

# Effects of fiber-type on the microstructure and mechanical properties of carbon/carbon composites

Zhen-hua Hou, Ming-yang Hao, Rui-ying Luo\*, Qiao Xiang, Wei Yang, Hai-dong Shang, Huai-zhe Xu

School of physics and Nuclear Energy Engineering, Beijing University of Aeronautics and Astronautics, Beijing 100191, China

**Abstract:** Two carbonized and oxidized polyacrylonitrile fiber (OPF) felts and one polyacrylonitrile-based carbon fiber (CF) felt were used as preforms to prepare three carbon/carbon composites by chemical vapor infiltration and the effects of the fiber type on the microstructure and mechanical properties of the composites were investigated. The microstructure was characterized by polarized light microscopy and Raman spectrometry and the mechanical properties were characterized by nanoindentation and three-point bend tests. The two carbonized OPFs are surrounded by a dark laminar layer of about 1.4-2.6  $\mu\text{m}$  thickness followed by a rough laminar layer about 10.2-11.6  $\mu\text{m}$  thick while the CFs are surrounded by a smooth laminar layer about 8.8  $\mu\text{m}$  thick and a rough laminar layer about 4.4  $\mu\text{m}$  thick. Nanoindentation indicates that the elastic modulus and hardness of the carbonized OPFs are obviously lower than that of the CFs, and the elastic modulus and hardness of the matrix decrease with increasing extinction angle determined from polarized light microscopy. The tensile and flexural strengths and moduli of the OPF-derived composites are lower than that of the CF-derived one, due to the lower moduli of the OPFs. However, for the OPF-derived composites the ductility factor increases by about 224-235% owing to the high content of rough laminar carbon and the obvious shrinkage of the OPFs after graphitization. A model involving the three components in the composites is proposed to predict the tensile modulus and the differences between the experimental and predicted results were less than 10%.

**Key Words:** Carbon/carbon composites; Microstructure; Chemical vapor deposition; Carrier gas

## 1 Introduction

It is of prime importance to achieve high bulk densities of carbon/carbon (C/C) composites with rough laminar (RL) pyrolytic carbon (PyC), which is the key point for fabricating C/C braking materials with high mechanical, outstanding thermal and braking properties<sup>[1-5]</sup>. But the high bulk density is frequently limited by blocking of the pore entrances through chemical vapor infiltration (CVI) route. To overcome this problem, the reduction of the infiltration rates on the surface and inhibition an overgrowth of the pore entrances from the outside are recommended<sup>[6]</sup>. Therefore, the new processes, such as temperature and pressure gradients, forced and pulsed flow processes, have been investigated<sup>[7,8]</sup>, but they do not replace the conventional isothermal CVI techniques for mass production of carbon brake disks due to their homogeneous deposition of PyC. Moreover, Zhang<sup>[6]</sup> have proved that the inside-out infiltration can be obtained by adjusting the processing parameter (e.g. temperature, pressure, residence time and the ratio of C/H). Further, the new carrier gas, H<sub>2</sub>, has been proposed by Becker et al.<sup>[9]</sup> because it can inhibit

carbon deposition by blocking the free active sites, and they obtained the high bulk density C/C composites in the laboratory<sup>[10]</sup>. Tang et al<sup>[11]</sup> has employed hydrogen as carrier gas to prepare large size carbon brake disks fabricated by thermal gradient CVI. The average bulk density, the radial density gradient and the texture of PyC with H<sub>2</sub> as carrier gas were improved compared with N<sub>2</sub>.

Although numerous works have been devoted to improve traditional CVI efficiency and reduce the processing time, the high bulk density usually comes off second-best. To gain the high bulk density, especially above 1.70 g/cm<sup>3</sup>, brake disk often needs to remove the surface crusts in the last stage of infiltration. Unfortunately, this is very inefficient and even futile. In this work, a new carrier, CO<sub>2</sub>, was proposed because it can obviously reduce the surface deposition and do not obviously inhibit the in-pore infiltration, and the effect of the type of carrier gas (H<sub>2</sub> and CO<sub>2</sub>) on the densification rate, bulk density and microstructure of the carbon disks fabricated by isothermal CVI was investigated.

Received date: 25 Feb. 2015; Revised date: 20 Jul. 2015

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

Copyright©2015, Institute of Coal Chemistry, Chinese Academy of Sciences. Published by Elsevier Limited. All rights reserved.

DOI: 10.1016/S1872-5805(15)60196-2

## 2 Experimental

### 2.1 Preparation of C/C composites

In the present work, a quasi three dimensional needled carbon fiber preform was used as a substrate for CVI. The density of the preform was about  $0.55 \text{ g/cm}^3$ . The size of preforms is  $\Phi 450\text{-}\Phi 230 \times 20 \text{ mm}$ . Carbon fiber preforms were firstly heat-treated at  $2300 \text{ }^\circ\text{C}$  for 2 h, and infiltrated by isothermal CVI at  $1080\text{-}1130 \text{ }^\circ\text{C}$  with a total pressure of 1-3 kPa. Methane ( $\text{CH}_4$ ) was used as the precursor.  $\text{H}_2$  and  $\text{CO}_2$  were used as carrier gases. The ratio of precursor to carrier gas was about 7 : 1. All the infiltration experiments were performed stepwise. The density of preforms was tested after each infiltration run of 50 h. The preforms were machined with 300# corundum abrasive papers after an initial infiltration for 50 h in order to measure the bulk volume. The preforms were not machined to remove the crust in the rest infiltration time in order to obtain the real surface topography and density gradient. Finally, the carbon disks were graphitized at  $2300 \text{ }^\circ\text{C}$  for 2 h.

