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Characterization of partially densified 3D C_f/SiC composites by using mercury intrusion porosimetry and nitrogen sorption

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Abstract

The microstructure of partially densified three-dimensional carbon fiber fabrics reinforced silicon carbide (C_f/SiC) composites are characterized by both mercury intrusion porosimetry (MIP) and isothermal nitrogen sorption (INS). By comparison, MIP is preferable to the characterization for its wide effective probing ranges. Based upon multiple measurements, in the C_f/SiC composite, exists a complicated three-dimensional porous network formed by the interconnecting pores and necks with various sizes, diverse shapes and rough surfaces.

Keywords: C_f/SiC composite; Microstructures; Pore network; Mercury intrusion; N_2 sorption

1. Introduction

Carbon fibers reinforced silicon carbide (C_f/SiC) composites have been considered as one of the promising materials for various high temperature structural applications, e.g. aviation and aerospace, transportation system, energy production and chemical engineering, etc. [1–3]. Therefore, great efforts have being made to the R&D of C_f/SiC composites for decades, especially the continuous carbon fibers' fabrics reinforced ones.

Precursor infiltration and pyrolysis (PIP) is one of the most important fabrication processes for C_f/SiC composites, distinguished by its lower fabrication temperatures, simpler facilities and more feasibilities to produce components with complicated shapes. Due to the mass pyrolysis gas escaping and incomplete precursor infiltration, there are inevitably many microvoids and cracks in the interior of PIP fabricated C_f/SiC even after several PIP cycles, as shown in Fig. 1. This specific microstructure determines their mechanical and thermal properties, hence the characterization of it is valuable. Most previous evaluations of C_f/SiC composites were carried out by SEM images analysis [4], but it might be difficult to evaluate overall the materials' bulk quantitatively, because it analyzed two-dimensional limited fields only. Mercury intrusion porosimetry (MIP) and isothermal nitrogen sorption (INS) are classic methods for porous materials evaluation [5]. In this work, the above two approaches are used to characterize PIP-C_f/SiC composites, further microstructure information, besides surface area and pore size distribution (PSD), was obtained referring to the other porous materials' cases.

2. Experimental

The samples were produced through subjecting three-dimensional (3D) carbon fibers fabrics (T300, Toray Inc., Japan) to some infiltration–pyrolysis cycles, using polycarbosilane (PCS) as the precursor. The obtained 3D C_f/SiC composite had been incompletely densified. The morphology of samples' cross-section were characterized by SEM (JSM-5600LV, JEOL). Mercury intrusion porosimetry (AutoporeIII9420 (Micrometics, US)) assumed that the contact angle of Hg on the samples is 130°, and its surface tension is 0.485 N/m. The nitrogen sorption data were gathered by Autosorb-1 (Quantachrome Inc., USA) at 77 K.

3. Theory

3.1. Mercury intrusion porosimery (MIP)

Mercury intrusion is a popular method employed to evaluate porous materials microstructures. Conventional interpretations

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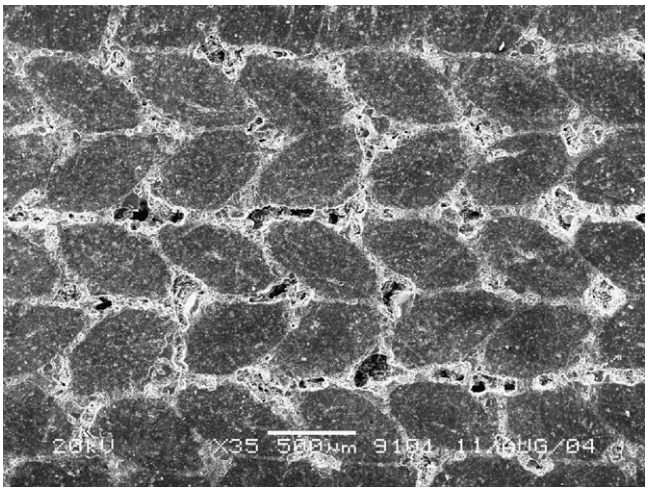

Fig. 1. SEM photo of 3D C_f/SiC composite's cross-section, note the obvious pore chambers and throats inside the bulk.

of original MIP data include porosity, density, and pore size distribution (PSD), etc. Besides these, other structural factors have also been determined based on some calculation models, such as:

3.1.1. Surface area

Surface area is a critical characteristic for porous materials. With the assumption that the contours of pores and channels are uniform, surface area S_w of open pores and connecting channels can be calculated by integrating the P–V curve of mercury intrusion branch, according to the expression as follows [6]:

$$S_w = \frac{1}{\sigma M \cos\alpha} \int_0^{V_{max}} P \, dV \tag{1}$$

where P, M, α, V is the intrusion pressure, sample's mass, contact angle and intruded mercury volume, respectively.

