THE MICROSTRUCTURES OF C/C COMPOSITES BY CHEMICAL LIQUID-VAPOR INFILTRATION IN THE CASE OF CATALYST

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Abstract

Carbon/carbon (C/C) composites had been prepared by chemical liquid-vapor infiltration process between 700℃ and 1000℃ using toluene as precursor. As the catalyst, the corresponding weight percentage of ferrocene in 2D-PAN carbon felt preforms were 1, 3, 5 and 10, respectively. The influence of ferrocene content on the microstructures of C/C composites had been discussed. The textures of C/C composites were observed by polarized light microscope. Results show that the microstructures of C/C composites mainly consist of regeneration laminar pyrocarbon when ferrocene content is 1 wt.% in the preform. As the increasing of ferrocene content, the microstructures are apt to rough laminar (RL) pyrocarbon firstly around carbon fiber, and then smooth laminar (SL) pyrocarbon. The layer width of SL pyrocarbon increases and the layer width of RL pyrocarbon decreases with the increase of ferrocene contents in the preforms. The pyrocarbon was a cone-like structure. Generally, the mixture of RL and SL laminar can be obtained because of the quite differences of deposition temperature and gas concentration in the different positions of the preforms during the process.
Key words: Carbon composites; Catalyst; Pyrolysis;

Introduction

As a rapid technology of densification for carbon/carbon (C/C) composites, chemical liquid-vapor infiltration (CLVI) makes the densification rate two or more orders magnitude larger than the traditional isothermal CVI/CVD process. In the case of CLVI process, the different microstructures have been usually obtained ranging from nearly isotropic to highly anisotropic pyrocarbon because of the complexity of deposition condition during the process. Most researchers have been focus on the microstructures obtained by the modification of liquid precursor (cyclohexane, benzene, chlorobenzene, toluene and kerosene) and process parameters (e.g. deposition temperature, time), but little have been reported on the effect of catalyst on the textures.

In the present study, using the ferrocene as catalyst and toluene as the liquid precursor, C/C composites had been prepared by CLVI process. The influences of catalyst contents on microstructures were briefly discussed.

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Experimental

Preforms used in this study were 3K-PAN carbon fiber needled felts (fiber diameter: 6~8 μm, porosity: about 70 % measured by penetration technique) with the apparent density about 0.6 g/cm^3. The size of disk-shaped preforms was 110mm o.d. ×25 mm thickness. Ferrocene ($(C_5H_5)_2Fe$) was used as the source of catalyst. The weight percentages of catalyst added are 1, 3, 5 and 10 in the preforms, respectively. 2D-C/C composites had been prepared by CLVI process at 700~1000℃. The microstructures of C/C composites were observed by polarized light microscopy (PLM).

Results and discussion

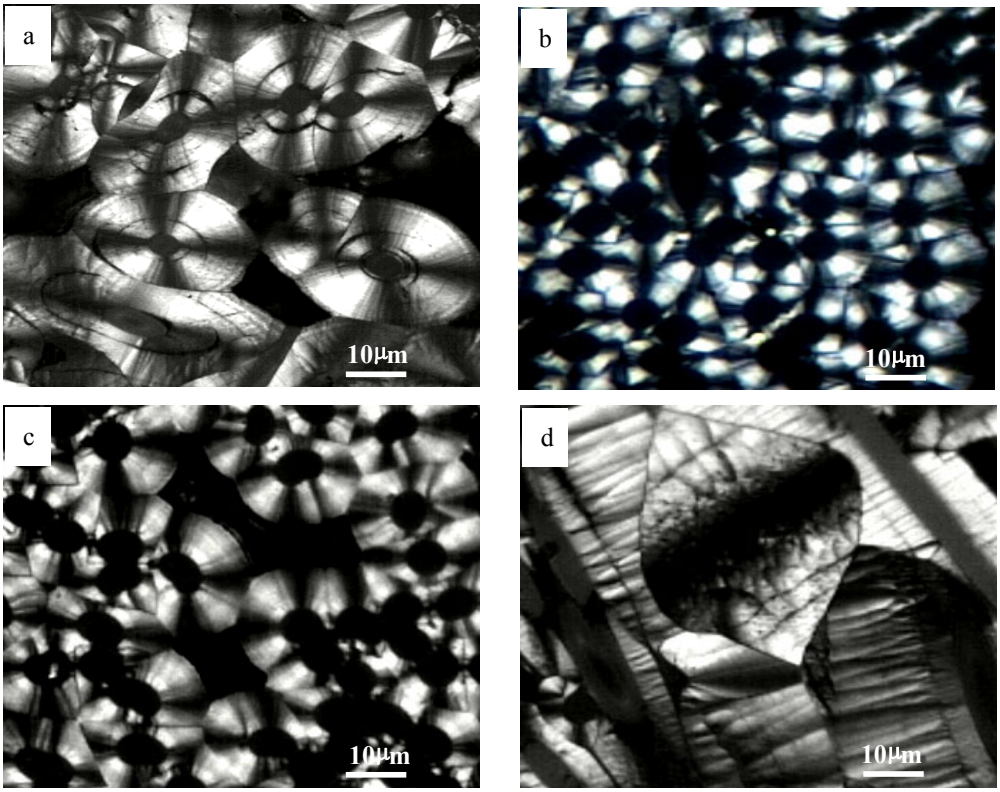

Figure 1. PLM photographs of C/C composites

a:1 wt.%, b:3 wt.%, c:5 wt.%, d:10 wt.%

Fig.1 shows the PLM photographs of C/C composites. As seen from Fig.1, when ferrocene content is 1 wt.% in the preform, regeneration laminar (ReL) pyrocarbon are obtained (Fig.1 (a)), which possesses a high brieflectance and a value of 20° for the A_e but exhibits a smooth extinction of the Maltese-cross in polarized light. But some annular cracks have been found in the ReL pyrocarbon, this may be caused by the mismatch of thermal dilatability between catalyst particles and carbon matrix. As ferrocene contents increase from 3 wt.% to 10 wt.%, the microstructures are apt to rough laminar (RL) pyrocarbon firstly around carbon fiber, and then smooth laminar (SL) pyrocarbon (Fig.1 (b), (c)). But the layer width of SL pyrocarbon increases and the layer width of RL pyrocarbon decreases. Iron particles pyrolyzed from ferrocene deposit on the carbon fiber surface and act as the active sites for

carbon deposition, the pyrocarbon exhibits a cone-like structure (Fig.1 (d)). In each cone, the pyrocarbon grows laminar by laminar in pattern of the curved surface, resulting in the cone-like outline of the deposited pyrocarbon with the growth laminar propagating. In the case of catalyst, the microstructure of C/C composites tends to form “ISO” pyrocarbon, together with the presence of carbon nanostructure. But carbon nanofibers/nanotubers have not been found in this work, this may be determined by deposition mechanism and process parameters. Generally, the mixture of RL and SL laminar can be obtained because of the quite differences of deposition temperature and gas concentration in the different positions of the preforms during the process.

Conclusions

Using ferrocene as catalyst and toluene as the liquid precursor, 2D-C/C composites had been prepared by CLVI technology. After adding ferrocene to the preforms, the microstructures mainly consist of ReL pyrocarbon when ferrocene content is 1 wt.%. With the increase of ferrocene content, the microstructures are apt to RL pyrocarbon firstly around carbon fiber, and then SL pyrocarbon together with the increase of layer width of SL pyrocarbon and decrease of the layer width of RL pyrocarbon.

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