

The mechanical property and resistance ability to atomic oxygen corrosion of boron modified carbon/carbon composites



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ABSTRACT

Before being densified by chemical vapor deposition, carbon preform was modified by boron. The mechanical property and resistance to atomic oxygen corrosion of carbon/carbon composites were investigated. The results show that fiber surface modification induces the deposition of high texture pyrocarbon and a moderate interfacial transition layer between carbon fibers and matrix carbon. After being modified by boron, the flexural and compressive strength of carbon/carbon composite is significantly increased. The bending curve has been adjusted with obvious pseudo-ductility phenomenon. The resistance ability to atomic oxygen corrosion is improved significantly. The mass loss and corrosion degree of the modified composite are lower than that of pure carbon/carbon composite.

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1. Introduction

Carbon/carbon composite (C/C composite) is carbon fiber reinforced carbon matrix composite. Due to the excellent properties such as lightweight, strength, high thermal conductivity and high temperature stability, C/C composites are considered as the best candidate materials in the field of aerospace [1–3]. In the harsh application environment of spacecraft, the excellent comprehensive performance of C/C composites is required. On the one hand, good mechanical property is the basis. On the other hand, among many environmental factors in low earth orbit (LEO), atomic oxygen (AO) is one of the most damaging and dominant neutral species (~80%). It has high chemical reactivity and high impinging energy (approximately ~5 eV with a flux of 10^{14} – 10^{15} atoms/cm² s) to material surfaces [4,5]. No doubt, the damaging effects of AO corrosion on the spacecraft materials during operation should be highly focused on. In recent years, many methods have been introduced to improve the resistance ability of composites to AO corrosion, along with the development of space science and technology.

For C/C composites, the fiber/matrix interface is the bridge between carbon fibers and matrix, which is the transformation channel of structure, stress and heat [6,7]. No doubt, the fiber/matrix interface is seriously affecting the performance of C/C composites. Therefore, it is significant to find good methods to

improve the fiber/matrix interface. Pyrocarbon (PyC) is one kind of carbon matrix. PyC comes from the pyrolysis and deposition of hydrocarbon gas during chemical vapor deposition (CVD). There are four typical structures for PyC, high texture (HT), medium texture (MT), low texture (LT) and isotropic [8]. Different kinds of PyC structures have different properties and different bonding state with carbon fibers, resulting in the different microstructure and properties of C/C composites [9–11].

Boron has been known as one of the efficient catalysts for the graphitization of carbon materials [12]. In addition, boron is the only catalyst that can form the solid solution with carbon [13]. In this study, the surface of carbon fiber is treated by boron. Boron solutes into the fiber surface and influences the graphitization process of carbon fibers, which adjusts the surface state of carbon fibers. The modified carbon fibers further influence the deposition of PyC and the interface bonding between carbon fibers and PyC during CVD. The present paper aims to investigate the mechanical properties and resistance ability to AO corrosion of boron modified carbon/carbon composites. In addition, the effect mechanism of boron on the structure and property of carbon/carbon composites is thoroughly analyzed.

2. Experimental

2.1. Preparation of samples

Two 2.5D needled felts (carbon preform) with fiber volume fraction of 40% were made from T300 polyacrylonitrile-based

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carbon fiber. Preform B was first immersed in 5% boric acid solution for 24 h with a water bath at 80 °C and then dried for 4 h at 80 °C. For comparison, preform A was not treated by boric acid. Then the two preforms endured high temperature treatment at 2500 °C in a graphitizing furnace. After high temperature heat treatment, CVD was applied to densify the two preforms to the same density in an isothermal furnace, at about 900 °C, with propylene as the carbon source. The densified composites, named composite A (C/C composite) and composite B (modified C/C composite), were finally graphitized at 2500 °C.

2.2. Characterization

The flexural and compression strengths in the direction vertical to the carbon cloth were measured on the electronic universal testing machine (CSS-44100).

Flexural testing used the three-point bend configuration. A span-to-depth ratio of 10:1 and crosshead speed of 0.5 mm/min were used. Specimens for flexural testing were machined from the two composites in the dimension of 55 mm × 10 mm × 4 mm. Compression testing was performed according to GB8489-87 with a loading speed of 0.2 mm/min. Specimens for compression testing were machined from the two composites in the dimension of 10 mm × 10 mm × 10 mm. Six specimens were prepared for each testing condition and the presented data was the average value of the six specimens.

