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RESEARCH PAPER

Influence of the microstructure of the carbon matrices on the internal friction behavior of carbon/carbon composites

Wei Yang, Rui-ying Luo*, Zhen-hua Hou, You Zhang, Hai-dong Shang, Ming-yang Hao
School of Physics and Nuclear Energy Engineering, Beihang University, Beijing 100191, China

Abstract: Three carbon/carbon composites with rough laminar, smooth laminar and dual matrix carbon were prepared by chemical vapor infiltration (CVI) using hydrogen-diluted methane, CVI using nitrogen-diluted propane, and two-step CVI using first methane/hydrogen and carbon dioxide and then furan resin impregnation and carbonization. The influence of the microstructure of the carbon matrix on the internal friction behavior of the composites was investigated. Results indicate that the microstructure of the carbon matrix plays an important role in the internal friction. The overall internal friction is related to the motion of dislocations, the sliding of the fiber/matrix interface and the sliding of the carbon planes. The internal friction of the composite is very sensitive to temperature and amplitude, but less sensitive to frequency. Among these composites, the dual matrix carbon has the highest density of crystal-defects and the highest internal friction while the rough laminar carbon has perfect carbon planes and the lowest internal friction.

Key Words: Carbon/Carbon Composites; Densification process; Mechanical characterization; Internal friction

1 Introduction

Carbon/carbon (C/C) composites are considered to be potentially ideal high-temperature structural materials for advanced aero-engine applications owing to their low density, outstanding mechanical properties, high thermal conductivity and low thermal expansion coefficient (CTE)[1]. In addition, the engine weight can be effectively reduced, contributing to low fuel consumption. Especially at some engine parts, such as gasket and sealing rings, have to undergo high-speed rotational and dynamic load. Therefore, it is very essential to investigate the internal friction of C/C composites to match the requirement of engine components. Based on the research of internal friction, scientific and technological workers can design aeronautic and astronautic structural materials with satisfying internal friction to ensure that the component can be used reliability. Moreover, the internal friction analysis can be used as a non-destructive characterization method to evaluate the composite properties.

The matrix microcracks in fiber reinforced glass matrix composites are assessed by internal friction[2]. According to the study of unidirectional C/C composites, the internal friction of C/C composites decreases with the frequency from 0.01 to 1 Hz, and then it slightly increases to 5 Hz, where the value of the internal friction reach about 0.5×10^{-2}[3]. But the internal friction of C/C composites increases with the frequency from 0.01 to 2 Hz, and the value of the internal friction is very small (only 3×10^{-3})[4]. In a recent report, the internal friction of C/C composites decreases with the frequency from 0.01 to 1 Hz, and then increases obviously, and the value of the internal friction is about 1×10^{-2}[5]. In the work of C/C composites at elevated temperatures, before temperature reach 2 000 K, the internal friction of C/C composites is almost constant below 2 000 K and increase with temperature above 2 000 K [6]. The internal friction of C/C composites decreases with bulk density and increases with the volume fraction of fibers[7]. There is little research literature on the issue of using internal friction to characterize the matrix microstructure of C/C composites.

Various carbon matrices have exhibited excellent mechanical properties, however, these carbon matrices with different structures have hardly been considered in terms of internal friction behaviors of C/C composites in former studies. The aim of this work is to investigate internal friction behaviors of C/C composites with the three typical carbon matrices, and try to find the relationship between internal friction behaviors and the microstructure of carbon matrix. The C/C composites with satisfactory dynamic mechanical properties are obtained through CVI or PIP, and we can design aeronautic and astronautic structural materials with satisfying internal friction.

2 Experimental

A quasi three dimensional needled polyacrylonitrile based carbon fiber felts were used as a preforms and the density of the preform was about 0.55 g/cm^3. The size of preform was *Φ* 230×20 mm, and the carbon fiber preform was firstly heat-treated at 2 300 °C for 2 h. Chemical vapor

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***Corresponding author.** E-mail: ryluo@buaa.edu.cn

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infiltration using nitrogen-diluted propane and hydrogen-diluted methane was used to densify the preform to prepare C/C composites, named as SLC and RLC, respectively. The preform was firstly densified by chemical vapor infiltration using methane/ hydrogen and carbon dioxide till the density up to 1.50-1.60 g/cm^3, and then impregnated with furan resin and carbonized to yield another C/C composite, named as DMC. Finally, the three kinds of composites were heat-treated at 2 400 °C after the densities are about 1.72 g/cm^3.

