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Effects of dip-coated BN interphase on mechanical properties of SiC_f/SiC composites prepared by CVI process

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Abstract: BN interphase was successfully synthesized on SiC fiber fabrics by dip-coating process using boric acid and urea as precursors under N_2 atmosphere. The morphology of BN interphase was observed by SEM, and the structure was characterized by XRD and FT-IR spectra. The SiC_f/SiC composites with dip-coated BN interphase were fabricated by chemical vapor infiltration (CVI) process, and the effects of BN interphase on the mechanical properties of composites were investigated. The results show that the SiC fibers are fully covered by BN interphase with smooth surface and turbostratic structure (*t*-BN), and the thickness is about 0.4 μm. The flexural strengths of SiC_f/SiC composites with and without BN interphase are about 180 and 95 MPa, respectively. Compared with the as-received SiC_f/SiC composites, the composites with BN interphase exhibit an obvious toughened fracture behavior. From the microstructural analysis, it can be confirmed that the BN interphase plays a key part in protecting the fibers from chemical attack during matrix infiltration and weakening interfacial bonding, which can improve the mechanical properties of SiC_f/SiC composites remarkably.

Key words: SiC_f/SiC composites; BN interphase; dip-coating; CVI; mechanical properties

1 Introduction

SiC_f/SiC composites exhibit a combination of attractive physical-chemical properties such as high strength, stability at elevated temperature, high corrosion resistance, low thermal expansion and low activation characteristics. Because of these superior properties, SiC_f/SiC composites have been widely studied and used for in nuclear fusion reactors and high temperature aerospace [1−5]. However, owing to the inherent brittle failure behavior of SiC matrix, the fracture toughness of the composites is not high.

For fiber-reinforced ceramic composites, the concept of weak interphase was promoted to increase the fracture toughness [6−9]. The weak fiber/matrix interphase has significant influence on the fracture behavior and mechanical properties of ceramic matrix composites (CMCs). This can attribute to the occurrence of various well identified energy-dissipating mechanisms, i.e., crack deflection, fiber-matrix debonding, fiber sliding and fiber pullout [10,11].

Pyrocarbon (PyC) was once the most commonly used weak interphase in the past. However, these composites often experienced severe embrittlement when they were tested at high temperature in oxidizing atmospheres [4,12]. So far, materials with a layered crystal structure such as hexagonal boron nitride (*h*-BN) have received much attention for use in SiC-based composites systems, since they have improved oxidation resistance compared to PyC. Hexagonal boron nitride (*h*-BN) is a promising interphase material for SiC_f/SiC composites, which is attributed to its unique properties such as high thermal conductivity, high temperature stability, high thermal shock resistance, and self-lubricating [12−14]. A barrier against oxidation by formation of B_2O_3 provides a better protection for fibers with the consequence to maintain the integrity of the composites for a longer duration under loading [12].

Up to now, the BN interphase is elaborated most by chemical vapor deposition (CVD) from $BCl_3/BF_3-NH_3-H_2/N_2$/Ar mixtures at a moderate temperature. However, it is associated with several disadvantages, including hazardous precursors and byproduct,

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degradation of fiber caused by reaction with HF or HCl, high cost and scale-up problems [15,16]. Dip-coating method has received considerable attention in recent years, which is attributed to its advantages compared to the chemical vapor deposition (CVD). The dip coating process, which is simple and practical, is used to synthesize coatings on nanowires, carbon nanotubes and powders [17].

In this work, BN interphase of SiC_f/SiC composites was fabricated by dip-coating process in boric acid and urea solutions followed by nitridation at nitrogen atmosphere. The morphology and structure of BN interphase were characterized. The effects of BN interphase on the mechanical properties of SiC_f/SiC composites were investigated.

2 Experimental

2.1 Materials

The SiC fiber fabrics were provided by Su Zhou SaiLiFei (SLF) Ceramic Fiber Co., LTD, China. The parameters of SiC fiber are listed in Table 1. The fabric structure is 2.5D shallow bending joint, as shown in Fig. 1. The fiber volume fraction of the fabrics is 45%.

