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Mechanical properties enhancement of carbon/carbon composites by in situ grown carbon nanofibers

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ABSTRACT

Carbon/carbon (C/C) composites with in situ grown carbon nanofibers (CNFs) were fabricated. Results showed that CNFs had significant benefit on improving mechanical properties of C/C composites. It was concluded that CNFs could be involved in serving as a second reinforcement for composites and absorbed more energy leading to higher mechanical properties.

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1. Introduction

Carbon/carbon (C/C) composites have superior characteristics, such as low density, high strength, high thermal conductivity and low thermal expansion coefficient together with good frictional performance, so they are widely applied in different fields [1,2].

Nevertheless, low toughness and brittle fracture behaviors of C/C composites limit their further usage as structural materials. To improve the toughness of composites, much effort has been devoted on this [3,4]. The fillers such as expanded graphite, vapor-grown spiral carbon fiber and carbon black were used as secondary reinforcement in the carbon matrix for fabrication of pitch based C/C composites [5,6]. Lu found a method to improve toughness of C/C composites by intercalating bromine into graphite microcrystal of pyrocarbon [7]. After intercalation, a tough-like fracture mode was obtained and the flexural modulus and deflection was increased by 10% and 18%, respectively. As the mechanical properties of composites were mainly controlled by microstructures, the proper modification of structures and nano-texture of matrix may improve the toughness of composites. Carbon nanotubes/nanofibers were used as reinforcement to improve the toughness and strength of composites [8-10]. Nevertheless, the nanometer level materials were hardly to be dispersed uniformly among composites due to their large specific area and their role

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of enhancement in mechanical properties could not be completely played. So in this work, in order to overcome the problem, CNFs were introduced into C/C composites by in situ chemical vapor infiltration (CVI) process and higher mechanical properties and better toughness of composites were obtained.

2. Experimental

2.1. Fabrication of carbon nanofibers (CNFs) in carbon felts and C/C composites

2-Dimensional (2D) carbon felts with density of 0.4 g/cm³ were used as preform which were fabricated by repeatedly overlapping the layers of 0° weftless ply, short-cut fiber web and 90° weftless ply with needle-punching step-by-step. First, the felts were impregnated in solution of ferrous nitrate with concentration of 10%. Then the felts were dried in air. After the weight gain reached 1% with cycles of impregnation and drying, the preform was reduced under hydrogen atmosphere at 673 K in an electric resistance furnace. CNFs were grown in felts at 1323 K with natural gas as carbon source and nitrogen as diluted gas for 2 h. Mesophase pitch (Mitsubishi Gas Chemical, Japan) was supplied as precursor for fabrication of matrix of C/C composites by cycles of impregnation/carbonization at atmospheric pressure. The details of impregnation/carbonization could be seen in the former report [11]. For comparison, C/C composites without CNFs were fabricated by six cycles of impregnation/carbonization under the same condition. The C/C composites grown with CNFs were marked as CC-CNFs and C/C composites without CNFs were marked as CC-no.

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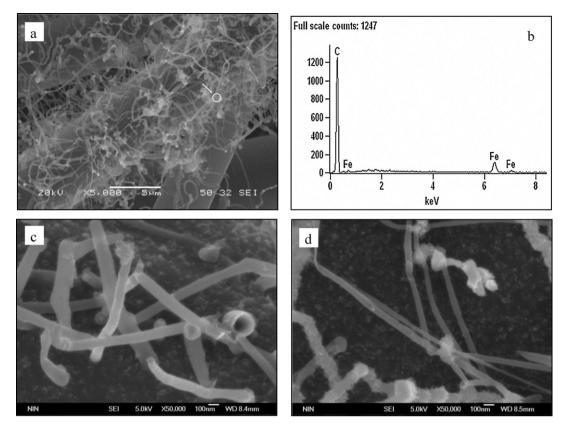


Fig. 1. The SEM of carbon nanofibers grown in the carbon felts (a: SEM under low magnification; b: EDS of the spot arrowed in a; c and d: SEM under high magnification).

2.2. Flexural test and microscope morphology

To study the effect of in situ grown CNFs on mechanical properties of C/C composites, three-point bending test was performed on CMT5304-30KN universal testing machine, conducted at loading speed of $0.5 \, \text{mm/min}$ and support span of $40 \, \text{mm}$ length. The size of bending specimens was $55 \, \text{mm} \times 10 \, \text{mm} \times 4 \, \text{mm}$. The number of specimens used in the flexural test was no less than five for every test. The morphology of grown CNFs and fracture surface of C/C composites were examined by JSM-6460 scanning electron microscope (SEM). Energy dispersive spectroscopy (EDS) was used to detect the component of nanofibers.

3. Results and discussion

Fig. 1 shows SEM images and EDS of the CNFs grown in 2D carbon felts. The CNFs with different length in the range of 10–40 μm are dispersed among carbon felts without being coated on the surface of carbon fibers. Fig. 1c and d displays that the diameter of CNFs is 50–100 nm. The CNFs are grown curvedly and a few of them are twined together among carbon fibers. Several carbon nanotubes also can be observed (Fig. 1c). EDS analysis of the selected area (arrowed in Fig. 1a) reveals that the main content of nanofibers is carbon, the detected ferrum is residual catalytic particle, as shown in Fig. 1b. It is found that individual carbon nanofiber can be touched with several carbon fibers, shown in Fig. 1a. This may induce fiber/CNFs bridging which is beneficial to the enhancement of mechanical properties of C/C composites.

The flexural properties of C/C composites obtained by different processes are presented in Table 1. Fig. 2 shows the corresponding flexural load–displacement curve. The results demonstrate that CNFs grown in carbon preform are beneficial to C/C composites as the composites with CNFs show a great increase in bending

Table 1The flexural properties of *C/C* composites with and without CNFs.

Samples	Density/g cm ^{−3}	Flexural modulus/GPa	Flexural strength/MPa
CC-no	1.64	19.44	60.74
CC-CNFs	1.63	17.93	108.53

properties compared with C/C composites without CNFs. The bending strength of CC-CNFs is improved from 60.74 MPa to 108.53 MPa, i.e. the flexural strength is increased by 78% than CC-no, as the bending modulus almost remains in the same value. Fig. 2 is also the direct evidence that CC-CNFs have better mechanical properties. Meanwhile CC-CNFs seem to yield pseudo-plastic behaviors. The

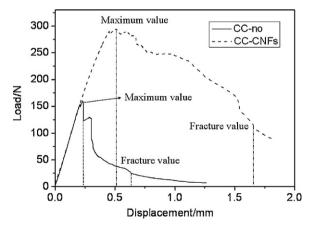


Fig. 2. The flexural load–displacement curve of *C/C* composites fabricated with and without CNFs (CC-no: *C/C* composites without CNFs; CC-CNFs: *C/C* composites with CNFs).

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