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Fast densification of thick-walled carbon/carbon composite tubes using electrically coupled chemical vapor infiltration

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ABSTRACT

Rapid densification of thick-walled carbon/carbon (C/C) composite tubes by electrically coupled chemical vapor infiltration (EC-CVI) was investigated. The bulk densities and pore sizes of the C/C composites after different densification periods were measured. The results show that C/C composites were able to be densified by EC-CVI rapidly and efficiently. A thick-walled C/C composite tube, whose outer diameter, inner diameter and height was 240, 60 and 200 mm, respectively, was able to be densified to 1.71 g/cm^3 after a densification time of 400 h. If the outer diameter and inner diameter were increased to 400 and 100 mm, respectively, the final density of the component reached 1.50 g/cm^3 under the same experimental condition. After densified by EC-CVI, the amount of the pores with a size larger than 100 μm decreased significantly, and the porosity was even close to zero for the composites with a density of 1.62 g/cm^3. It indicates that EC-CVI can effectively fill the large pores in the composites. In the densified composite by EC-CVI, the pyrocarbon on the carbon fibers presented a rough laminar and smooth laminar morphology dominantly. Finally, the densification mechanism of the C/C composites by EC-CVI was simulated by the fractal method.

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1. Introduction

Chemical vapor infiltration (CVI) is one of the most common methods to densify carbon/carbon (C/C) composites [1,2]. However, the main drawback of the conventional CVI is long processing time and high costs. Therefore, new approaches, such as thermal gradient CVI (TG-CVI) [3] and electrified heating CVI (ECVI) [4–6], were explored to shorten the processing time and to reduce the costs. Densification efficiency of TG-CVI has been greatly enhanced compared with conventional CVI. Especially, TG-CVI can be used to prepare nozzle and other tube-like C/C composites with thick wall [7,8], but its densification process is still controlled by Fick and Knudsen diffusion [9,10], similar to the conventional CVI process. Therefore, it is not an effective method to prepare thick-walled C/C composites. ECVI is a kind of electrified preform heating fast densification technology. In comparison to the methods mentioned above, C/C composites are able to be densified in a shorter period by ECVI [4–6]. In this process, preforms are used as heat producing resistance, temperature gradient is generated in the composites and the deposition takes place from the inner part to the outer part of the preform. In addition, an electromagnetic field can be generated surrounding the fibers in the preform. This field can drive radicals, which acts as an intermediate for the pyrolysis of hydrocarbon gas, to flow directionally. The gas diffusion rate

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and infiltration rate can be thus improved. C/C composites, whose length, width and thickness were 200, 100 and 25 mm, respectively, were able to be densified to a final density of 1.70 g/cm^3 after a deposition time of 20 h by ECVI [11]. According to the work of Farhan et al. [12], the bulk density of a C/C cylinder with a dimension of ϕ75mm × 355 mm increased from 0.15 to 1.78 g/cm^3 after a deposition time of 67 h by ECVI. However, ECVI is not suitable for the densification of thick-walled C/C composites, since preforms themselves act as a resistance heater and it is difficult to form a relatively high temperature gradient in the preforms at initial stage of densification, which limits the densification rate intensively. Moreover, electric resistance varies continuously during the densification of the preform, it is thus necessary to adjust the current and voltage frequently and a special electric power source is required. In reality, this process is hard to be controlled and it is not suitable for the preparation of thick-walled C/C composites.

In the present study, an electrically coupled chemical vapor infiltration (EC-CVI) process, which combines the advantages of TG-CVI and ECVI, was developed to manufacture thick-walled C/C composite tubes. The coupling here refers to the interaction and influence between two or more systems and movements. Finally, the mechanism of the densification process by EC-CVI was investigated.

2. Experimental

2.1. Experimental set-up

Fig. 1 illustrates the set-up of EC-CVI based on the conventional TG-CVI. The round rod or cylindrical heating element was located in the center of the tubular carbon fiber felt and they were kept in contact with each other as a coupling for EC-CVI. Because of the large difference in the resistances between the carbon felt and the heating element, the current flows mainly through the heating element at the beginning of the process. Taking advantage of the similar resistances between pyrocarbon and graphite, the deposition layer will be gradually involved in the electric conduction and heating with an increasing deposition time and density of the preforms during the densification process, which can improve the heat transfer efficiency and generates a gradient electromagnetic field in the densification region.

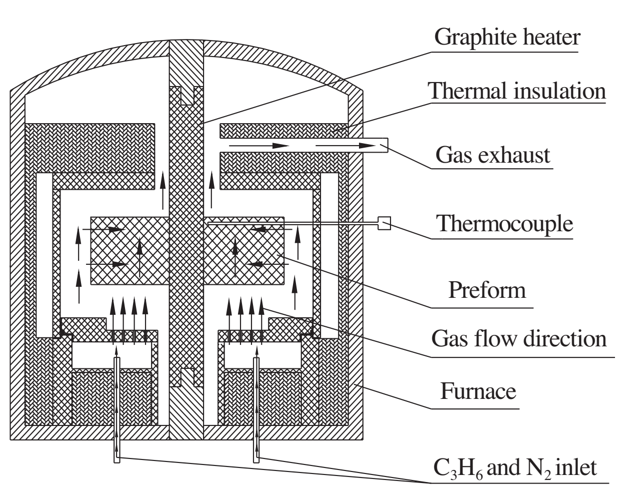

Fig. 1 – Experimental set-up of EC-CVI for fabrication C/C composites with EC-CVI.

Compared to TG-CVI with only infrared heating, both infrared heating and electric resistance heating of densified zone exist during the EC-CVI process. Its heating efficiency and densification rate are thus able to be improved. Previous results [4,5] tell us that such an electromagnetic field is able to adsorb the radicals, which act as the intermediates of the pyrolysis of hydrocarbon, which enhances the infiltration efficiency of the process. Moreover, the adsorption capability increases with increasing density of the deposited layer. In view of these facts, EC-CVI exhibits a great potential in producing dense C/C throats and other thick-walled C/C composite tubes.

2.2. Materials

Two sets of thick-walled tube-shaped carbon felt preforms (Xi'an Aerospace Composites Research Institute, China) were used in the present study. The densities and the specifications of the preforms are listed in Table 1. The Precursor and carrier gas used in the present study are propylene (C_3H_6) and nitrogen (N_2), respectively.

2.3. Densification of preforms

During the EC-CVI process, the temperature in the inner wall of the preforms was kept at 1150 °C. Both of the flow rates of propylene and nitrogen were 1.0 m^3/h. The pressure inside the furnace was kept at a value of 8–10 kPa. Bulk density of the preforms was measured after each period of 80 h. In the first measurement, both inner wall and outer wall surfaces of the preforms were machined in order to obtain a regular geometrical shape of the preforms for the density measurement. In the subsequent measurements, only the inner wall surface of the preforms was machined, since the heating element and the preform adhered with each other after the deposition and it led to local roughness of the inner wall surface, which can influence the electric conduction during the subsequent densification steps. The samples used for the measurement of porosity were cut along a circular ring of tube end, which was 10 mm away from the internal wall of Felt-NO. 1.

2.4. Characterization

The microstructure of pyrocarbon was investigated by a polarized light microscopy (MeF3A, Germany). The surface morphology of the C/C composites was investigated by a scanning electron microscopy (HITACHI S-450, Japan). The porosity and its distribution were measured by a mercury intrusion porosimeter (Micromeritics 9310, America). The samples used to study the density distribution along the radial and axial directions were machined from the positions of Felt-NO. 1, as shown in Fig. 2.

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