CNTs introduced into the felts by EPD. CNTs deposited uniformly on CFs are no obvious agglomeration or acutely curly body. The directions of all CNTs are in the plane parallel to the CF axis, showing a great randomness and forming a three dimensional (3D) network structure on CFs (Fig. 2). Besides, EPD CNTs are directly deposited onto CFs with the help of electric field force, rather than grafted on CFs as the case of CCVD, which avoids the damage of CFs surface structure [11,12].

3.2. Microstructure of the composites

PyC is generally classified into four categories, namely isotropic (ISO) PyC, low-textured PyC, medium-textured and high-textured PyC [20]. The polished transverse sections of the composites viewed by PLM are shown in Fig. 3. For REF-C/C composites (Fig. 3(a)), PyC presents low optical activity, unclear extinction cross and few growing cones, which is low texture. Besides, the F/M interface is clear and boundaries of PyC grown around CFs are extremely evident. Many homocentric annular cracks (shown by dotted arrows in Fig. 3(a)) are also observed. However, after introducing CNTs, PyCs at different positions in CNT-C/C composites demonstrate various textures because the whole matrix exhibits inconsistent optical activity. PyC next to CF shows no optical activity, which can be classified as ISO texture. PyC far from CF has a higher optical activity than that of REF-C/C composites and there are some distinct growing cones. Hence, it can be called medium-textured PyC. In addition, the F/M interface presents a compound and transitional state. Much smaller sizes of PyC and fewer homocentric annular cracks (Fig. 3(b)) can be observed.

Fig. 4 shows the cross-section morphology of fracture surfaces of the composites. For REF-C/C composites PyC possesses relatively small growth curvature, and crack in the F/M interface is distinct as shown in Fig. 4(a). The loosely packed wrinkled flakes of carbon layers present in the inset of Fig. 4(a), which can be classified as low-textured PyC. However, PyCs in the CNT-C/C composites shows different morphology at different positions. On one hand, PyC next to CF exhibits littery texture in Fig. 4(b). The carbon layers grown

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