



The influences of carbon nanotubes introduced in three different phases of carbon fiber/pyrolytic carbon/silicon carbide composites on microstructure and properties of their composites

Jie Wang^{a, b}, Xiaming Zhang^c, Yulong Miao^a, Yaoyao Li^{a, b}, Xianfeng Xi^{a, b},
Xiqiang Zhong^a, Xueliang Pei^{a, *}, Liu He^{a, **}, Qing Huang^{a, ***}

^a Ningbo Institute of Material Technology and Engineering, Chinese Academy of Sciences, Ningbo Zhejiang 315201, PR China

^b University of Chinese Academy of Sciences, 19 A Yuquan Rd, Shijingshan District, Beijing 100049, PR China

^c Beijing Spacecrafts, Beijing 100190, PR China

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ABSTRACT

The influences of carbon nanotubes (CNTs) introduced in three different phases of carbon fiber/pyrolytic carbon/silicon carbide (Cf/PyC/SiC) composites on the microstructure and properties of the composites were investigated in this paper. The results showed with CNTs introduced at PyC-PyC interface, tensile strength and work of fracture (WOF) of a Cf/PyC/SiC composite had the largest increase, and significantly improved 54.9% and 130.3% respectively from those of the CNTs-free composite. Multiple interfaces (CF-PyC interface, CNT-PyC interface, PyC-PyC interface) failure and a great number of carbon fiber and carbon nanotube pull-outs were observed from tensile fracture morphology of the composite, which indicated weakening of PyC-PyC interface due to CNTs and accounted for the remarkable improvement in WOF of the composite. The increase of tensile strength of the composite was resulted from the reduction of residual stress of the composite.

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1. Introduction

Carbon fiber/silicon carbide (Cf/SiC) composites have potential for being used in rocket nozzle, aerospace engine and even nuclear protection field due to its low density, excellent strength and good resistance to high temperature [1–4]. The ways to prepare Cf/SiC composites mainly include reactive melt infiltration (RMI) [5–7], chemical vapor deposition (CVI) [8–10] and precursor infiltration and pyrolysis (PIP) [11–14]. As far as PIP is concerned, it needs much lower processing temperature than RMI to prepare composites and relieves degradation of carbon fibers (CFs) caused by high temperature. It also takes less time and saves enormous expense compared with CVI [15]. In addition, PIP has other advantages such as controllable ceramic compositions, availability of

complex reinforcements and near-net shape technologies [16]. Therefore, PIP attracts considerable attention from researchers in recent years.

Carbon nanotubes (CNTs) were discovered in 1991 [17] and found to have extremely high modulus, high strength and low density with further researches [18–20]. Up to now, CNTs have been widely exploited to be one important and effective interface material to improve polymer matrix composites' mechanic properties in recent years [21–24], but there were still few references reporting that CNTs were incorporated to SiC matrix composites. Zhao et al. [25] prepared a SiC/SiC composite by PIP, with insitu grown CNTs on the fiber fabric. The bending strength and fracture toughness of the SiC/SiC composite with CNTs increased by 22.3% and 103% from those of the SiC/SiC composite without CNTs. Feng et al. [26] reported that the flexural strength, fracture energy and interfacial shear strength of the SiC/SiC composite with introduction of CNTs on fiber perform increased by 17.4%, 25.7% and more than 100% from those of the SiC/SiC composite without CNTs, respectively. Mei et al. [27] studied the effects of electrophoretically deposited CNTs on the strength and toughness of CNT-C/SiC composites prepared via CVI. The results indicated that increasing

* Corresponding author.

** Corresponding author.

*** Corresponding author.

E-mail addresses: peixueliang@nimte.ac.cn (X. Pei), heliu@nimte.ac.cn (L. He), huangqing@nimte.ac.cn (Q. Huang).

electrophoretic deposition (EPD) time of 5, 8, 10, and 15 min were associated with CNT–C/SiC composite strengths that increased by 10.7%, 39.3%, 45.2%, and 28.9% compared with pristine C/SiC, respectively. For the same EPD periods, work of fracture of composites increased by 49.4%, 82.7%, 120.8%, and 32.3%, respectively, indicating significant improvement in toughness of composites due to CNT presence. Up to that time, there was still no systematic study about the effects of CNTs located in different phases of the same composite on the mechanical properties of the composite.

Until lately, Mei et al. [28] reported the effects of CNTs, introduced in different phases of carbon fiber-reinforced silicon carbide (C/SiC) composites prepared by CVI, on the microstructure and properties of the composites. With CNTs introduced at carbon fiber-pyrolytic carbon (C-PyC) interface, the tensile strength and work of fracture of the composite were found to increase by 67.3% and 107.2%, respectively, from the CNT-free case. With CNTs introduced at PyC–SiC interface, the mechanical properties of the composite had little change. However, the reasons why the tensile strength of different C/SiC composites was different were just downplayed, and the effects of CNTs introduced into PyC interlayer on mechanical properties of composites have not been studied in their work yet.

In this paper, Cf/SiC composites with phenolic resin-derived PyC interlayer, containing CNTs or not, were prepared by PIP process. The effects of CNTs introduced in three different phases of Cf/PyC/SiC composite by dip-coating process, especially CNTs located in PyC–PyC phase, on the tensile strength, work of fracture and microstructure of Cf/PyC/SiC mini-composites were investigated. The mechanisms for properties change of these composites were also disclosed and discussed deeply.

