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Journal of the European Ceramic Society 27 (2007) 4813–4820
www.elsevier.com/locate/jeurceramsoc

Microstructure-induced thermal stresses in pyrolytic carbon matrices at temperatures up to 2900 °C

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Received 25 November 2006; received in revised form 16 March 2007; accepted 25 March 2007
Available online 21 May 2007

Abstract

Carbon/carbon composites produced by chemical vapor infiltration consist of carbon fibers embedded in a pyrolytic carbon matrix with a cylindrically layered structure at the microscale. Each coating layer has a different texture and different mechanical properties that depend on temperature. Stress distributions in such carbon matrices subjected to thermal loading and their possible failure scenarios are analyzed. A two-scale numerical model is developed. At the nanoscale, material properties of each layer are determined using a methodology based on the Eshelby's theory for continuously distributed inclusions. The resulting material parameters for each layer are then used in the finite element modeling at the microscale. Calculations are conducted for composites with different matrix structures for several cases of thermal loading. Calculated stress distributions show zones of maximal stress concentration and provide information on possible failure regions which correspond well with experimentally identified failure regions.

Keywords: Chemical vapor infiltration; Composites, Failure analysis; Thermal properties; Carbon

1. Introduction

Carbon/carbon composites fabricated by chemical vapor infiltration (CVI) of carbon fiber felts have a complex hierarchical microstructure.[1,2] They consist of carbon fibers embedded in a pyrolytic carbon matrix. This matrix around the fibers has a cylindrically layered structure with each layer having different mechanical properties in the axial, radial and circumferential directions. These layers correspond to different dominating orientation distributions of basal planes (texture) in pyrolytic carbon (see Fig. 1). Using classification proposed in Ref. [3] the different textures of pyrolytic carbon matrices can be defined as isotropic (ISO), low-textured (LT), medium-textured (MT) and high-textured (HT). The texture of the pyrolytic carbon can be strongly influenced by the chemical vapor infiltration parameters (temperature, pressure, residence time, ratio between surface area and free volume) and by the post-heat treatment conditions (temperature, dwell time).

To fabricate carbon/carbon composites for industrial applications, heat treatments above 2000 °C are applied commonly after densification to a suitable density.[4] Our investigations of the influence of heat treatments on the microstructure and properties of carbon/carbon composites show that extensive cracks appear in the medium-textured pyrolytic matrix after heat treatment. Similar observations are reported in Ref. [5]. Cracks induced during the heat treatment may reduce dramatically the performance of the composite. In this work we analyze the influence of heat treatments on stress distributions and crack initiation in a medium-textured and high-textured pyrolytic carbon matrix produced by chemical vapor infiltration of carbon fiber felts. After presenting the experimental procedure, a two-scale modeling of stress distributions in these composites under high temperature is presented. Note that stress fields for fiber composites under

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doi:10.1016/j.jeurceramsoc.2007.03.023

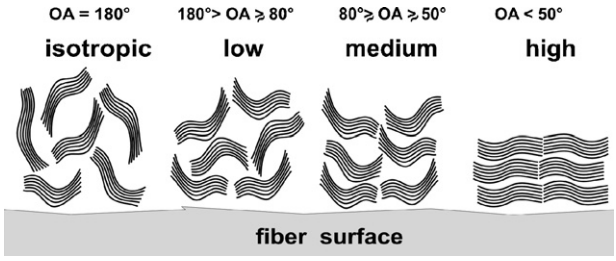

Fig. 1. Schematic presentation of the different dominating orientation distributions of basal planes in different layer textures.

thermomechanical loading and some related problems were analyzed, among others, in Refs. [6–9]. This was done mostly at the microscale with the use of concentric cylinder models. Here, the analysis focuses on both the micrometer and the nanometer scales. Stress distributions in and around carbon fibers for different textures of the surrounding pyrolytic carbon layers are studied for different cases of thermal loading.

2. Experimental procedure and observations

Carbon fiber felts (CCKF 1001, Sintec, Germany), made of randomly oriented chopped carbon fibers as shown in the SEM micrograph Fig. 2a, are used as preform for the infiltration. The fiber volume content is about 12%. The felts were infiltrated by means of an isothermal chemical vapor infiltration (I-CVI) process at different infiltration parameters (Fig. 2b) to obtain resulting material (Fig. 2c). The infiltration was carried out at the Institute for Chemical Technology of the Karlsruhe University (group of Prof. Hüttinger) in a vertical gap reactor shown in Fig. 2 using pure methane as precursor gas. Details on the infiltration procedure are given in Ref. [10].

