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Internal friction behavior of unidirectional carbon/carbon composites after different fatigue cycles

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ABSTRACT

Internal friction behavior was utilized as an indirect metric to study structural change in carbon/carbon composites after fatigue tests. In this work, two kinds of unidirectional carbon/carbon composites with different densities were prepared by isothermal chemical vapor infiltration (ICVI), and loaded under stress level of their fatigue limit. The internal friction behavior of the composites after different fatigue cycles was studied. After the initial 10^4 fatigue cycles, since the matrix began to break and shed, frictional damping that happened between the fiber and matrix interfaces increased and the bulk internal friction increased rapidly. Between 10^4 and 5×10^5 fatigue cycles, holes formed at interfaces because of continued shedding of the matrix. Since the holes reduced contact area between different phases, frictional damping decreased, as along with the internal friction increment ratio. Between 5×10^5 and 10^6 fatigue cycles, interfacial bonding between carbon fiber and matrix was in a steady state, so structure and internal friction of the composites did not change. The results revealed that internal friction was an effective method to characterize structural change of carbon/carbon composites after fatigue tests.

1. Introduction

Carbon fiber reinforced carbon composites, often known as carbon/carbon composites (C/C), have superior mechanical properties and structural integrity for aerospace structural applications at temperature till 3000 °C either in vacuum or in inert environment. C/C composites have complicated structures, i.e. carbon fibers, matrix and interface between the two phases. Fatigue behavior of C/C composites was related to the interaction between these phases of the composites [1,2]. As a result, fatigue failure of C/C composites was really complicated, and there was no systematic theory that had been widely accepted.

Currently, characterization methods used for characterizing structural changing of metal materials and composites after fatigue test can be divided into two classes, i.e. direct observation and indirect characterization. Conventional direct observation methods were optical microscope, scanning electron microscope (SEM), polarizing microscope (PLM), etc. And the common indirect characterizations included X-ray micro-tomography [3], sound emission [4], electronic resistance monitoring [5], etc. Since the internal structure of C/C composites was quite complicated, various structures had different structural changes after fatigue tests. The observation ranges of SEM and PLM were limited, so the structural changes in local region should not represent the whole composites. Indirect characterization predicted internal structural change by monitoring a varying performance of the composites, and had received wide attention. Since performance was the ultimate macroscopic consequences caused by a large number of structural changes inside the composites, indirect characterizations could characterize changing of internal structures more objectively than direct characterization.

Internal friction or damping depended on the microstructural change of materials. Since internal friction was sensitive to the variation of microstructure [6], internal friction characterization was an effective and accurate tool to study microstructural changes and the generation of defects in materials. Currently, internal friction had gained increasing attention in the composite field [7–10]. Hong [11] studied the internal friction of C/SiC composites coated with two kinds of environment barrier coatings in corrosive environments. He reached the conclusion that internal friction was sensitive to corrosion behavior and was a possible method to evaluate the corrosion behavior of composites. Boccaccini [12] utilized internal friction to assess the development and healing of micro-cracking damage in SiC fiber reinforced glass matrix composites. The results showed that internal friction was an excellent indicator of micro-cracking evolution. These studies

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showed that internal friction characterization was a powerful technique for investigating not only molecular structure and motion, but also structural variation and damage accumulation of composites.

There is little research literature focusing on the issue of using internal friction to characterize the microstructural change of C/C composites after fatigue tests. In this study, the relationship between internal friction and microstructure during fatigue tests is discussed in details. This study is aimed to obtain an empirical conclusion that the fatigue life of C/C composites can be predicted using internal friction characterization.

2. Experimental

2.1. Materials

Unidirectional C/C composites were reinforced by polyacrylonitrile-based carbon fibers (Toray, T300-3K) aligned in only one direction. The model used for constraining fibers was divided into many parallel grooves, and carbon fibers were wounded in these grooves. The fiber volume fraction of the unidirectional carbon fiber preforms was 39–41%. Using methane (CH_4) as precursor gas and nitrogen (N_2) as diluted gas, the unidirectional carbon fiber prefabrications were densified in the process of ICVI, at 1050 °C and a total pressure of 2.5 kPa.

Two kinds of samples – termed sample 1 and sample 2 – were investigated. Densification times of sample 1 and sample 2 were 120 h and 240 h, respectively. Average density of sample 1 was 1.60 ($\pm$0.01) g/cm^3 and that of sample 2 was 1.70 ($\pm$0.01) g/cm^3.