The AO irradiation experiment was carried out on an AO ground-based simulation device with coaxial source. The circulation density was 1.53×10^{16} atoms/cm² s. The energy of OA was 5–8 eV. The irradiation time was 5–75 h and the vacuum degree was 0.3 Pa. The surface vertical to the carbon cloth was selected to be exposed in the AO beam.

The bulk density of the composite was measured by the Archimedes water immersion method at room temperature analyzer. Data of diffraction peaks (002) were obtained using Rigaku-3014 X-ray diffraction. The surface structure of carbon fibers was examined using

scanning electron microscope (SEM), energy spectrum analysis, X-ray powder diffraction and Raman spectroscopy. The morphology and microstructure of PyC were studied by polarized light microscopy (PLM) and Raman spectroscopy at cross-section of the composites. The structure of interface was studied using transmission electron microscope (TEM).

3. Results and discussion

3.1. The effect of boron on carbon fibers

Fig. 1 shows the SEM images of carbon fibers in two preforms after heat treatment at 2500 °C. As shown in Fig. 1a, the fiber surface of preform A is smooth. However, the fiber surface of preform B is rough with coarsening folds and grooves along the direction of fiber axis, as shown in Fig. 1b. Energy spectrum analysis on carbon fibers of preform B indicates that there are trace amounts of boron on the surface. According to the X-ray results, the graphitization of fiber B is much higher than that of fiber A. The higher graphitization degree of preform B suggests that the surface structure of carbon fibers is improved after being modified by boron.

Many data show that [14] H₃BO₃ dehydrates to B₂O₃ during high temperature treatment. B₂O₃ then reacts with PhCN in carbon fibers, forming BN. BN decomposes into free B and N₂ at 2000 °C. N₂ exhausts from carbon fibers, whereas the free B diffuses into the surface layer of carbon fibers at 2500 °C. There is a good compatibility between boron and carbon because the covalent radius difference between them is 0.011 nm (0.088 and 0.077 nm, respectively). The diffusion coefficient of boron in graphite is up to 6320 cm²/s [15]. So it is easy for boron to diffuse into the surface of carbon fibers at 2500 °C. On the other hand, boron is prone to be electron-poor and easy to attract electrons. So the diffused boron will attract the electrons of carbon atoms, leading to the breaking of C–C bond and the rearrangement of framework structure in carbon atoms. The solid solution of boron in carbon lattice has

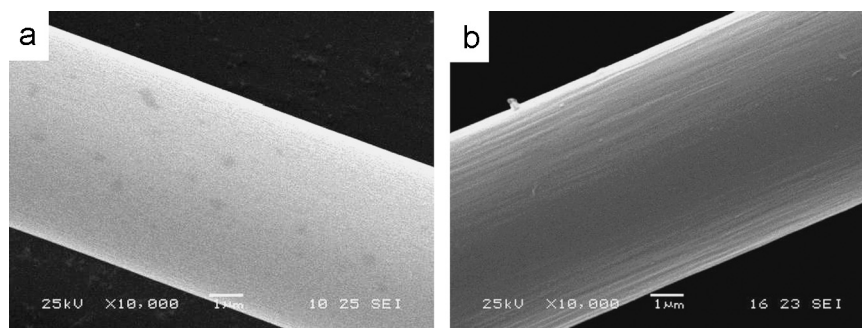


Fig. 1. SEM images of carbon fibers after heating at 2500 °C. (a) Carbon fiber in preform A and (b) Carbon fiber in preform B.

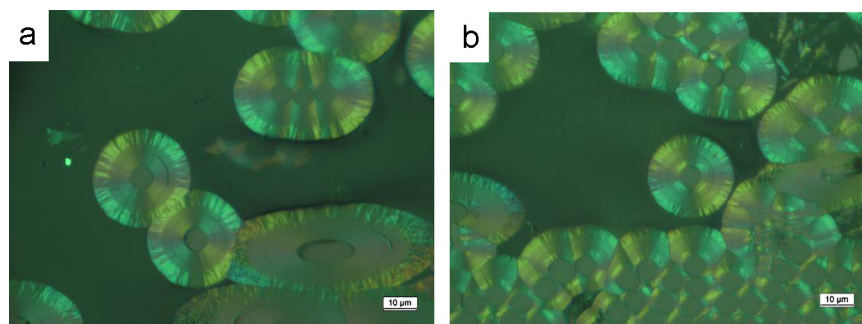


Fig. 2. Optical photographs (under PLM). (a) Composite A and (b) Composite B.

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