Materials and Design 30 (2009) 3738–3742

Contents lists available at ScienceDirect

Materials and Design

journal homepage: www.elsevier.com/locate/matdes

Influence factors of C/C–SiC dual matrix composites prepared by reactive melt infiltration

Jiang Si-Zhou, Xiong Xiang *, Chen Zhao-Ke, Xiao Peng, Huang Bai-Yun

State Key Lab for Powder Metallurgy, Central South University, Changsha 410083, China

ARTICLE INFO

Article history:
Received 24 September 2008
Accepted 9 February 2009
Available online 21 February 2009

Keywords:
A. Ceramic matrix composites
B. Fabrics
E. Mechanical

ABSTRACT

The effects of the type of carbon matrix, the infiltration temperature and high temperature treatment (HTT) on the infiltration behavior of molten Si and preparation of C/C–SiC dual matrix composites have been investigated. The results showed that: Resin carbon matrix is benificial to the infiltration of molten Si than pyrolysis carbon matrix. 1650 °C is more suitable for the infiltration of molten Si in porous C/C preforms than 1550 °C. The high infiltration depth at 1650 °C is responsible for the high density of C/C–SiC composites. HTT facilitates to the infiltration of molten Si and the formation of more SiC. High density and low open porosity C/C–SiC dual matrix composites can be prepared by optimal reactive molten infiltration method. The flexural strength, elastic modul and impact toughness of C/C–SiC composites are high up to 265.4 MPa, 28.1 GPa and 28.5 kJ/m^2 respectively. Heat treatment at 2300 °C decreases the composites flexural strength, elastic modul and impact toughness, but improve the fracture behavior of C/C–SiC dual matrix composites.

1. Introduction

C/C composites are the most promiseful brake disc materials for applications in military and civilian aircrafts. Unfortunately, they are susceptible to oxidation at elevated temperatures, and ease to failure in humidity environment [1–3]. Carbon fiber reinforced carbon and SiC dual matrix composites, namely C/C–SiC composites, of many advantages such as low density, good mechanical and tribological properties, good thermal properties, good oxidation and erosion resistance, can survive in severe service conditions of aviation and space missions. Compared to C/C composites, C/C–SiC composites have less change for high temperature failures. Therefore, as novel friction and wear materials, C/C–SiC composites have been considered to have wide application prospect [4–9].

Reactive melt infiltration (RMI) is one of the successful methods to prepare C/C–SiC composites. Compared to other processes such as slurry hot pressure, polymer infiltration pyrolysis and chemical vapor deposition (CVD), RMI possesses many advantages including short fabrication period, low cost, near net shape, low porosity, etc. [10–12] and has recently become a commercialized method of great market competition.

However, to prepare C/C–SiC composites by RMI, the influence factor and infiltration behavior of molten Si may be very complex. In this article, the influence of the type of carbon matrix, the infiltration temperature and high temperature treatment (HTT) on the infiltration behavior of molten Si and the preparation of C/C–SiC composites will be investigated in detail. In addition, in the RMI process, one may degrade carbon fibers, leading to a catastrophic failure of rupture. Therefore, the mechanical properties of C/C–SiC composites had been also discussed.

2. Experimental procedures

2.1. Raw materials

To prepare porous C/C preforms with resin carbon matrix or pyrolysis carbon matrix, PAN carbon fibre needled felt with a density of 0.6 g/cm^3, Furan resin with a viscosity of 40–150 MPa s (at 25 °C), C_3H_6 and N_2, were used as preform, impregnant, CVD carbon source and dilute gas respectively. In RMI process, silicon powders of 99% purity and 75 μm in diameter were used as Si source to prepare C/C–SiC dual matrix composites.

2.2. Materials preparation

2.2.1. Preparation of porous C/C preforms

PAN carbon fibre needled felts are used as preforms. The carbon fiber was PAN-based (T300, 12k, Toray, Japan). The needled felts were prepared by the three-dimensional needling technique, starting with repeatedly overlapping the layers of 0° non-woven fiber cloth, short-cut-fiber web, and 90° non-woven fiber cloth with needle-punching step by step.

* Corresponding author. Tel.: +86 731 8836079; fax: +86 731 8836079.
E-mail address: Xiong228@sina.com (X. Xiang).

0261-3069/$ - see front matter © 2009 Elsevier Ltd. All rights reserved.
doi:10.1016/j.matdes.2009.02.013

Porous C/C preforms with Resin carbon matrix or pyrolysis carbon matrix were prepared by impregnation and carbonization (IC) process or CVD respectively. IC process was consisting of three steps, Furan resin impregnation in preforms, solidification at 180–200 °C for 1 h, and carbonization at 800–1000 °C for 2 h. The isothermal CVD was operated at 900–1150 °C with C_3H_6 as the reactant gas and N_2 the dilute gas.

2.2.2. Preparation of C/C–SiC composites

Porous C/C prerforms were embedded in Si powders in a graphite pot. The pot was then placed in a high temperature induction furnace to prepare C/C–SiC dual matrix composites at either 1550 °C or 1650 °C for 1–2 h. In infiltration process, the high temperature furnace was protected by inert gas at 1 atm pressure.

In order to study the influence of HTT, porous C/C preforms with resin carbon matrix were treated at 2100–2300 °C for 2 h prior to RMI process.

