

Improvement of film boiling chemical vapor infiltration process for fabrication of large size C/C composite

Ji-ping Wang^{*}, Jun-min Qian, Guan-jun Qiao, Zhi-hao Jin

State Key Laboratory for Mechanical Behavior of Materials, Xi'an Jiaotong University, Xi'an 710049, People's Republic of China

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Abstract

An improved film boiling chemical vapor infiltration process was developed to fabricate a large size C/C composite with homogeneous density and microstructure. The C/C composite was prepared by processing a disc-shaped carbon felt preform, whose upper and lower sides were fixed and heated simultaneously by two flat surfaces of two heat sources, with kerosene as a precursor at 1050 °C for 3 h at an atmospheric pressure. The in-situ temperature distribution along the radial direction of the preform upper surface was analyzed to get better information and control of the process. Experimental results show that the average density of the composite of $\Phi 110 \times 10 \text{ mm}^3$ size is about 1.72 g/cm³ and its maximal difference along radial direction is 0.05 g/cm³. Polarized light microscopy (PLM) and scanning electron microscopy (SEM) reveal that the carbon fibers of the composite are surrounded by ring-shaped pyrocarbons with a thickness of $\sim 20 \text{ }\mu\text{m}$, and that pyrocarbons are delaminated to 4–6 layers. A schematic model is proposed to analyze the process by dividing the reactor into different regions associated with specific functions. © 2005 Elsevier B.V. All rights reserved.

Keywords: Carbon/carbon composite; Chemical vapor infiltration; Rapid densification; Microstructure

1. Introduction

Carbon/carbon (C/C) composites are widely applied in many fields for their low density, excellent thermal and mechanical properties with smooth frictional behavior and good biocompatibility [1]. Currently, the main method for fabricating C/C composites in industry is the isothermal chemical vapor infiltration (CVI) technique. However, it has a major intrinsic drawback, namely, a long processing period is inevitable to obtain desired density [2,3]. Fortunately, another method called as film boiling chemical vapor infiltration (FBCVI) or chemical liquid-vaporized infiltration (CLVI) [4–7] has been developed to increase the deposition efficiency. It appears very attractive to prepare C/C composite in a short processing time with a high carbon yield which is about one order of magnitude larger than by classic isothermal CVI.

It is known that the FBCVI method involves a strong thermal gradient inside cold wall reactor. A mobile densification front is created in a porous preform which is directly immersed into a

liquid hydrocarbon precursor. The principle, the experimental device, and the influences of some basic parameters (temperature, pressure, precursors, etc.) have already been well studied [3,5,6]. Further investigations are carried out experimentally or theoretically to reveal the complex chemical reactions leading to the pyrocarbon matrix in a confined place and the role of the heat and mass transfers inside porous preform [8–11].

Nevertheless, these studies were mainly carried out in laboratory reactors. The prepared C/C composites are usually of thin-walled tubular shape with small dimensions (the wall thickness is below 35 mm). Moreover, spatial density gradients exist in the composites, where the density at the interior regions near the heat source is the highest and that at the outer surfaces near the liquid precursor is the lowest [11,12]. Therefore, further improvement of this process is necessary for preparing large bulk C/C composite of more regular shape with homogeneous density distribution and uniform microstructure.

For this purpose, a double heat source design is firstly developed in the present work. A large size C/C composite disc was fabricated by this improved FBCVI method. To get better information and control of the whole process, the in-situ temperature distribution in the preform was recorded and

^{*} Corresponding author. Tel.: +86 29 82667942; fax: +86 29 82665443.
E-mail address: wang.jiping@tom.com (J. Wang).

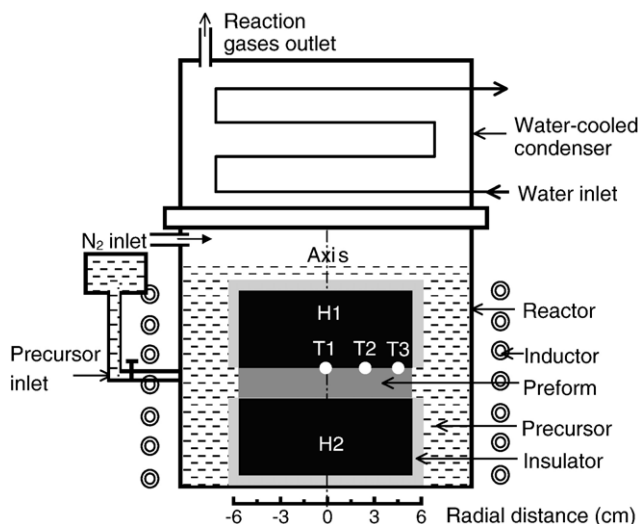


Fig. 1. Sketch of experimental device for preparing C/C composite disc.

analyzed. The density, porosity and microstructures of the prepared composite are characterized by Archimedes principle, polarized light microscopy and scanning electron microscopy techniques. Finally, a schematic model is proposed to study the densification process.