Open porosity of sample 1 and sample 2 was 12–14% and 10–12%, respectively. The open porosity was measured according to ASTM C20 by utilizing a water saturation method.

2.2. Mechanical test

Static flexural strengths of the samples were measured by a three-point bending test in ambient environment with a 40 mm span and a crosshead speed of 0.5 mm/min by means of an electrohydraulic-servo testing machine (Instron Co. Ltd., 8872). The number of specimens used in every test was eight. The specimens for mechanical tests (as shown in Fig. 1) were prepared using a diamond saw, and then the specimens were hand ground using 400 grit size silicon carbide (SiC) sand papers. The dimension of specimens used in mechanical tests was 4 mm (thickness) $\times$ 8 mm (width) $\times$ 55 mm (length).

The test machine used for flexural fatigue was the same as the static flexural tests, as well as the test mode and span length. The applied stress ratio (R: ratio of minimum to maximum applied stress) of the fatigue tests was 0.1, the flexural fatigue loading frequency was 15 Hz, and the fatigue loading waveform was a sine wave. For all cases, the diameter of the loading pin was 5 mm and the dimensions of specimens used in fatigue tests were the same as those used in the static test. In this study, the fatigue limit of the specimens was determined as the value where the specimen was not broken even though the stress was repeated 10^6 times.

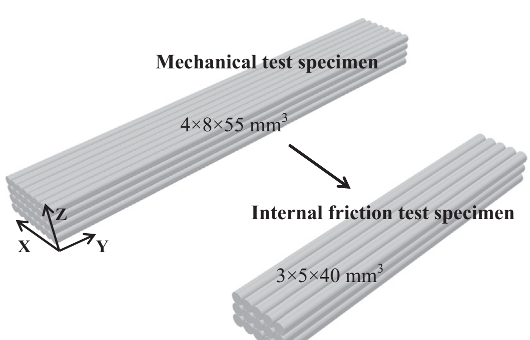

Fig. 1. Schematics of specimens used in mechanical test and internal friction test.

2.3. Internal friction test

Specimens used for internal friction tests were cut using a diamond saw, and then hand ground using 800 grit size SiC sand papers to the dimension of 3 mm (thickness) $\times$ 5 mm (width) $\times$ 40 mm (length) (as shown in Fig. 1). The internal friction tests were performed on a dynamic mechanical analyzer by means of a forced non-resonant technique at room temperature. The range of frequency was from 0.01 to 9.9 Hz, and the strain amplitude was of the order of 8×10^{-5}. The internal friction (Q^{-1}) was measured according to Eq. (1) as follows:

$$Q^{-1} = \tan\delta = E''/E' \quad (1)$$

where δ is the loss angle between the applied stress and strain, E' is the dynamic storage modulus (elastic), and E'' is the loss modulus (viscosity).

For each Q^{-1} value after different fatigue cycles, three specimens were tested. The average value was then calculated.

2.4. Microstructure test

As indicated in Fig. 1, the X–Z plane of the as-prepared samples was sanded and polished, and then was observed under polarized light microscope (PLM, Leica DMLP optical microscope).

Structural changes of samples were observed using a scanning electron microscope (SEM, JSM-6360).

3. Results and discussion

3.1. Results

Fig. 2 is a PLM image of as-prepared C/C composites. According to their different optical activities under PLM, pyrocarbon could be divided into four textures, i.e. ISO, DL, SL and RL. The microstructure of pyrocarbon significantly influenced the mechanical properties of C/C composites [13]. Binary layer textures (marked as A and B) were observed between carbon fiber bundles. A was RL texture and B was SL texture. In addition, pyrocarbon deposited inside carbon fiber bundles (marked as C) was thin and had a typical RL texture.

The average flexural strengths of sample 1 and sample 2 were 209.5 MPa and 227.8 MPa, respectively. As the density increased,

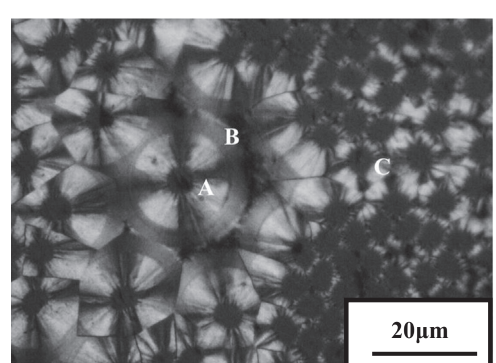

Fig. 2. PLM image of as-prepared carbon/carbon composites: A and B refer to binary layer textures between fiber bundles, C refers to pyrocarbon inside fiber bundles.