The dynamic mechanical properties were characterized with a dynamic mechanical analyzer (DMA800) by means of three-point bending forced vibration in air. The specimens were rectangular bars with a size of 60 mm×4 mm×2 mm, cut from the fabricated composites. The span was 40 mm. Loading direction was perpendicular to the cloth layer direction. The testing frequency was ranged from 0.1 to 50 Hz, and the amplitude was from 0.004% to 0.05% of the strain. The temperature was from 25 to 450 °C and the heating rate was 5 °C/min. The microstructures of the three C/C composites were characterized by polarized light microscopy (PLM, Neophot21). Then, the polished surfaces of the C/C composites were analyzed by Raman spectrometry (LabRAM Aramis) with a laser excitation wavelength of 532 nm. The powder samples were examined by X-ray diffraction (XRD, D/M-2200) in the 2θ range of 15° and 80° with monochromatic Cu *K*a radiation. According to Bragg's law, d_{002} was obtained from the Equation (1):

$$d = \frac{\lambda}{2\sin\theta} \tag{1}$$

Where λ is the wavelength of Cu *K*a radiation and θ is the diffraction angle in radians. Crystallite size L_c is obtained from the Scherrer Equation (2):

$$L_c = \frac{0.9\lambda}{B\cos\theta} \tag{2}$$

Where B is the half maximum intensity in radians of the (002) peak. Graphitization degree g is calculated from the Maire and Mering Equation (3):

$$g = \frac{0.3440 - d_{002}}{0.3440 - 0.3354} \times 100\% \tag{3}$$

In addition, the matrix morphologies of the C/C composites were observed by a scanning electron microscope (SEM, JSM-6700F).

3 Results and discussion

3.1 The microstructure of the C/C composites

Fig. 1 presents the microstructure of the three kinds of C/C composites SLC, RLC and DMC under polarized light. It can be seen that the C/C composites are composed of three parts: carbon fibers, matrix carbon and small pores. Carbon fibers are fabricated by polyacrylonitrile, exhibiting obviously optically isotropic. It is very clear that both SLC and RLC present single optical structure with regular extinction crosses, which represent smooth laminar (SL) and rough laminar (RL) pyrocarbon structure, respectively, while the DMC composite exhibits dual matrix including the pyrocarbon with the overgrowth cones, the resin carbon (RC) and the interface of pyrocarbon/RC. Both the interface of pyrocarbon/RC and the RC exhibit low anisotropy. This is because that dual matrix weakens the optical anisotropy and the uniformity of matrix.

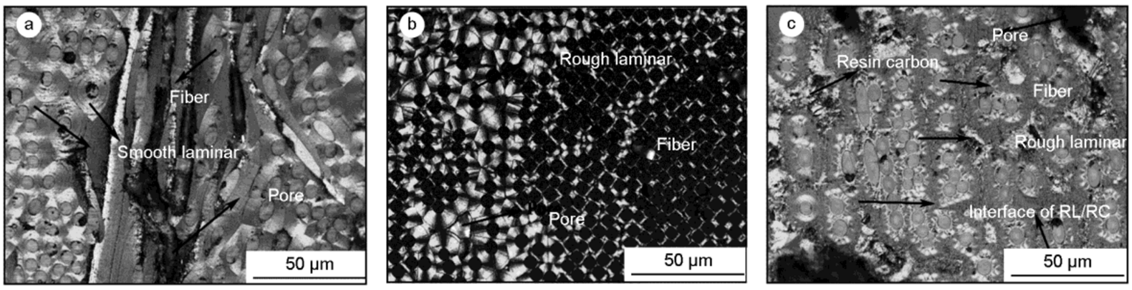

Fig. 1 PLM images of the C/C composites: (a) SLC, (b) RLC and (c) DMC.

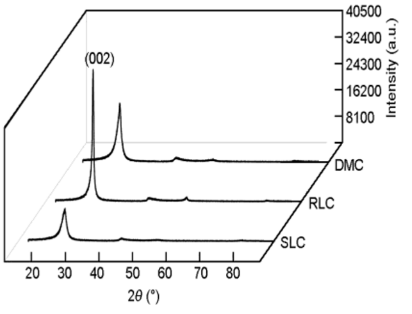

Fig. 2 XRD patterns of the three kinds of C/C composites.

The XRD spectra of the C/C composites are shown in Fig. 2. All of the three kinds of composites exhibit a sharp (002) peak, and their physical properties are listed in Table 1. Since RL pyrocarbon is easy to be graphitized, RLC has the highest g and L_c among the three kinds of composites. In contrast, SL pyrocarbon is difficult to be graphitized, so the corresponding g and L_c are rather low. Although graphitization of RC is also hard, DMC has the relatively high average values of g and L_c.

The C/C composites with dual matrix[8] possess a high g value, which may be due to the fact that the interface stress of pyrocarbon/RC would thermally induce the stress