Polarized light microscopy (PLM) investigations on polished cross-sections presented in Fig. 2 shows that the pyrolytic carbon matrix around the fibers has the shape of cylindrical layers at the micrometer scale. These have different widths and optical anisotropy.[1,11] An increased optical anisotropy correlates with an increased degree of texture. The textures of the deposited pyrocarbons were determined using an OLYMPUS AX70 microscope equipped with a polarizor and rotating analyzer according to their optical activity and value of the extinction angle A_e as described in Ref. [12]. The corresponding pyrocarbon optical textures with a progressive anisotropy degree are defined as isotropic (ISO, $A_e < 4°$), low-textured or dark laminar (LT or DL, $4° \leq A_e < 12°$), medium-textured or smooth laminar (MT or SL, $12° \leq A_e < 18°$), and high-textured or rough laminar (HT or RL, $A_e \geq 18°$).[12,3] These textures can also be characterized by selected area electron diffraction (SAED)[13] on sub-μm scale. The SAED investigations were carried out on transversal sections perpendicular to the fiber orientation using a 120 keV Zeiss EM 912 Omega transmission electron microscope equipped with an electron-energy filter integrated into the projection lens system. The extracted orientation angles (OA) are: $80° < OA < 180°$, $50° \leq OA < 80°$ and $OA < 50°$ for LT-, MT– and HT–carbon, respectively.

We study here materials with two different pyrolytic carbon matrix types, HT and MT. Their PLM micrographs are presented in Fig. 3. The HT-material (Fig. 3a) has a predominantly high-textured pyrolytic carbon matrix, the MT-material (Fig. 3b) has a mainly medium-textured pyrolytic carbon matrix.

These materials were treated in a vertical high temperature furnace (Thermal Technology Inc.) with a graphite-heating element for 2 h under a very pure helium atmosphere. Due to the high thermal radiation flux, a uniform temperature can be supposed for the entire sample. Fig. 4 shows schematically the realized experimental setup. In the experiment, two thermal regimes have been applied: heating up to 2200 °C and heating up to 2900 °C. The process of the thermal loading is the following. First the temperature is raised up to 1500 °C (60 min) in a power control regime. Then the thermal loading continued up to 2200 °C with a rate of 10 °C/min. Up to 2500 °C,

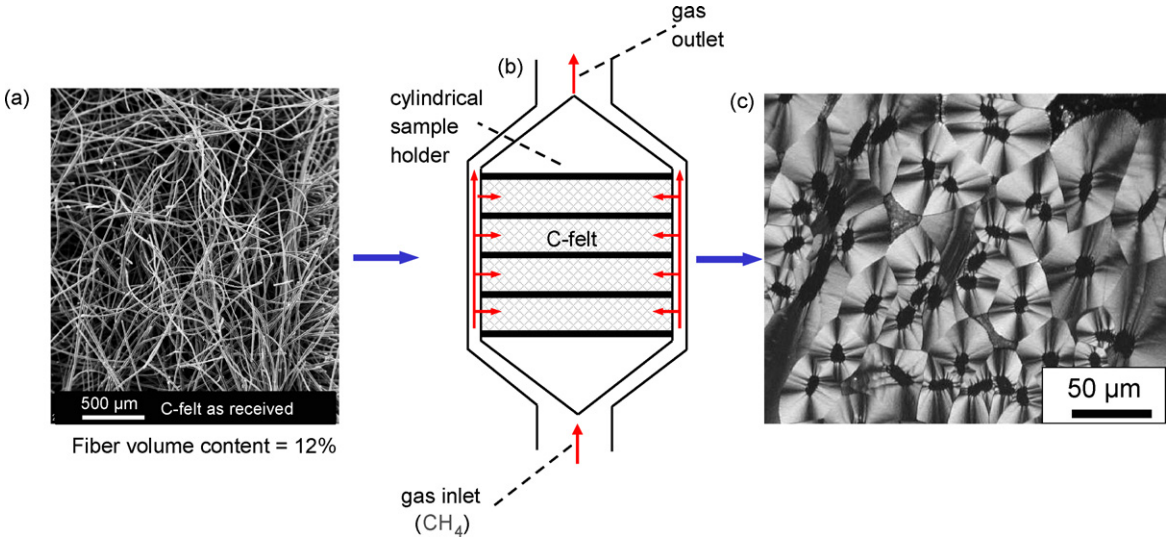

Fig. 2. Experimental setup and microstructure of the investigated materials: (a) SEM image of carbon fiber-felt preform; (b) scheme of CVI reactor; (c) PLM image of the resulting material.