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Short communication

Preparation of high texture three-dimensional braided carbon/carbon composites by pyrolysis of ethanol and methane

Junjie Ren, Kezhi Li*, Shouyang Zhang, Xiyuan Yao, Hejun Li

State Key Laboratory of Solidification Processing, Northwestern Polytechnical University, Xi'an 710072, China

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Abstract

A three-dimensional (3D) carbon/carbon (C/C) composite was prepared using the gaseous mixture of ethanol and methane as the precursor by isothermal chemical vapor infiltration. The preform was infiltrated at 1100 °C with the gas pressure from 2 to 10 kPa. The texture of the infiltrated carbon was studied by polarized-light microscopy and characterized with the aid of the extinction angle. Texture and fracture morphology of the pyrolytic carbon matrix were observed using a scanning electronic microscope. After 150 h infiltration, the average bulk density was up to 1.72 ± 0.02 g cm^{-3}. 3D C/C composites with high texture pyrolytic carbon matrix were obtained by pyrolysis of ethanol and methane. The average flexure and tensile strengths of the composites are 362 MPa and 116 MPa, respectively.

Keywords: Carbon/carbon composites; High texture; Pyrolytic carbon; Ethanol; Mixture precursor

1. Introduction

Carbon/carbon (C/C) composites have been used in both military and commercial aircrafts because of their high thermal conductivity, low thermal expansion coefficient, ablation resistance, wear resistance, high thermal stability and excellent mechanical properties under high temperature [1–3]. Recently, high speed and heavy load are the trend for aircrafts [4]. It is necessary to improve the properties of the structure components.

As the reinforced component of C/C composite, the fiber preforms play a dominant role for their mechanical properties. Various braided techniques have been developed for the manufacture of fiber preforms [5]. C/C composites with three-dimensional braided preforms are widely recognized [6]. The 3D braided technology can offer new fiber architectures and shapes for composite design, which is good for the material properties and lightweight design of aircrafts [7,8]. However, researches on preparation of 3D braided C/C composites are few.

As another key component in C/C composites, carbon matrix also has major contribution to the characteristics of the composites. It has been found that tribology and ablation characteristics of C/C composites are greatly related to the textures of the pyrolytic carbon matrix [9–12]. The textures of pyrolytic carbons can be determined by their optical activity and extinction angle (A_e) with the aid of polarized light microscopy (PLM) [13,14]. The textures are defined as high texture (HT, $A_e \geq 18°$), medium texture (MT, $12° \leq A_e < 18°$), low texture (LT, $4° \leq A_e < 12°$), and isotropic ($A_e \leq 4°$) [15]. Among them, HT pyrolytic carbon is desired due to its advantages on ablation resistance and tribological properties [9–11].

Microstructure of 3D braided preforms has distinct differences from that of 2D laminated preforms. Inter-spaces of carbon fibers in 3D braided are bigger than those of 2D laminated, but those intra-spaces are smaller. The greater gap between inter- and intra-space would affect the uniformity of the pyrolytic carbon structure, especially for the formation of HT pyrolytic carbon [16]. To our knowledge, relatively less research has been carried out on the preparation of HT pyrolytic carbon in 3D braided preforms.

C/C composite with both HT pyrolytic carbon and 3D five-directional braided preform is expected to have the combination of both advantages. The densification of the 3D braided C/C composites by ICVI with the mixture precursor was studied.

*Corresponding author. Tel.: +86 29 88494197; fax: +86 29 88495764.
E-mail address: likezhi@nwpu.edu.cn (K. Li).

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2. Experimental procedure

2.1. Material preparation

Polyacrylonitrile-based (PAN-based) three-dimensional braided integrated felts with dimension of $8 \times 3.5 \times 70$ mm^3 were used as the preforms. The preforms were braided from T300 1 K carbon fibers. Initial relative porosity of the preforms is 69%. The carbon fibers have a typical density of 1.76 g cm^{-3}.

The preforms were infiltrated by ICVI at 1100 °C under the pressure of 2–5 kPa. The preform was fixed in a self-designed holder (as seen Fig. 1) during deposition. The precursor was the gaseous mixture of methane and ethanol. Nitrogen was employed as the carrier and diluent gas. The average density of the samples was upto 1.72 ± 0.02 g cm^{-3} after 150 h.