2. Experimental procedures

2.1. Materials preparation

Carbon fiber tow (12k, T700) was obtained from Zhongfushenying Co. Ltd (Suzhou, China). CNTs (multi-walled, 10–30 μm in length, 10–20 μm in diameter) were purchased from Chengdu Organic Co. Ltd (Chinese Academy of Sciences, Chengdu, China). PFNH-200 phenolic resin and hexamethylenetetramine were provided by Bomafenfang Co. Ltd (Xinxiang, China). Polycarbosilane (PCS) with number average molecular weight of 1742 and softening point of 175 $^{\circ}\text{C}$ was obtained from Sailifei Ceramic Fiber Co., Ltd. (Shuzhou, China). All other chemical reagents (analytical grade) were purchased from Aladdin (Shanghai, China).

Carbon fiber tow was desized with heat treatment at 500 $^{\circ}\text{C}$ for 1 h in an N_2 atmosphere, and then it was oxidized at 140 $^{\circ}\text{C}$ for 10 min in tube furnace using 40 mg/L ozone to improve its compatibility with phenolic resin solution. Phenolic solution prepared by uniformly mixing phenolic resin and hexamine (weight ratio of 10:1) in methanol was used as the precursor of PyC interlayer of composite. Oxidized CFs were tightly fixed on graphite frame with 10 mm in length and then were dipped in 4 wt% phenolic solution for half hour. After that, CFs were cured (80 $^{\circ}\text{C}$, 12 h; 145 $^{\circ}\text{C}$, 2 h; 210 $^{\circ}\text{C}$, 2 h) and pyrolyzed (keeping at 700 $^{\circ}\text{C}$ for 1 h with a heating rate of 10 $^{\circ}\text{C}/\text{min}$ in an N_2 atmosphere) to obtain PyC coating. 4 wt% was an optimized concentration neither making the bonding of CFs nor making coating on CFs too thin after CFs' dip-coating. Our previous research has found a complete and uniform PyC layer with thickness of approximately 120 nm could be coated on CFs through the above processes.

Incorporation of CNTs onto CFs surface was accomplished by dipping CFs fixed on graphite frame in CNTs suspension for 30 min followed by drying at 105 $^{\circ}\text{C}$ overnight. CNTs suspension was obtained through adding a certain amount of CNTs and home-

prepared surfactant into deionized water, then mixing them with ultrasonic (22 KHz, 600w) for 2 h.

The Cf/PyC/SiC mini-composites were prepared using 9 cycles of PIP with 50 wt% PCS–xylene solution, and the pyrolysis of PCS was conducted at 1100 $^{\circ}\text{C}$ for 1 h with a heating rate of 10 $^{\circ}\text{C}/\text{min}$ in N_2 atmosphere. The detailed procedures have been described elsewhere [29,30]. The Cf/PyC/SiC mini-composites with the first PIP process were cut off from graphite frame. Each final composite sample had length of 100 mm and cross-section area of approximately 0.95 mm^2 . Due to the same treatments during preparation of Cf/SiC mini-composites except for the processing sequences, the weight ratio of total PyC interlayer to CFs for all composites was approximately 8%, the weight ratio of CNTs to CFs for all composites was approximately 0.1%, and the volume content of fiber for all composites was near 48%. All Cf/PyC/SiC composites attained density of around 1.79 g/cm^3 and porosity of around 14.2%, as shown in Table 1.

The preparation processes of four types of Cf/SiC composites are shown in Fig. 1. The Cf/SiC composite with two PyC interlayers was denoted as Cf/PyC/PyC/SiC composite. The Cf/SiC composites with

Table 1
The properties of the as-received composites.

Composites	Fiber content (vol.%)	Density (g/cm^3)	Porosity (%)	Tensile strength (MPa)	WOF (KJ/m^2)
Cf/PyC/PyC/SiC	48	1.81	13.8	246 \pm 43	66 \pm 8.9
Cf/CNT/PyC/PyC/SiC	48	1.77	14.7	205 \pm 44	39.5 \pm 5.6
Cf/PyC/CNT/PyC/SiC	48	1.79	14.2	381 \pm 56	152 \pm 20.6
Cf/PyC/PyC/CNT/SiC	48	1.78	14.6	292 \pm 48	77.9 \pm 10.8

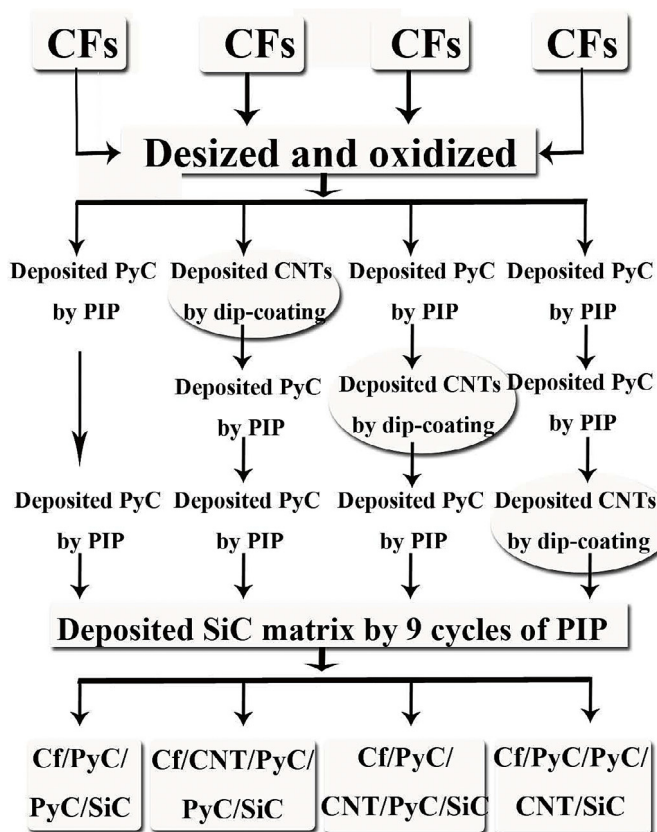


Fig. 1. Preparation processes of Cf/PyC/SiC composites.

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