### 2.2 Characterization of C/C composites

Specimens of  $20 \times 10 \times 6 \text{ mm}^3$  were sliced from each sample at different positions along the radial and thickness directions as shown in Fig. 1, to evaluate the homogeneity of density. The surface topography of the composites was characterized by scanning electron microscopy (SEM, S-4800). The microstructure of the composites perpendicular to needle punched surface was observed under a polarized light microscope (PLM, Neophot21). Then, the polished surfaces of C/C composites were analyzed by Raman spectroscopy (LabRAM, HR800), with two laser excitation wavelengths of 514.5 nm and 325 nm. Meanwhile, the powder samples were examined by X-ray diffraction (XRD, D/M-2200) between  $15$  and  $80^\circ$  ( $2\theta$ ) with monochromatic (40 kV, 40 mA) Cu ka radiation to determine  $d_{002}$ -spacing and crystallite size ( $L_c$ ). Powdered samples of the composites were characterized by transmission electron microscopy (TEM, JEOL2100).

## 3 Results and discussion

### 3.1 Effect of carrier gases on densification rate and bulk density

The effect of carrier gases on the densification rate of the carbon disks is shown in Fig. 2. In the initial 50 h, the densification rate obtained from  $\text{CH}_4\text{-H}_2$  is obviously higher than that from  $\text{CH}_4\text{-CO}_2$ , while the densification rate from  $\text{CH}_4\text{-H}_2$  is lower than that from  $\text{CH}_4\text{-CO}_2$  in the rest of infiltration time, especially in the last 200h. The average bulk density of the carbon disk obtained from  $\text{CH}_4\text{-H}_2$  ( $1.626 \text{ g/cm}^3$ ) is obviously lower than that obtained from  $\text{CH}_4\text{-CO}_2$  ( $1.723 \text{ g/cm}^3$ ). In comparison to  $\text{H}_2$ ,  $\text{CO}_2$  acting as an oxidizing carrier gas, plays a quite different role in CVI.  $\text{H}_2$  can inhibit both the homogeneous pyrolysis reactions and the

heterogeneous deposition reactions. For the gas-solid heterogeneous reaction, on the one hand,  $\text{CO}_2$  favors the  $\text{CO}_2 + \text{C}(\text{PyC}) = 2\text{CO}$  reaction leading to a reduction of the carbon deposition rate effectively. For the homogeneous pyrolysis reactions, on the other hand,  $\text{CO}_2$  plays an active role in the methane pyrolysis [12]. Only a small fraction of reactive species formed by pyrolysis of  $\text{CH}_4$  in the gas phase can be chemisorbed on out surface of the preform under a low ratio of the surface area to the deposition volume ( $[A/V]$ ). A large fraction of the reactive species is removed by the flowing gases. Because of a much high ratio of  $[A/V]$  inside the preform, the reaction species formed should immediately be chemisorbed and pyrolyzed into PyC by several complex gas-solid heterogeneous reactions inside pores. Therefore, the gas-gas homogeneous reaction only plays a minor role, and the adsorption and gas-solid surface reactions play a critical role in CVI.

In the initial stage, the low densification rate from  $\text{CH}_4\text{-CO}_2$  may result from the oxidizing effect of  $\text{CO}_2$  because the CVI are controlled by chemical reaction. With an increase of the degree of pore filling, diffusion gradually becomes dominant, and the diffusion rate of the reaction species largely determines the densification rate. The diffusion rate of  $\text{H}_2$  is so much high that the inhibition effect of  $\text{H}_2$  will not obviously decrease. While the oxidizing effect of  $\text{CO}_2$  gradually reduces with increasing depth of pores or decreasing diameter of pores

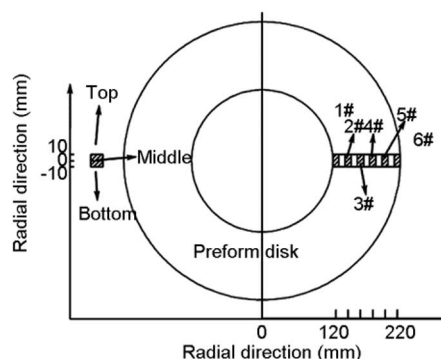


Fig. 1 Configuration of specimens sliced from the carbon disk for density measurements.

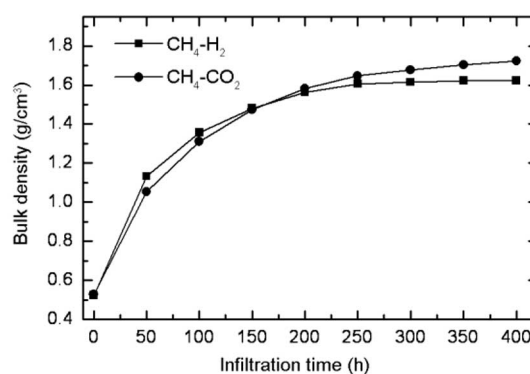


Fig. 2 Average bulk densities of the carbon disks obtained from  $\text{CH}_4\text{-H}_2$  and  $\text{CH}_4\text{-CO}_2$  as a function of infiltration time.

Download English Version:

<https://daneshyari.com/en/article/1558330>

Download Persian Version:

<https://daneshyari.com/article/1558330>

[Daneshyari.com](https://daneshyari.com)