2.3. Characterization

The density and open porosity of porous C/C preforms and C/C–SiC composites were measured according to Archimedes principle. Samples with a size of 40 mm×5 mm×3 mm were used to test the flexural strength by INSTRON CSS – 44100 machine. The gauge size was 30 mm and the cross head speed was 0.5 mm/min. The load–displacement curves were recorded by computer at the same time.

Film composition was analyzed by a D/max 2550 VB+18 kW rotating target X-ray diffraction (XRD) analyzer (Rigaku Ltd., Japan, Cu Kα radiation, λ = 1.54056 Å). Microstructure was analyzed by a JEOL-6360LV scanning electron microscopy (SEM) with working voltage of 25 kV.

3. Results and discussion

3.1. The influence of carbon matrix

To study the influence of carbon matrix on the infiltration behavior of molten Si, we conducted more tests at 1550 °C. The results are showed in Table 1. For resin carbon matrix, the density of composites is high with low porosity. For pyrolysis carbon matrix, the density is relative lower with slight larger porosity.

Table 1
Influence of carbon matrix on density and open porosity of C/C–SiC composites.

Matrix	Porous C/C preforms		C/C–SiC composites		$\Delta\rho$ (%)
	ρ_1 (g cm^{-3})	ε_1 (%)	ρ_2 (g cm^{-3})	ε_2 (%)	
Resin carbon	1.38	24.0	2.25	1.0	63.4
Pyrolysis carbon	1.34	19.5	2.04	3.9	52.2

Note: ρ_1 and ε_1 are the density and open porosity of porous C/C preforms; ρ_2 and ε_2 are density and open porosity of C/C–SiC composites.

The main reason for higher density of C/C–SiC composites with resin carbon matrix is the easy infiltration and uniform distribution of molten Si. The uniform distribution facilitate the reaction of molten Si with resin carbon. As a result, SiC was formed and uniformly distributed in the composites (see in Fig. 1).

This can be testified by studying the morphology of SiC. The morphologies of SiC in resin carbon matrix and pyrolytic carbon matrix are quite different (see in Fig. 2). For resin carbon matrix, molten Si can flow into porous C/C preform easily and react with resin carbon to form a 2–7 μm thick SiC layer. While for pyrolytic carbon matrix, molten Si can only react with pyrolytic carbon in the outer layer. The obtained SiC layer is thin and takes the original morphology of pyrolytic carbon. Note that the infiltration process of molten Si can be influenced by the different SiC morphologies. In porous C/C preform with resin carbon matrix, the wetting angle (θ) changes from that between molten Si and solid C to that between molten Si and solid SiC due to the formation of a continuous SiC layer. Then the wetting angle is decreased with improved wetting performance. Therefore, the infiltration process of molten Si will be enhanced accordingly. This is to say, Resin carbon matrix is beneficial to the infiltration of molten Si.

3.2. The influence of temperature

Porous C/C preforms with resin carbon matrix were used to study the influence of temperatures on the infiltration behavior of molten Si. The results were listed in Table 2. The infiltration of molten Si was difficult at 1550 °C and the composites were ended up with low relative density and high porosity (ε > 12%). At 1650 °C, the infiltration was successful with low porosity (ε < 5%) of the composites.

Assuming the pore shape in porous C/C preform is cylinder and ignoring the ascending inertia force of molten Si, Washburn has established the infiltration equation [13–15],

$$\frac{dh}{dt} = \frac{C}{8\mu h}\left\{\frac{2\sigma\cos\theta}{r(t)} - \rho g h\right\} r(t)^2 \quad (1)$$

where h denotes the infiltration height or depth in m; t the infiltration time in s; μ the viscosity of molten Si in Pa S; σ the surface tension of molten Si in N/m; θ the wetting angle between molten Si and solid; $r(t)$ the mean capillary radius at the time in m; g the gravity in m/s^2; ρ the density of molten Si in g/cm^3. C is a factor whose value is usually taken to be 1/3, assuming isotropic porous media.

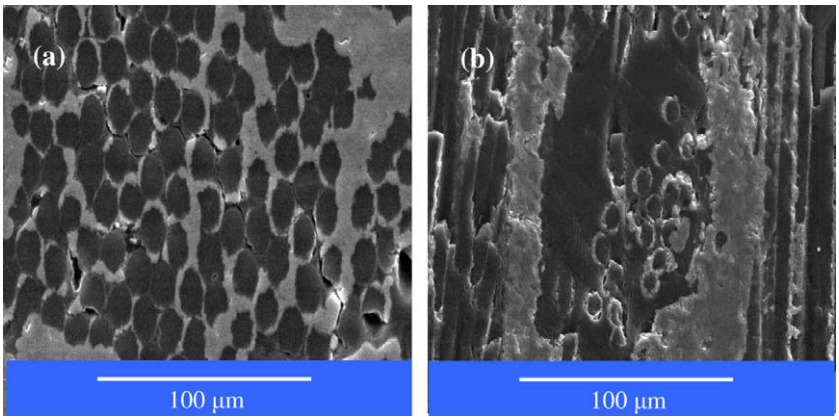

Fig. 1. Microstructures of C/C–SiC composites with resin carbon matrix (a) inter-fiber and (b) inter-bundle.