3.1.2. Fractal dimension

Fractal dimension (D) is a parameter to characterize the self-likeness degree of porous solids' microstructure. Owing to their great roughness and high distributing irregularity of pores, the porous materials have a much bigger D than ideal dense ones, undoubtedly. To calculate D from MIP, an expression is deduced from fractal theories and Washburn equation as follows [7]:

$$\log\left(\frac{dV}{dP}\right) = \log(k_2) + (D-4)\log P \tag{2}$$

where P is the intrusion or extrusion pressure, V the corresponding cumulative volume and k_2 is the constant. Hence, D values can be derived from $\log(dV/dP)$ versus $\log P$ plots.

3.2. Isothermal nitrogen sorption (INS)

Also an intruding approach, INS method measures the N_2 adsorption/desorption volumes of the sample at different pressures, from which one can get structural information, e.g. the surface area can be calculated as following [8]:

$$S = 4.36 V_m \tag{3}$$

where V_m is obtained by B.E.T. equation:

$$\frac{x}{V(1-x)} = \frac{1}{V_m C} + \frac{C-1}{V_m C} x, \quad x = \frac{P}{P_0} \tag{4}$$

The pore size distribution can be got based on Barrett–Joyner–Halenda (B.J.H.) model; and fractal dimension can be calculate by Frenkel–Halsey–Hill (F.H.H.) model [9]:

$$\ln\left(\frac{V}{V_m}\right) = \text{const.} + S \ln\left(\ln\left(\frac{P_0}{P}\right)\right) \tag{5}$$

where

$$S = \begin{cases} \dfrac{D-3}{3} & \text{at low pressures} \\ D-3 & \text{at high pressures} \end{cases}$$

Besides F.H.H., Song et al. [10] deduced the B.E.T. equation using to calculate D at low pressures, according to Friat's B.E.T. model:

$$\frac{1}{A} - \frac{1}{2^{2-D} C} \left[\frac{1}{x} + (C-1)\right] \tag{6}$$

where

$$A = \frac{1}{x}\left[\frac{V}{V_m} - \frac{Cx}{1+(C-1)x}\right]$$

4. Results and discussions

4.1. N_2 sorption isotherm and mercury intrusion curve

The N_2 sorption isotherm and mercury intrusion curves of the 3D C_f/SiC composite are shown in Figs. 2 and 3, respectively. In Fig. 2, the adsorption branch ascends slowly and continuously with the pressure increases at the beginning, when the relative pressure exceeds 0.8, the curve jumps

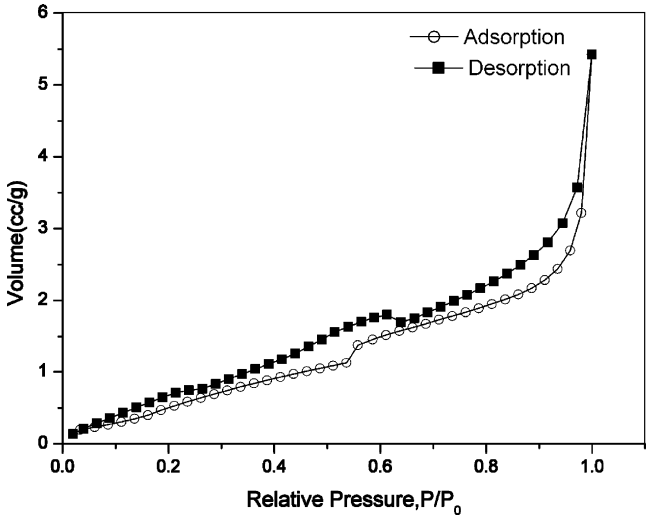

Fig. 2. N_2 sorption isotherm of the 3D C_f/SiC composite.