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# Effect of structure of pyrocarbon on the static and dynamic mechanical properties of carbon/carbon composites



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#### ARTICLE INFO

#### ABSTRACT

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Keywords: Composites Internal friction Mechanical characterization Carbon/carbon composites with two types of pyrocarbon matrices were prepared by Chemical Vapor Infiltration using methane and hydrogen, with or without a small added amount of carbon dioxide, as source and carrier gases. The pyrocarbon structures were respectively the usual Rough Laminar structure and a complex hybrid structure featuring Rough Laminar and overgrowth cones. Their microstructures, static and dynamic bending performances were characterized. Experimental results indicate that the hybrid structure can effectively improve the flexural and damping properties of composites compared with the simple structure. By introducing carbon dioxide in the gas phase, defects were formed in the deposits and the resulting overgrowth cones led to a better interconnection of matrix deposited on adjacent fibers. They deteriorate the texture degree of pyrocarbon but effectively improve the matrix strength, and they not only make the flexural strength of composites increase by 27.3% compared with reference composites, but also result in storage modulus, loss modulus and internal friction of composites increase by 3.5%, 30.5% and 23.1%, respectively, at 25° C, 10 Hz and 0.025% strain. The defects make the internal friction of composites more sensitive to temperature and amplitude, but on the other hand make the storage modulus of composites less sensitive to temperature.

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#### 1. Introduction

Carbon/carbon (C/C) composites prepared by Chemical Vapor Infiltration (CVI) exhibit a wide variety of mechanical, thermal and braking properties depending on their matrix structures, which make them very interesting for application in several domains [1– 3]. Some C/C composites components, such as disks and sealing rings, are often considered to be used for high-speed rotating parts and bear dynamic load. Therefore, it is essential to investigate the dynamic mechanical properties of C/C composites. Moreover, some useful structural information can be obtained by studying dynamic mechanical properties. Wang et al. [4] have implied that the internal friction of C/C composites decreases with an increase in bulk density and increases with an increase in the volume fraction of the fiber. Hou et al. [5] have proposed two internal friction mechanisms, the thermoelastic mechanism and the static hysteresis mechanism, to explain the special internal friction characteristics of C/C composites. However, the effect of the texture and structure of the pyrocarbon matrix, which confers its excellent mechanical properties to C/C composites, on their

\* Corresponding author. Tel.: +86 10 82338267; fax: +86 10 5173 6729. *E-mail address:* ryluo@buaa.edu.cn (R. Luo). dynamic mechanical properties has not been reported in former studies, to our knowledge.

The purpose of present work is to investigate the static and dynamic mechanical properties of C/C composites fabricated by the CVI technique from different gas sources. In this paper, a complex rough laminar hybrid structure with overgrowth cones and simple rough laminar structure C/C composites were prepared from different gas mixtures, and their static and dynamic mechanical properties were present. The internal friction characteristics were explained in terms of the internal friction mechanisms.

#### 2. Experimental

A quasi three dimensional needled polyacrylonitrile based carbon fiber preform was used as reinforcement. The density was about 0.55 g/cm<sup>3</sup>, and their size was  $\Phi$ 230 mm × 20 mm. Carbon fiber preforms were firstly heat-treated at 2300° C for 2 h, and infiltrated by isothermal CVI at 1080–1110° C with total pressure of 1–5 kPa. Methane (CH<sub>4</sub>) was used as the precursor, hydrogen (H<sub>2</sub> for composite I) and a hydrogen–carbon dioxide gas mixture (H<sub>2</sub>–CO<sub>2</sub> for composite II) were adopted as auxiliary gases. The volume ratios of CH<sub>4</sub>/H<sub>2</sub> and CH<sub>4</sub>/H<sub>2</sub>/CO<sub>2</sub> were 2/1 and 2/1/ 0.06, respectively. The residence time of mixture gas was about 0.1 s. The infiltration experiments were performed stepwise. The mass increase of the preforms was measured on a balance every 50 h of the infiltration run. A crust was formed on the H<sub>2</sub>-prepared C/C composites: it has been removed by machining. The surface of CO<sub>2</sub>-prepared C/C composites has not formed any crust until the end of the run, at 400 h. After a final graphitization processing (2400° C during 2 h), the final density was 1.72 g/cm<sup>3</sup> for both composites.

The dynamic properties characterizations were carried out in air from room temperature (RT) to  $450^{\circ}$  C in a dynamic mechanical analyzer (DMA800) by means of three-point bending forced vibration. The specimens were rectangular bars with a size of 2 mm × 4 mm × 60 mm, cut from the fabricated composites. The span was 40 mm and loading direction was perpendicular to the cloth layer direction, as shown in Fig. 1. The testing frequency was



**Fig. 1.** Schematic of three-point bending test on DMA 800 (all dimensions are in millimeters; P is the bending load).



Fig. 2. PLM images of C/C composites: (a) I; (b) II.

from 0.1 to 70 Hz, and the strain amplitude was from  $1 \times 10^{-4}$ % to 0.05%. Heating rate was 5° C/min. For both materials, only one test of each kind was performed: one temperature scan at fixed deformation amplitude and frequency, one frequency scan at fixed temperature and amplitude, one amplitude scan at fixed frequency and temperature. The static mechanical properties were tested by three-point bending test according to a previously reported method [6], and the fracture surfaces were examined by scanning electron microscope (SEM, CS3400). The microstructure of C/C composites was characterized by polarized light microscopy (PLM, NEOPHOT21). Then, the polished surfaces of C/C composites were analyzed by Raman spectrometry (LabRAM, HR800), with two laser excitation wavelengths of 514.5 nm and 325 nm, respectively. Powdered samples of both composites were characterized by transmission electron microscopy (TEM, JEOL2100).

#### 3. Results

#### 3.1. Microstructure

PLM images of C/C composites I and II are shown in Fig. 2. The matrix of composite I from  $CH_4$ - $H_2$  consists of simple rough



Fig. 3. The multi-wavelength Raman spectra of C/C composites I and II.

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