2.2. Characterization of C/C composites

After deposition, the samples were sanded to rectangular bars for further test. The density and open porosity of the samples were determined by the Archimedes principle [17]. The textures of the pyrolytic carbon were observed using a Leica DMLP polarized light microscope (PLM). The mechanical properties of the composites were characterized by tensile and three-point bending test. The tests were carried out on a MTS CMT5304 universal test machine. The force and position accuracy was $\pm 0.5\%$. During the three-point bending test, the samples were cut to the dimension of $55 \times 7 \times 3$ mm^3 along the braided direction. The span was 40 mm and the loading speed was 0.5 mm/min. In the tensile test, the dimension of the samples was $70 \times 7 \times 3$ mm^3 with the gage length of 20 mm. Morphology of the fractured surface of the sample was observed using a ZEISS SUPRA 55 scanning electron microscope (SEM).

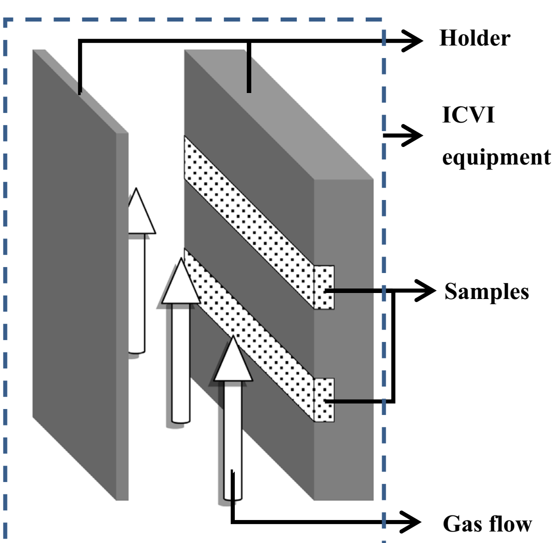

Fig. 1. Schematic of the infiltration holder.

3. Results and discussion

3.1. Densification

The average density of the samples is up to 1.72×0.02 g cm^{-3} after 150 h infiltration with an open porosity of 10.1%. Fig. 2 shows the bulk density of the C/C composites as a function of the deposition time. It is found that the densification rate is relatively high in the early stage. Then the rate has a rapid decrease and keeps a lower value. The average densification rate (1.72 ± 0.02 g cm^{-3} after 150 h) is lower than that of the 2D needle-punched integrated felt (1.8 ± 0.02 g cm^{-3} after 85 h), which has been reported in the previous work with similar process condition of ICVI [18]. The difference between the densified rates can be attributed to the preform types. 3D braided preform has more inter-spaces and fewer intra-spaces. The small intra-spaces can be filled in the early stage of the deposition, but the inter-spaces are entirely different. The inter-spaces in the 3D braided preforms have a lower [A_S/V_R]-ratio (where A_S stands for the surface area of the substrate and V_R for the volume of the deposition reactor) and fewer active sites per unit volume, compared with those in 2D preforms, which lead to a lower deposition rate [19]. After the intra-spaces being filled, some active sites disappear, accompanied by the vanishing of the pores, and the deposition mainly takes place in the inter-spaces. So the densification rate decreases after 30–60 h, and then keeps in a low value. After deposition for 150 h, the open porosity is still as high as 10.1%, which proves the above explanation and show the possibility of further densification. The CVI processes may be optimized by adjusting the temperature, the pressure or residence time, under the precondition of forming of HT high texture pyrolytic carbon. Furthermore, the big pores are not likely to be sealed in the early stage of deposition, which is beneficial to the densification. The densification rate may be improved for further appropriate study. Above all, it is confirmed that the gaseous mixture is qualified as the precursor for the densification of 3D braided C/C composites.

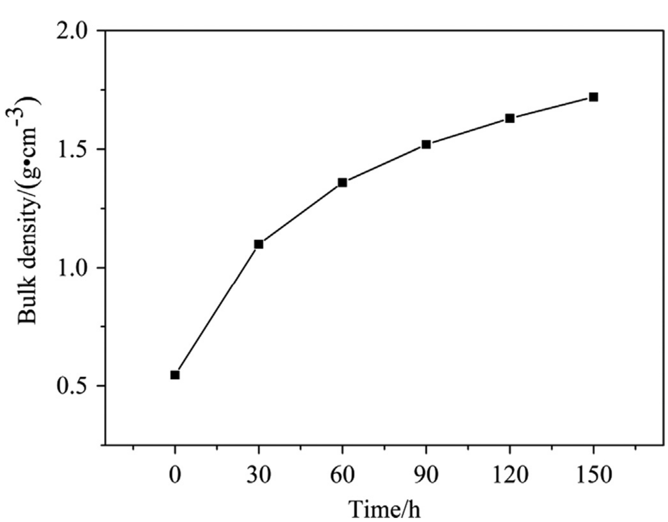

Fig. 2. Bulk density of deposited composites as a function of deposition time.