2. Experimental procedure

2.1. Material preparation

2.1.1. Preform and precursor

A PAN-based carbon felt (thickness: 10 mm, bulk density: $\sim 0.20 \text{ g/cm}^3$, fiber diameter: $\Phi 9\text{--}13 \text{ }\mu\text{m}$) was used as a preform in this study. A liquid hydrocarbon mixture, kerosene (molecular formula: $\text{C}_{10}\text{H}_n\text{--C}_{16}\text{H}_m$) with a boiling temperature range of $180\text{--}230 \text{ }^\circ\text{C}$, was chosen as a precursor, which was proven to be a feasible and efficient precursor [12].

2.1.2. Experimental set-up

The experimental device is schematically shown in Fig. 1. In a cylindrical quartz glass reactor, two graphite cylinders (H1 and H2) with diameter of $\Phi 110 \text{ mm}$ were placed at the same axis as the reactor and inductively heated by an inductor coil. The preform cut in disc shape ($\Phi 110 \text{ mm}$) was fixed between the lower surface of H1 and the upper surface of H2 and heated by both of them. The residual surfaces of the two graphite cylinders were wrapped with a thermal insulator. Both the cylinders and the preform were immersed into the liquid precursor. Three thermocouples (T1, T2 and T3) were located at the interface of H1 and the preform. Their distances to the axis are 0 mm, 25 mm and 50 mm, respectively. Thus the inner, middle and outer temperatures of the upper surface of the preform can be simultaneously measured by them during the process.

2.1.3. Processing

The deposition of the preform was performed at $1050 \text{ }^\circ\text{C}$ (measured by T1 thermocouple) for 3 h at an atmospheric pressure by the improved FBCVI. During the process, the

heating rate was controlled by adjusting the power of an inductive generator. N_2 flowed through the reactor for safety consideration. With the increasing deposition time, the precursor was consumed and resupplied from the precursor inlet to the reactor.

2.2. Characterization

The dimensions of the as-prepared C/C composites are $\Phi 110 \times 10 \text{ mm}^3$. In order to study the homogeneity of density and microstructure, three specimens labeled as S1, S2 and S3 were cut off from the composite along the radial direction, which was located at the distance of 0 mm, 25 mm and 50 mm to the axis (corresponding to the T1, T2 and T3 location), respectively. The densities and open porosities of the three specimens were measured using Archimedes principle. Microscopy observations on the polished surfaces and the fracture surfaces of the specimens were carried out by polarized light microscope (PLM, Reichert, MeF3) and scanning electron microscope (SEM, Hitachi, S-2700) operated at 20 kV and 20 mA, respectively.

3. Results and discussion

3.1. Temperature distribution in the preform

The key point of this process is the control of temperature distribution in the preform, either in axial or in radial direction [5,11]. Therefore, we designed a double heat source. This creates two extensive thermal gradients in the axial direction of the preform. Both sides of the preform are heated simultaneously by H1 and H2. The temperatures of the upper and lower surfaces of the preform are the highest. The deposition firstly occurred on these two surfaces and then the formed densification zones moved successively to the lower temperature zone. Thus, a rapid single cycle densification of the preform can be achieved due to the double high temperature gradients along the axis.

In the radial direction of the preform, a uniform temperature of the heat surface is required to obtain uniform thickness and microstructure of the composite. Under the experimental conditions of the present paper, the temperature distribution in the radial direction of the upper surface of the preform is shown in Fig. 2, which was recorded

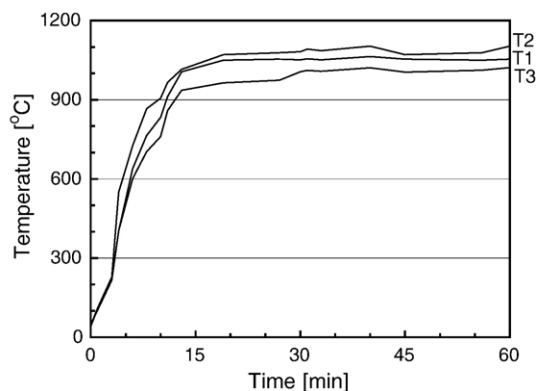


Fig. 2. Temperatures vs. time of three thermocouples recorded during the process.

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