Table 1 Parameters of SiC fiber

Parameter	Value
Diameter/μm	11−14
Density/($g\cdot cm^{-3}$)	~2.5
Mole ratio of C/Si	~1.16
Number of filaments/($fil\cdot yarn^{-1}$)	~600
Tensile strength/MPa	1500−1800
Chemical compositions of surface layer	Si−C−O

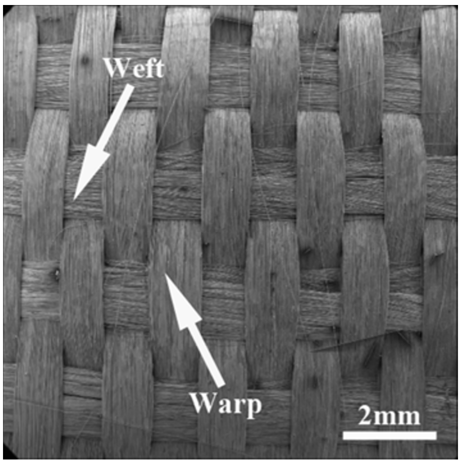

Fig. 1 SEM image of SiC fiber fabrics

2.2 Preparation of BN interphase

Before dip-coating, the SiC fiber fabrics were degreased in vacuum at 600 °C for 30 min, then washed with acetone and ethanol, respectively. In order to prepare the precursor solution, boric acid and urea (mass ratio of 1:9) were dissolved in 300 mL distilled water under stirring until it became transparent. Prior to CVI, the BN interphase was deposited on SiC fiber by dip-coating process. The degreased SiC fiber fabrics were dipped in the precursor solution and ultrasonically vibrated for 15 min, then infiltrated in vacuum for 10 min. After infiltration, the fabrics were dried in air for 12 h. Finally, the coated fiber fabrics were placed in a furnace for nitridation at 1000 °C at a rate of 10 °C/min under nitrogen atmosphere, and four times of dipping-annealing treatment were applied. The schematic diagram for the preparation of BN interphase is shown in Fig. 2.

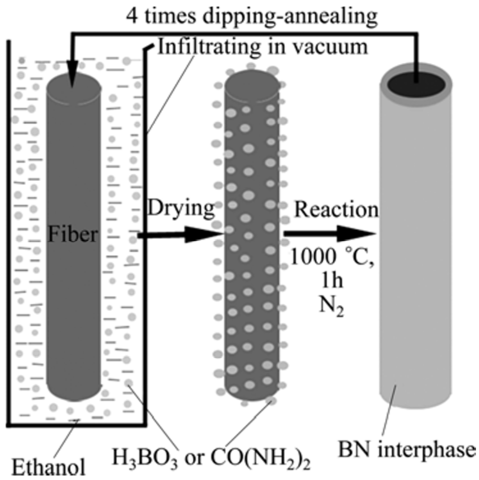

Fig. 2 Schematic diagram for preparation of BN interphase on SiC fiber by dip-coating process

2.3 Characterization

The surface morphology of BN interphase was examined by scanning electron microscopy (SEM, TESCAN, VEGA 3). The phase structure and the chemical bonds of the nitridation production from BN precursor were characterized by X-ray diffraction (X" Pert PRO MPD, PANalytical, Almelo, the Netherlands) and Fourier transform infrared spectroscopy (Nicolet Nexus 670), respectively. The chemical compositions of BN interphase were also characterized by SEM equipped with EDS.

In order to investigate the influence of BN interphase on the mechanical properties of SiC_f/SiC composites, the composites without and with BN interphase were prepared by CVI. SiC matrix was obtained by thermally cracking MTS (methyl tri-chlorosilane, CH_3SiCl_3) in the presence of argon as carrier and diluents gas with mass flow rate of 1:15 at 2−3 kPa and 1000 °C. The infiltration time is 16 h.

Flexural test specimens measuring 40 mm in length, 4 mm in breadth, and 3 mm in thickness were prepared from composites without and with BN interphase through water jet cutting. The cut surfaces and edges of